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Relation between Physical Structure and Dyeability of Nylon 6 Fibers. II. Relation of Dyeability to Various Processing Factors in Bulky Yarn Production*

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Synopsis

The effects of tension, number of twists, and heat setting temperature on dyeing properties and the degree of crystallinity of nylon are examined. The amount of dyestuff adsorbed on stretch nylon decreases with tension but is not affected by number of twist, while the density is affected by neither of the above factors. The degree of twist fixing, crystallinity, and the amount of absorbed dyestuff increase with setting temperature. Factors such as setting temperature and tension are the main causes of uneven dyeing. To help in achieving level dyeing in industrial applications dyestuffs are classified according to their sensitivity to uneven stretch nylon 6 hosiery.

INTRODUCTION

The effect of heat setting on the fine structure and physical properties of nylon 6 fiber has been described in detail in the previous reports.[1-11]

In the present study, the relation of dyeability to various processing factors in bulky yarn production (Helanca process) in which nylon 6 fiber is subjected to heat setting was examined.

The effect of heat setting on nylon 6 filaments is influenced by many factors, such as tension, number of twists, temperature, and duration of the steam setting process; dyeability and mechanical properties are consequently also affected by these factors.

Various methods and devices are therefore being used to decrease the fluctuation of these factors in the manufacturing process. However, irregularities in heat setting are still often observed and are responsible for various types of difficulties in commercial processes.

For the dyer who deals with bulky yarn it is important that he be able to deal with these irregularities on dyeing and that he obtains material having good fastness properties. Such methods such as repeated heat setting, high pressure dyeing, and application of swelling agent, are commonly used to compensate for the irregularities; however, the proper selection of the dyestuff is by far the most effective.

* This material appeared in part in *Sen-i Gakkaishi*, **17**, 916 (1961).

The influence of heat setting on dye absorption is attributed mainly to the change in dye diffusion depending upon the molecular packing in the amorphous regions of the fiber. It is therefore assumed that molecular weight, shape, size, and properties of polar groups in the dyestuff would affect dye diffusion. A comprehensive study of these problems has not previously been reported. In this report the sensitivity of various dyestuffs for irregular heat-setting is described, and these results facilitate the selection of suitable dyestuffs for commercial dyeing processes.

EFFECT OF TENSION, NUMBER OF TWISTS AND HEAT SETTING TEMPERATURE ON DYEING AND HEAT SETTING PROPERTIES OF NYLON 6 FIBER

Experimental

Nylon 6 filaments were rewound from a pirn to a bobbin and were wound up on an aluminum cylinder after being given various numbers of twists by an Italian twister machine under various tensions. The filaments were then subjected to steam-setting by a steam-setting machine. The degree of fixing, density, and dye uptake were estimated after untwisting had been carried out.

Materials. Bulky yarn was prepared from nylon 6 filaments (100 den./24 fil.) under the following conditions: number of twists were 0, 500, 1000, 1500, 2000, 2500, 3000 or 3500 twists/m; the tension in the twisting process was 0.13, 0.19, or 1 0.21 g./den., depending on the weight of the flyer; the rate of spindle rotation was 9700 rpm; steam setting was carried out at 110, 113, 115, 120, 122, 125, 130, or 135°C. for 20 min.

Measurements of Degree of Twist Fixed by Heat Setting. After the twist of the filament had been released completely by keeping the filament free under a tension of 0.1 g./den., the remaining twist was measured by a twist count meter.

Density Measurement. Density was measured by means of a density gradient column composed of benzene and carbon tetrachloride as described previously[1].

Measurement of Amount of Dye Absorbed. Dyeing was carried out with two dyestuffs, under conditions which accurately delineate the physical character of the nylon filaments: (*1*) Supranol Cyanine R 5%, 60°C., 400 min., bath ratio 1:20, pH 5.5; (*2*) Solar Cyanine 5R, 0.65%, 80°C., 150 min., bath ratio 1:250, pH 4. The amount of dye absorbed on the fiber was measured photocolorimetrically on a sample dissolved in *m*-cresol.

