

Modification of Nylon 66 with Diisocyanates and Diacid Chlorides. I. Chemical Reaction*

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Synopsis

Four α,ω -diisocyanates (tetramethylene, hexamethylene, decamethylene, and eicosane) and six α,ω -diacid chlorides (succinyl, adipyl, pimeloyl, sebacyl, dodecane, and docosane) were allowed to react with two types of nylon 66 yarn made from polymers of 14,800 and 21,000 number-average molecular weights. The higher molecular weight yarn had a more ordered structure initially, as indicated by density measurements. Both diisocyanates and diacid chlorides crosslinked the yarn as shown by insolubility in 90% formic acid. Isocyanates were more efficient crosslinking reagents than diacid chlorides. Diacid chlorides caused simultaneous degradation of the yarn. The overall rate of reaction of the reagents with the yarn responded to temperature, reagent concentration, and time as expected for low molecular weight materials bearing the same functional groups. Catalysts had only a small effect on the rate of reaction. The reaction occurred in the less ordered regions of the yarn, as indicated by (1) a faster rate of reaction with the less ordered yarn, (2) the accelerating effect of swelling solvents on the rate, and (3) the reduction in extent of reaction with increase in molecular size of the reagent even though the reaction was not limited by diffusion. Both diisocyanates and diacid chlorides reduced the dyeability of yarn with acid dyes. Diacid chlorides increased the dyeability of yarn with basic dyes and diisocyanates decreased this dyeability.

INTRODUCTION

Although the reaction of nylon 66 with difunctional reagents was mentioned in the patent literature over twenty years ago,^{1,2} and then again more recently,³⁻⁵ no study of the nature of the crosslinking reaction and its effect on the physical properties of the fibers has been published. Crosslinking of nylon 66 by radiation usually leads to a degradation of properties.^{6,7}

The purpose of this article is to describe the nature of the reaction of four α,ω -diisocyanates and six α,ω -diacid chlorides of varying aliphatic chain lengths with two types of nylon 66 yarns. Specifically, information about the locus of the reaction and similarities and differences when compared to reaction between low molecular weight species is discussed. The yarn designated as yarn A has a less ordered structure in the solid state than does yarn B. The degree of order in a yarn is indicated by measurements such as density, refraction of x-rays, and rate of uptake of dyes.

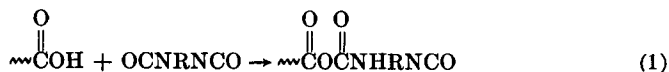
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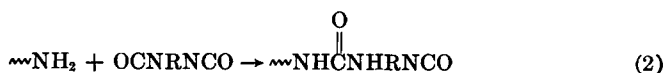
Also, yarn B is higher in molecular weight than yarn A. The effect of the chemical treatment on the physical properties of the yarns is the subject of a following article.

Diisocyanates may be expected to react in the simple manner shown in eqs. (1)–(3) with functional groups in the nylon 66 chain.

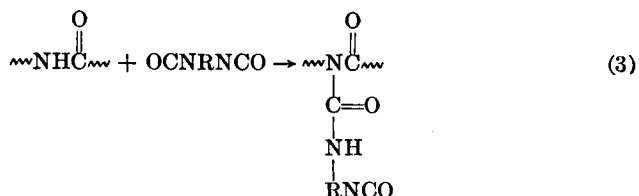
Acid endgroups:



Amine endgroups:



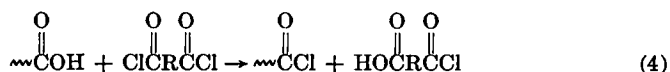
Amide groups:



where OCNRNCO represents a low molecular weight diisocyanate and the wavy bond represents a polymer chain.

The rate of reaction decreases in the order (2) >> (1) > (3).^{8,9} Crosslinking will result only by the further reaction of the free isocyanate group located on the product of reaction (3) with an amide group on another chain. If the free isocyanate groups on the products of reactions (1) and (2) react with other polymer endgroups, lengthening of the polymer chain will result. If these same free isocyanate groups react with amide groups on some other polymer chain, branched polymer molecules will be formed. A branched polymer molecule will result also from the reaction of the free isocyanate group located on the product of reaction (3) with the endgroup of a polymer chain. Another undesirable reaction of the isocyanate function, if efficient crosslinking is wanted, is reaction with the NH of the carbamates which were formed by reactions (1), (2), and (3).