Results and Discussion

Figures 1 and 2 show the influence of number of twists upon the force of the twist shrinkage and the degree of the shrinkage on nylon 6 filaments under conditions of constant length and constant tension, respectively. In commercial bulky yarn production, the filament is twisted under a

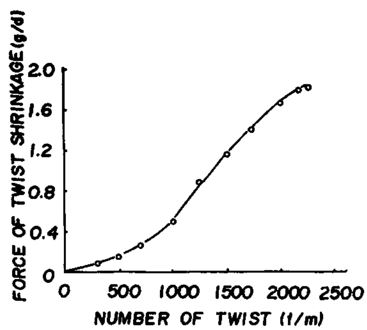

Fig. 1. Relation between force of twist shrinkage and number of twists.

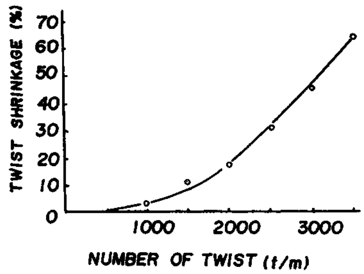

Fig. 2. Relation between degree of twist shrinkage and number of twists.

constant tension which is controlled by the weight of the flyer, so that the filament is allowed to shrink freely.

Figure 1 shows that the force of twist shrinkage increases rapidly at about 1000 twists/m. When the number of twists increases to 2200 twists/m., at which level the force of twist shrinkage is 1.8 g./den., the yarn is broken.

The relation between heat setting temperature and the degree of fixing is shown in Figure 3. The degree of fixing is defined by eq. (1):

Degree of fixing =

$$\frac{\text{Remaining number of twists after heat setting}}{\text{Number of twists before heat setting}} \times 100 \quad (1)$$

In the case of unset fiber, only about 30% of the number of twists is fixed, but in the fibers subjected to steam setting, more than 70% of the number of twists is fixed at above 100°C. The degree of fixing increases gradually at above 100°C. and reaches 98% at 135°C.

The relation between the number of twists and the degree of fixing is shown in Figure 4; a maximum in degree of fixing is found at about 2000 twists/m. Therefore 2000 twists/m. is a suitable number of twists for use in processing bulky yarn from the viewpoint of the degree of fixing.

The influence of the number of twists and temperature on dye absorption

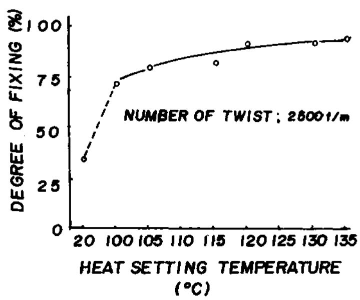

Fig. 3. Relation between degree of fixing and heat setting temperature.

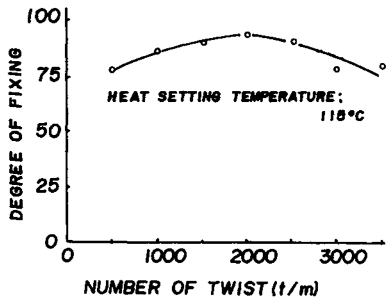

Fig. 4. Relation between degree of fixing and number of twists.

is shown in Figures 5 and 6, respectively. There are no appreciable changes on dye absorption in the range of 0–3500 twists/m., but the amount of dye absorbed increases markedly with increasing of heat setting temperature, especially at more than 115°C.

However, in commercial yarn processing the tension varies appreciably, even with the same flyer, owing to the difference of the degree of defacement and resistance in taking up from a pirn. Therefore it is important to know

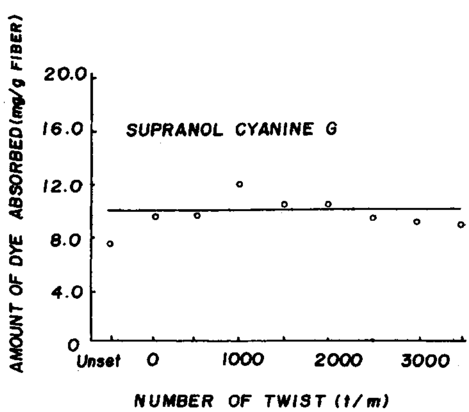

Fig. 5. Relation between amount of dye absorbed and number of twists.

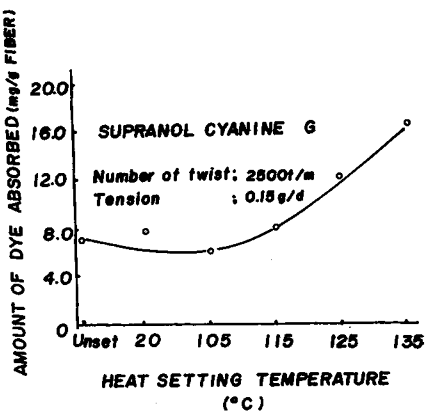

Fig. 6. Relation between amount of dye absorbed and heat setting temperature.

the relation between the variation of tension and heat setting temperature. Results[12] are shown in Figures 7 and 8 for a study made with Solar Cyanine 5R. The amount of dye absorbed increases with heat setting temperature as described above, but decreases appreciably with increasing tension.

These results indicate that 113–116°C. and 0.13–0.19 g./den. are the optimal ranges of heat setting temperature and tension, respectively, because the variation of dye absorption is minimal.

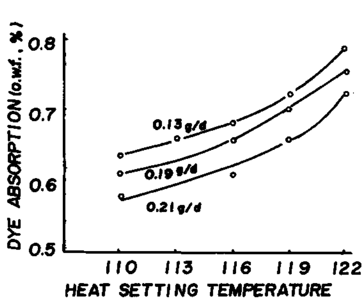

Fig. 7. Relation between dye absorption and heat-setting temperature.

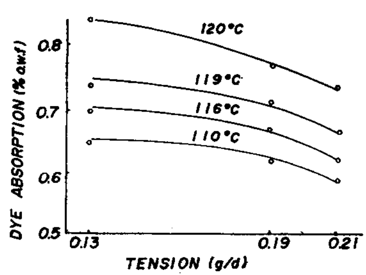

Fig. 8. Relation between dye absorption and tension.

TABLE I
Fastness and Covering Properties of Acid Dyestuffs

Dyestuffs	Light-fastness	Wash-fastness	Covering[a] property
Polar Yellow R	4	3–4	5
Xylene Fast Yellow RPN	5	4	5
Carbolan Yellow 3GS	5	4–5	5
Sun Fast Yellow GGL	3–4	3–4	5
Suminol Yellow MR	5	5	5
Sun Fast Orange GL	3	3–4	4
Xylene Fast Orange P	4	4	4
Sumitomo Nylon Brilliant Orange SHL	3–4	5	5
Polar Orange GS	4	4–5	4–5
Supranol Brilliant Red 3BW	3–4	3–4	4
Polar Red GRS	4	3–4	4
Supramine Red B	4–5	4	5
Supranol Fast Red B conc.	4–5	4	5
Dupont Milling Red SWB	4	4–5	4–5
Supracen Violet 3B	3	4	4
Sumitomo Nylon Violet 2BL	3–4	3–4	4
Telon Fast Violet EF	3–4	4	4
Polar Brilliant Violet BL	4–5	4	4
Supramine Bordeaux B	3–4	3	4
Tetramine Fast Bordeaux SL	3	3–4	3
Telon Fast Bordeaux O	4–5	4	4–5
Polar Red Brown V	4	3–4	4
Sumitomo Nylon Brown G	2	3–4	4
Supranol Fast Brown 5R	4	4	4
Coomassie Fast Brown RS	4–5	4	4–5
Telon Brown GRC	3	3	3
Tetramine Fast Brown BL	1–2	3–4	4
Alizarine Light Blue FG	4	4	4
Erio Anthracene Brilliant Blue 2GL	3–4	4	5
Xylene Fast Blue FF	4	4	4
Basolan Dark Blue R	3–4	4	4
Alizarine Light Blue FF	4	4	4
Dupont Anthraquinone Blue SWF	4	4	5
Xylene Fast Blue PR	4	2–3	4
Erio Glaucine FL	1	4	4
Coomassie Green T	3	3–4	3–4
Acilan Black Green B	3–4	4	4
Carbolan Green G	4	4	3
Sumitomo Nylon Green SW	2	3–4	3
Tetramine Fast Light Green BBL	1–2	3–4	3

[a] Covering property[13] is defined as follows: 5, very good; 4, good; 3, poor; 2, fairly poor; 1, very poor.