Acid chlorides react with functional groups on the nylon chain in an analogous manner with a probable rate order of (2) >>> (3) >> (1).^{10,11} In addition, secondary reactions such as acid chloride interchange [eq. (4)] can occur.¹¹



Even monoacid chlorides react with nylon 66 to give a product insoluble in 90% formic acid, as determined some years ago in this laboratory and as reported recently.¹²

EXPERIMENTAL

Raw Materials

Hexamethylene diisocyanate (The Carwin Company) was distilled (b.p. 109°C./4 mm.) in an oxygen- and water-free atmosphere in glass apparatus. Tetramethylene diisocyanate was prepared in 40% yield starting from adipyl chloride via the azide and the Curtius rearrangement (b.p. 112–113°C./20 mm.; calcd. N 20.0%, found 19.54%). Decamethylene diisocyanate was prepared in 42% yield in a similar manner starting from dodecanedioyl chloride, and in 89% yield by allowing the corresponding diamine hydrochloride to react with phosgene (b.p. 129°C./0.5 mm.; calcd. N 12.5%, found 11.85%). Eicosane diisocyanate was prepared in 95% yield via the diamine reaction with phosgene (m.p. 42.5–43°C.; calcd. N 7.7%, found 7.77%).

Diacid chlorides used included succinyl (Eastman White Label, b.p. 64°C./6 mm.), adipyl (Eastman White Label, b.p. 109°C./4 mm.), pimeloyl, sebacyl (Eastman White Label, b.p. 105°C./1 mm.), 1,12-dodecane, and 1,22-docosane. The boiling points noted above were recorded during redistillation of the materials for further purification. The other diacid chlorides were prepared by reaction of thionyl chloride with the dicarboxylic acids using a trace of dimethylformamide as a catalyst. Pimeloyl was obtained in 86% yield, (b.p. 103°C./2 mm.; calcd. Cl 36.05%, found 36.20%); 1,12-dodecanedioyl was obtained in 66% yield, distilled at 0.0001 mm. Hg. (calcd. Cl 26.6%, found 26.03%); 1,22-docosanedioyl was obtained in 55% yield, purified by seven recrystallizations from *n*-hexane (m.p. 56.5°C.; calcd. Cl 17.45%, found 16.86%).

Solvents, C.P. grade except as noted, were dried over CaH₂ and further purified by distillation. The propylene carbonate (Eastman Org. Chemicals, Cat. No. P7050) was handled in the same manner and the fraction boiling at 77–79°C./0.14 mm. was used. Triethylenediamine (Dabco) was obtained from K&K Laboratories (Lot No. 30604F).

Preparation and purification of the raw materials were carried out under argon that had been dried over molecular sieves. Connections to the apparatus were glass and these, together with the apparatus itself, were flamed dry in a stream of argon prior to reaction, distillation, or recrystallization.

Scarlet 4RA (Dyestuff Division of the General Aniline Corporation) is a red acid dye. Sevron Blue 2G (Du Pont) is a blue basic dye.

Yarns

Nylon 66, yarn A (Monsanto Company) had NH₂ content of 70×10^{-6} eq./g., COOH content of 96×10^{-6} eq./g., 0.028% TiO₂, total denier of 70, 34 filaments per bundle. Nylon 66, yarn B (Monsanto Company) had NH₂ content of 26×10^{-6} eq./g.; COOH content of 71×10^{-6} eq./g., 0.032% TiO₂, total denier of 840, 140 filaments per bundle. Yarn B had a draw ratio of 4.5–4.9 and a density of 1.149 g./cc.; yarn A had a draw ratio of 2.6–2.8 and a density of 1.144 g./cc.

Reaction

Approximately 1 g. of nylon 66 yarn was wound loosely onto a glass core. Textile finish was removed by extraction in a Soxhlet with petroleum ether (30–60°C. fraction) for 2 hr. After drying 24 hr. at 100°C. in a vacuum oven, dry N₂ was admitted. The fiber sample now enclosed in a weighing bottle was handled hereafter in dry, inert atmospheres. Reaction with diisocyanate or diacid chloride was carried out in solvent under the conditions of time, temperature, concentration of reagent, and catalyst as indicated in the Results. Precautions for the complete exclusion of moisture and air were taken as described for the handling of raw materials. Also, liquids were transferred in syringes which had been baked at 130°C. and cooled in dry N₂. After reaction, the treated yarn was rinsed with fresh toluene for several hours at room temperature to remove most of the excess reagent. Residual unreacted crosslinking agent was extracted further by treating with fresh toluene for 24 hr. at 100°C. The extracted yarn sample was dried for 24 hr. at 100°C. under vacuum and reweighed. The extent of reaction was found by comparing the weight change of the sample with a control treated in exactly the same manner but without crosslinking reagent.