The influence of twist on the density is shown in Figure 9. The number of twists as well as tension has a very small effect on the degree of crystallinity. The density increases markedly with increase of heat setting temperature, as observed in Figure 10, which is in excellent agreement with the previous results.[1]

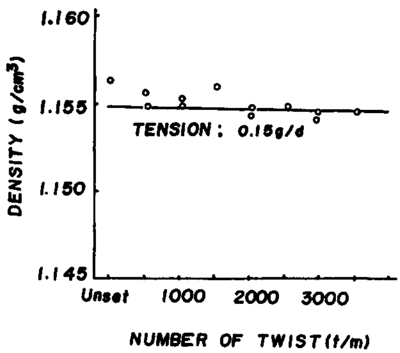

Fig. 9. Relation between density and number of twists.

TABLE II
Fastness and Covering Properties of Disperse Dyestuffs

Dyestuffs	Light-fastness	Wash-fastness	Covering property
Artisil Yellow RG	5	4	4–5
Perliton Yellow G	5	4	5
Cibacet Yellow GN	4	4	4–5
Amacel Yellow GW	5	4	5
Easton Yellow GN	3	3	5
Artisil Orange RFL	3–4	4	5
Diacelliton Fast Orange RF	4	3	4–5
Dispersol Fast Orange B	5	4	4
Easton Orange R	3–4	4	5
Perliton Brilliant Pink R	3–4	4	5
Amacel Scarlet III	5	4	5
Duranol Red GN	5	4	5
Celliton Fast Red GG	5	4	5
Easton Scarlet BG	4	4	5
Artisil Direct Violet RP	5	4	5
Diacelliton Fast Violet BF	5	3	5
Cibacet Violet RB	5	4	5
Perliton Brown G	3–4	3–4	5
Celliton Fast Brown 3R	3	4	5
Perliton Brilliant Blue B	3–4	4	5
Easton Blue BNN	4	3–4	5
Duranol Brilliant Blue CB	4	4	5
Celliton Fast Blue FFB	4	4	4
Celliton Fast Blue Green B	3–4	4	5
Perliton Brilliant Green 3G	4	4	5
Celliton Fast Green FFG	4	4	5
Diacelliton Fast Green BF	5	4	5
Perliton Grey N	3–4	3–4	5
Multamine Black B	5	5	4

Temperature, number of twists, and tension are thus found to be very important factors in the processing of Helanca type bulky yarn. The twist influences the amount of dye absorption and the degree of crystallinity only

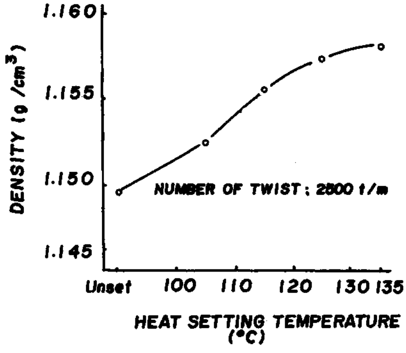

Fig. 10. Relation between density and heat setting temperature.

very slightly but has a pronounced effect on the bulkness, shrink character, and hand of bulky yarn. An increase in tension does not appreciably influence the density, but does decrease the amount of dye absorption. Heat setting temperature markedly increases the degree of crystallinity. These results agree completely with the results of our previous studies.[1–12]

TABLE III. Fastness and Covering Properties of Premetallized Dyestuffs

Dyestuffs	Light-fastness	Wash-fastness	Covering property
Palatine Fast Yellow ELN	5	5	4–5
Palatine Fast Yellow GRN	5	3–4	4–5
Neopalatine Yellow G	5	4	4–5
Neopalatine Orange GR	4	4	3–4
Neopalatine Red GG	5	5	4
Palatine Fast Red BEN	5	4	4–5
Palatine Fast Red LBN	5	1	4–5
Palatine Fast Bordeaux BN	4	5	4
Palatine Fast Brown GGN	5	4	4
Palatine Fast Blue BN	4–5	3	4
Palatine Fast Navy Blue RRN	4	3–4	4–5
Palatine Fast Dark Blue GN	3–4	3	4
Palatine Fast Dark Green BN	5	3	4–5
Palatine Fast Grey GLN	5	4	4–5
Aizen Opal Black NG	5	2–3	3–4
Palatine Fast Black WAN h/c.	5	4	4
Floslan Yellow GRL	5	4–5	4
Carbolan Brilliant Yellow 3GL			4–5
Irgalan Yellow 2RL	5	3	4–5
Lanasyn Brilliant Yellow 5GL	5	3	4–5
Floslan Orange RL	5	2	4–5
Irgalan Orange RL	4	3–4	4–5
Lanasyn Orange RLN	5	3	4
Cibalan Brilliant Scarlet RL	5	2–3	3–4
Lanasyn Scarlet GL	4–5	4	3–4
Floslan Red 3GL	4	3–4	4–5

(continued)

TABLE III (*continued*)

Dyestuffs	Light-fastness	Wash-fastness	Covering property
Cibalin Red 2GL	5	3	4
Floslan Violet BL	5	2	4
Cibalan Violet RL	5	1–2	3–4
Lanasyn Dark Violet RL	5	2	3
Irgalan Violet 5RL	5	2	3
Floslan Bordeaux RL	5	4	2–3
Cibalan Bordeaux RL	5	4–5	3–4
Irgalan Bordeaux 2BL	5	4	4
Lanasyn Bordeaux RL	5	2–3	2–3
Cibalan Brown BL	4–5	2–3	2–3
Cibalan Brown VRL	5	2–3	3
Floslan Brown GL	5	5	4
Irgalan Brown 2RL	5	3–4	4–5
Lanasyn Brown GRL	4	2	3
Floslan Blue BL	5	3	3
Cibalan Blue BL	5	3	3–4
Cibalan Navy Blue RL	5	4	3
Irgalan Blue RL	5	3–4	3
Irgalan Navy Blue 5RL	5	3	4
Lanasyn Brilliant Blue GL	5	1–2	3
Floslan Olive Green GL	5	3	3–4
Cibalan Olive BL	5	4	3
Irgalan Brilliant Green BL	5	4	4
Lanasyn Brilliant Green BL	4	5	3
Cibalan Grey 2GL	4–5	3–4	3
Irgalan Grey RL	5	3	2–3
Floslan Grey GL	5	2	4–5
Lanasyn Grey BL	5	4–5	4
Cibalan Black BGL	5	2	4
Irgalan Black RBL	5	2	2–3

RELATION BETWEEN STEAM SETTING TEMPERATURE AND SENSITIVITY OF DYESTUFFS FOR HEAT SETTING IRREGULARITY

Peters[13] has reported on the relation between the covering property of dyestuffs and the change of physical structure produced in the processing of nylon, but no study on the covering property of dyestuffs has hitherto been made for yarns showing irregularities caused by uneven heat setting. The conventional method for selection of dyestuff is described below.

Experimental

Covering Test of Dyestuffs Used for Irregular Heat Setting. In the processing of bulky (Helanca type) nylon yarn, the heat setting temperature was varied in the three stages at 110, 115, and 120°C. The yarns obtained were knitted side by side to hosiery by a circular knitting machine. A piece of this knit fabric which contained the three kind of yarn was used as a sample.

The sample was dyed with acid dyestuffs, disperse dyestuffs, and premetallized dyestuffs which were suitable for nylon dyeing. The dyebath conditions were as follows: for acid dyestuffs, dyestuff 1.5% owf, pH 3–4; for disperse dyestuffs, dyestuff 0.5% owf, pH 6–7; for premetallized dyestuffs, dyestuff 1.5% owf, dispersing agent 0.4 g./l.; ammonium acetate 1% owf for 2:1 type dyestuff, formic acid 3% owf for 1:1 type dyestuff. In all cases the bath ratio was 1:50, the temperature was 100°C., and the dyeing time was 60 min.