Analytical Procedures

Solubility. A 50-ml. test tube containing 30 ml. of filtered 90% formic acid and a weighed sample of nylon yarn (ca. 25 mg.) was kept at 25°C. for 2 hr. with shaking of the tube every 30 min. The contents of the tube were filtered with suction through weighed filter paper (Whatman No. 1, 4.25 cm. diameter) which had been equilibrated with a standard atmosphere. The tube was rinsed once with 10 ml. of formic acid and the rinsing was filtered. The filter paper was dried at 25°C./760 mm. for 2 hr. and then in a vacuum oven for 24 hr. Finally, the sample and paper were equilibrated with the standard atmosphere and reweighed. Controls were run to determine the weight gain of the filter paper when treated with formic acid alone.

Dye Absorption. Dyeing was carried out in 8-in. Pyrex, crown-top pressure tubes which were sealed with polyethylene-lined caps. The tubes were tumbled for 2 hr. in a steam chamber at 100°C. All dyeing tests were to "saturation" (i.e., equilibrium conditions so that no further uptake of dye occurred when the time of dyeing was extended). Acid dye tests were conducted on "finish-free" yarns with 3% (on weight of fiber) of Scarlet 4RA in solution at a 40:1 liquor-to-fiber weight ratio. The solution also contained 1.2% formic acid to yield a pH of 3.3. The amount of dye absorbed was determined by measuring the absorbence on the fresh and exhausted dye solution using a Cary recording spectrophotometer at 510 μ . Basic dye saturation tests were carried out in a similar manner using 10% (on weight of fiber) of Sevron Blue 2G dye in solution at a 40:1 liquor-to-fiber weight ratio. This dye solution also contained 5.0% ammonium

acetate to buffer the pH at 6.7. Absorbance measurements were made at 638 μ .

RESULTS

Reaction

Diisocyanates react with nylon 66 yarn in much the same way as they do with low molecular weight species. Thus, the rate of reaction varies directly with the temperature, the time of reaction, and the reagent concentration (for typical data cf. Table I). Catalysis with amines has a

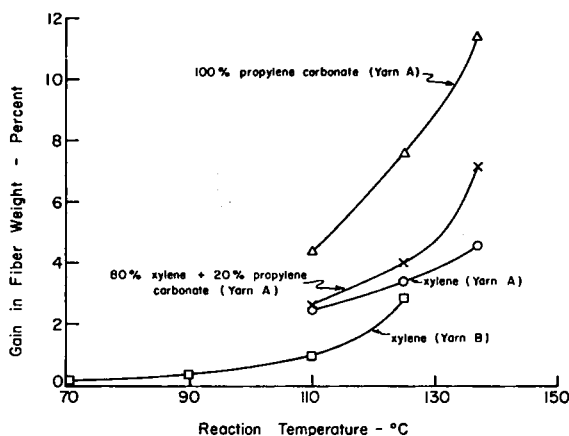


Fig. 1. Reaction of hexamethylene diisocyanate with nylon 66 yarn (0.2% triethylenediamine catalyst based on fiber weight; 0.525 mole/l. of diisocyanate; reaction time 24 hr.; reaction medium, temperature, and type yarn as indicated).

minor effect on extent of the reaction and only at elevated temperatures. Solvents, especially those which swell the nylon 66 yarn, increase the extent of reaction (Fig. 1).

The lower density, less ordered yarn A reacts more readily than the more ordered yarn B (Fig. 1). Other conditions being equal, the higher molecular weight difunctional reagents react less readily than do lower molecular weight species even when considered on a weight basis (Table II). However, by using forcing conditions (i.e., high temperatures and long times), any degree of reaction can be achieved for a given molecular weight diisocyanate. The uniformity of dyed cross-sections for all except those reactions which were carried out below 70°C. suggests that diffusion is not the factor which controls the degree of reaction.

Acid chlorides crosslink the yarn and degrade it simultaneously. If the treatment is severe enough, net weight losses are found even though the fiber becomes completely insoluble in 90% formic acid. Acid chlorides, like the isocyanates, react more readily with yarn which is less ordered (Table III).