Estimation was carried out by comparing the color difference among three dyed bulky yarns with the greyscale, defined in JIS L-1004. In this case grade 1 indicates the most marked color difference and grade 5 indicates no difference of color.

Fastness Measurements. The sample was prepared by knitting commercial bulky nylon yarn to hosiery. Dyeing with acid dyestuffs was carried out under the following conditions: for dyeing, dyestuff 1.5 or 0.5% owf, pH 3–4; bath ratio 1:50, temperature 100°C., time 60 min.; for tannin treatment, tannic acid 3%, acetic acid 3%, antimonyl potassium tartarate 2%, bath ratio 1:50, temperature 90°C., time 20 min.; for steaming, steaming temperature 120°C., time 1 min. Dyeing with disperse dyestuffs was carried out under the conditions: dyestuff 0.5%, dispersing agent 1 g./l., pH 6–7, bath ratio 1:50, temperature 100°C., time 60 min. For premetallized dyestuffs, dyeing conditions were: dyestuff 1.5%, dispersing agent 0.5 g./l., pH 6–7 (2:1 type dyestuff), pH 2.5–3.5 (1:1 type dyestuff), bath ratio 1:50, temperature 100°C., time 60 min.

In evaluation of lightfastness, samples dyed with 0.5% of various dyestuffs were exposed to light of a Fade-O-Meter under the following conditions: arc electric current 15–17 amp., voltage 125–130 v., temperature 40°C., humidity 50% R.H., exposure period 20 hr. Color changes were classified from grade 1 to 5 on comparison of the materials with the greyscale for fading.

In determining washfastness, the samples dyed with 1.5% of various dyestuffs were sewn to cotton and nylon cloth covering the dyeings, and were stirred for 20 min. in a 5 g./l. solution of soap at 60°C. (bath ratio 1:50), rinsed, dried, and evaluated by comparing the contamination of white patches and color change of the dyeing with greyscale.

Results and Discussion

Results are shown in Tables I–III. These tables indicate that the disperse dyestuffs in general have the best covering property for heat setting irregularity. On the other hand most premetallized dyestuffs are very sensitive to irregularity. The covering power of 2:1 type premetallized dyestuffs is the lowest, so the premetallized dyestuffs are not suitable for commercial dyeing.

In practice, dyestuffs which give more than grade 4 on the for covering property, lightfastness and washfastness are recommended.

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Résumé

Les effets de la tension, du nombre de torsions et de la température de traitement à chaud sur les propriétés de coloration et sur le degré de cristallinité du nylon sont examinés. La quantité de colorant adsorbé sur le nylon tendu diminue avec la tension, mais n'est pas affectée par le nombre de torsions, tandis que leur densité n'est pas affectée par les facteurs ci-dessus. Le degré de torsion, de cristallinité et la quantité de colorant adsorbé augmente avec la température de traitement à chaud. Les facteurs tels que la température de traitement à chaud et la tension sont les causes principales de coloration inégale. En vue de développer la coloration en une application industrielle, les colorants sont classifiés selon leurs sensibilité vis-à-vis du nylon 6 de tension inégale.

Zusammenfassung

Der Einfluss der Spannung, der Twistzahl und der Hitzebehandlungstemperatur auf die Färbeeigenschaften und den Kristallinitätsgrad von Nylon wird untersucht. Die Menge des an gerecktes Nylon adsorbierten Farbstoffes nimmt mit der Spannung ab, wird aber von der Twistzahl nicht beeinflusst; ihre Dichte wird durch obige Faktoren nicht beeinflusst. Der Grad der Twistfixierung, die Kristallinität und die Menge des adsorbierten Farbstoffes nehmen mit der Behandlungstemperatur zu. Faktoren wie Behandlungstemperatur und die Spannung sind die Hauptursache für eine ungleiche Anfärbung. Zum Zwecke der Erreichung einer gleichmässigen Anfärbung bei der industriellen Anwendung werden die Farbstoffe nach ihrer Empfindlichkeit gegen ungleichmässig gerecktes Nylon 6 in Wirkwaren klassifiziert.

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