TABLE I
Effect of Some Reaction Conditions on the Reaction of Diisocyanates with Nylon 66

Type of diisocyanate	Type of yarn	Diisocyanate concn., moles/l.	Triethylene-diamine catalyst, wt.-% based on fiber)	Solvent	Reaction temperature, °C.	Reaction time, hr.	Gain in fiber weight, %
Hexamethylene	Yarn B	0.525	0	Toluene	25	24	0
Hexamethylene	"	0.525	0.2	Toluene	25	24	0
Hexamethylene	"	0.525	0	Toluene	71	24	0.2
Hexamethylene	"	0.525	0.2	Toluene	71	24	0.2
Hexamethylene	"	0.525	0	Toluene	90	24	0.5
Hexamethylene	"	0.525	0.2	Toluene	90	24	0.5
Hexamethylene	"	0.525	0	Toluene	110	24	0.8
Hexamethylene	"	0.525	0.2	Toluene	110	24	1.1
Decamethylene	Yarn A	0.525	0.2	Toluene	110	24	0.6
Decamethylene	"	0.525	0.2	Toluene	110	66	1.4
Decamethylene	"	0.525	0.2	Xylene	137	24	1.9
Decamethylene	"	0.787	0.2	Xylene	137	24	2.5
Decamethylene	"	0.787	0.2	Xylene	137	48	3.2
1,20-Eicosane	"	0.525	0	Propylene carbonate	137	24	5.5
1,20-Eicosane	"	0.525	0	Propylene carbonate	137	48	5.9

TABLE II
Effect of Diisocyanate Chain Length
on the Extent of Reaction with Nylon 66

Type of diisocyanate	Type of yarn	Reaction time, hr.	Triethylene-diamine catalyst, wt.-% based on fiber	Gain in fiber weight, %
Hexamethylene ^a	A	24	0.2	1.4
Decamethylene ^a	A	24	0.2	0.6
1,20-Eicosane ^a	A	24	0.2	0.1
Tetramethylene ^b	B	48	0	8.2
Hexamethylene ^b	B	48	0.2	5.5
Decamethylene ^b	B	48	0.2	2.1
1,20-Eicosane ^b	B	168	0.2	0.6

^a 0.525 mole/l.; in toluene; 110°C.

^b 0.525 mole/l.; in xylene; 137°C.

TABLE III
Ratio of the Reactivity of Difunctional Reagents with Yarns A and B^a

Reagent	Solvent	Triethylene-diamine catalyst, wt.-% based on fiber)	R_1/R_2^b
Tetramethylene diisocyanate	Toluene	None	1.37
Hexamethylene diisocyanate	Toluene	None	1.50
Hexamethylene diisocyanate	Toluene	0.2	1.27
Hexamethylene diisocyanate	Propylene carbonate	0.2	2.75
Hexamethylene diisocyanate	Xylene	0.2	1.62
Decamethylene diisocyanate	Xylene	0.2	1.54
Pimeloyl chloride	Toluene	None	3.69
Sebacyl chloride	Toluene	None	1.10

^a For reactions in toluene and propylene carbonate: 0.525 mole/l. of diisocyanate, 24 hr. at 100°C.; For reactions in xylene: 0.525 mole/l. of diisocyanate, 24 hr. at 125°C.

^b R_1 is the per cent gain in weight of yarn A; R_2 is the per cent gain in weight of yarn B.

Crosslinking

Reactions with both diisocyanates and diacid chlorides lead to insolubility in 90% formic acid. The diisocyanates are more efficient crosslinkers than the diacid chlorides, since less weight of a given chain length reagent

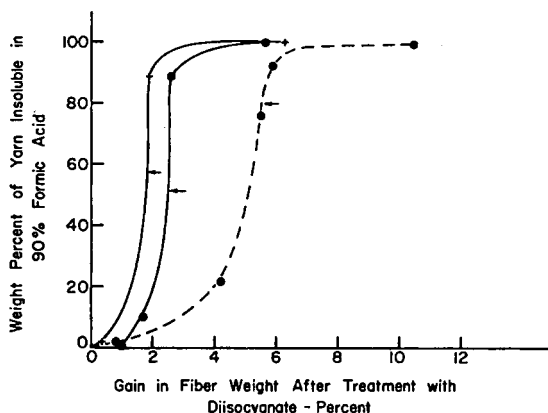


Fig. 2. Crosslinking efficiency of diisocyanates in reaction with nylon 66 yarn: (—) yarn treated with tetramethylene diisocyanate; (---) yarn treated with 1,20-eicosane diisocyanate; (●) yarn A; (+) yarn B. (The arrows denote the gel point calculated according to Flory.¹³)

is needed to achieve equal degrees of insolubility, even though the acid chlorides are favored by the weight loss due to polymer degradation (Table IV). Gelation occurs a little sooner than would be predicted from

TABLE IV
Crosslinking Efficiency of Diisocyanates and Diacid Chlorides with Yarn A^a

Reagent	Gain in weight, %	Insolubility in 90% formic acid, %
Tetramethylene diisocyanate	2.6	75
Hexamethylene diisocyanate	1.2	55
Pimeloyl chloride	4.8	65
Sebacyl chloride	2.2	48

^a For all reactions: 0.525 mole/l. of reagent; toluene; 110°C.; 24 hr.; no catalyst.

the theory of Flory¹³ (Fig. 2). For these calculations it is assumed that both acid and amine endgroups react completely before any reaction occurs with the amide groups. Therefore, the total amount of the difunctional reagent which reacts with the yarn is corrected for these "wastage" reactions before the crosslinking density is calculated. The higher molecular weight yarn gels before the lower molecular weight yarn, as expected. The deviation from theory is greater at high temperatures where spurious secondary reactions become more important.

Dyeability

Yarn treated with either diisocyanate or diacid chloride suffers a loss in acid dyeability (Table V). Basic dyeability is greater for acid chloride-

TABLE V
Dyeability of Yarn B

Reagent	Type of dye ^a	Fiber weight gain due to reaction, %	Dyeability, % of the control
Hexamethylene diisocyanate	Acid	0.2	85
Hexamethylene diisocyanate	Acid	2.9	36
Hexamethylene diisocyanate	Basic	0.2	117
Hexamethylene diisocyanate	Basic	2.9	62
Adipyl chloride	Acid	0.06	101
Adipyl chloride	Acid	0.2	60
Adipyl chloride	Acid	1.4	50
Adipyl chloride	Basic	0.06	74
Adipyl chloride	Basic	0.2	116
Adipyl chloride	Basic	1.4	325

^a Acid: Scarlet 4RA; basic: Sevron Blue 2G.

treated yarn at all levels of treatment. Basic dyeability is improved slightly by mild diisocyanate treatment and is worsened by extensive treatment with diisocyanates.

DISCUSSION

The data suggest that the reaction of nylon 66 with diacid chlorides and diisocyanates takes place in the less ordered or amorphous regions of the yarn: (1) the higher reactivity of yarn A as compared to yarn B; (2) the general effect of swelling solvent in extending the degree of reaction and the greater sensitivity of yarn A to the type of solvent; (3) the faster degradation of yarn A in the presence of acid chlorides; and (4) the decrease in reactivity as the molecular size of the reagent increases. Apparently, the accessibility of the sites rather than diffusion is the limiting factor in reaction. Also consistent with this picture is the effect of temperature and concentration of reagent on the extent of reaction. The reaction with nylon 66 of diisocyanates and diacid chlorides is similar in nature to reaction between low molecular species having the same functional groups. However, the interpretation of what controls the reactions differs. Thus, the effect of solvents can be due to their ability to swell the fiber and make sites available as well as to their effect on the chemical reaction rate itself. Isocyanates can even become solvents for nylon 66 at high temperatures.

It is not clear why diisocyanates are more efficient crosslinking agents than diacid chlorides. One reason may be the reactivity of the isocyanate group with the acid endgroup as well as with the amine endgroup of the polymer chain to build up the molecular weight of the base molecules. However, the almost linear increase in basic dyeability with increase in fiber weight as a result of acid chloride treatment does not fit in well with this explanation.

The close agreement between the calculated and experimental weight gain of the fiber at the gel point for diisocyanates is probably fortuitous.

The crystallinity is preserved in the fibers after the crosslinking treatment (see the following paper) so that the reaction is not random as the theory demands.

The improvement in basic dyeability at small weight gains of diisocyanate is unexpected. The rapid preferential reaction of isocyanate with NH_2 endgroups may free acid endgroups which had been tied up in salt form.

CONCLUSIONS

Nylon 66 can be crosslinked by reaction with diisocyanates or diacid chlorides. The weight gain of the yarn is a measure of the degree of crosslinking.

Diisocyanates are more efficient crosslinking agents than diacid chlorides in reaction with nylon 66.

The general effect of time, temperature, and concentration of reagent is the same as for similar reactions between low molecular weight species.

The extent of reaction varies inversely with the size of the crosslinking chain for a given set of reaction conditions.

The less ordered yarn A reacts more readily than the more ordered yarn B.

Reaction occurs in the less ordered regions of the yarns.

Acid chlorides degrade the nylon 66 polymer while simultaneously crosslinking the chains.

The dyeability of the fiber can be modified by reaction with diacid chlorides or diisocyanates.

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Résumé

Deux types de fil de nylon-66 ont été préparés au départ de polymères ayant des poids moléculaires moyens en nombre de 14.800 et 21.000; on a fait réagir avec ces deux types de nylon, quatre α,ω -diisocyanates (de tétraméthylène, d'hexaméthylène, de décaméthylène et d'éicosane) et six α,ω -chlorures de diacide (succinyle, adipyle, pimeloyle, sébacyle, dodécane et docosane). Le fil de poids moléculaire élevé a une structure plus fortement ordonnée initialement, comme le montrent les mesures de densité. Aussi bien, les diisocyanates que les chlorures de diacides provoquent le pontage du fil comme le montre son insolubilité dans l'acide formique à 90%. Les isocyanates sont plus efficaces comme agent portant que les chlorures de diacide. Les chlorures de diacide causent simultanément une dégradation du fil. La vitesse globale de réaction des réactifs avec le fil dépend de la température, de la concentration en réactif et de la durée, comme prévu pour des produits de bas poids moléculaire portant les mêmes groupes fonctionnels. Les catalyseurs n'ont qu'un faible effet sur la vitesse de réaction. La réaction se passe dans les régions les moins ordonnées du fil comme cela est indiqué par (1) une plus grande vitesse de réaction avec les fils les moins ordonnés, (2) l'effet accélérant de solvants gonflants sur la vitesse et (3) la réduction du degré d'avancement de la réaction avec une augmentation de la taille moléculaire du réactif bien que la réaction ne soit pas limitée par la diffusion. Aussi bien les diisocyanates que, les chlorures de diacide réduisent la teintabilité du fil par les colorants acides. Les chlorures de diacide augmentent la teintabilité du fil par les colorants basiques et les diisocyanates la diminuent.

Zusammenfassung

Vier α,ω -Diisocyanate (Tetramethylen, Hexamethylen, Dekamethylen und Eicosan) und sechs α,ω -Dicarbonsäurechloride (Succinyl, Adipyl, Pimeloyl, Sebacyl, Dodekan und Dokosan) wurden mit zwei aus Polymeren mit einem Zahlenmittelmolekulargewicht von 14800 und 21000 hergestellten Nylon-66-Garntypen zur Reaktion gebracht. Das höhermolekulare Garn besass anfänglich eine geordnetere Struktur als die Dichtemessungen erkennen liessen. Wie die Unlöslichkeit in 90% iger Ameisensäure zeigte, wird das Garn sowohl durch Diisocyanate als auch durch Dicarbonsäurechloride vernetzt. Isocyanate erwiesen sich als wirksamere Vernetzungsmittel als Dicarbonsäurechloride. Dicarbonsäurechloride verursachten einen gleichzeitigen Abbau des Garnes. Die Bruttoreaktionsgeschwindigkeit der Reagentien mit dem Garn verhielt sich gegen Temperatur, Reagenskonzentration und Zeit so, wie es für niedermolekulare Stoffe mit den gleichen funktionellen Gruppen zu erwarten ist. Katalysatoren besaßen nur einen kleinen Einfluss auf die Reaktionsgeschwindigkeit. Wie (1) die grössere Reaktionsgeschwindigkeit in weniger geordnetem Garn, (2) der beschleunigende Einfluss quellungsfähiger Lösungsmittel auf die Geschwindigkeit und (3) die Reduktion des Reaktionsausmasses mit zunehmender Molekülgrösse des Reagens, obgleich die Reaktion nicht diffusions-begrenzt ist, zeigt, verläuft die Reaktion in den weniger geordneten Bereichen des Garnes. Sowohl Diisocyanate als auch Dicarbonsäurechloride setzten die Anfärbarkeit des Garnes mit sauren Farbstoffen herab. Dicarbonsäurechloride erhöhten die Anfärbarkeit des Garnes mit basischen Farbstoffen und Diisocyanate setzten diese herab.

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