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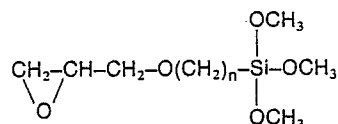
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⑤④ **Process for producing adhesive active polyester yarn and product.**

⑤⑦ An improvement is provided in a process for the production of polyester yarn involving spinning and drawing steps. The improvement comprises exposing the yarn subsequent to the drawing step to ultraviolet radiation, and treating the yarn with a finish composition which comprises water and a silane having the structural formula



wherein $n = 2$ to 5. The yarn is preferably used in tire cords for the construction of pneumatic passenger tires and results in excellent adhesion of tire cord to rubber.

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PROCESS FOR PRODUCING ADHESIVE
ACTIVE POLYESTER YARN AND PRODUCT

BACKGROUND OF THE INVENTION

Field of the Invention

5 The present invention relates to a process
for producing adhesive active polyester yarn.
More particularly, the present invention relates to
a process for producing adhesive active polyester,
preferably polyethylene terephthalate, yarn wherein
10 the yarn is coated with an aqueous emulsion which
contains an epoxy silane, and after drawing, the
yarn is exposed to ultraviolet radiation. The
present invention also relates to the yarn produced
in accordance with the process. The yarn preferably
15 is twisted into tire cords for the construction of
pneumatic passenger tires and results in excellent
adhesion of tire cord to rubber.

DESCRIPTION OF THE PRIOR ART

20 Polyester tire cord requires the application of
an adhesive layer to obtain bonding to the rubber.
Two types of adhesive systems, a single dip and a
double dip adhesive system, have been developed to
meet this need.

25 In the double dip system, polyester cords are
treated with a first dip which is a dispersion of a
phenol-blocked methylene bisphenylene diisocyanate,
an epoxy resin, wetting agents and water. The treated

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cord is cured, then treated with a second dip of resorcinol-formaldehyde-latex and cured again.

5 In the single dip system, the adhesive layer is applied to the polyester yarn in an overfinish subsequent to drawing of the yarn. The polyester yarn is plied into cords which are treated with a resorcinol-formaldehyde latex dip and cured. The need for the blocked diisocyanate dip in cord processing is eliminated by this system. Low
10 carboxyl polyester yarn treated with this system has very good ammonolytic and hydrolytic stability, but has poor adhesion to rubber when made into tire cord. Further, while regular carboxyl polyester yarn shows acceptable adhesion to rubber under
15 normal curing temperatures, a reduction of the curing temperatures has been found to adversely affect adhesion to rubber.

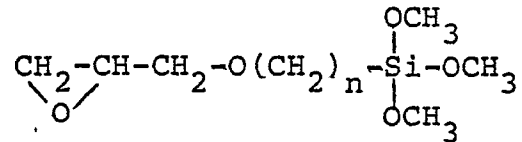
The present invention significantly improves yarn to rubber adhesion of polyester tire cords
20 treated with the single dip system for (a) low carboxyl polyester yarn wherein normal curing temperatures are utilized, and (b) regular carboxyl polyester yarn wherein reduced curing temperatures are utilized.

25 The closest prior art is believed to be U.S. Patents 3,642,518 to Miki et al., 3,672,977 to Dardoufas, 3,730,892 to Marshall et al., and 4,054,634 to Marshall et al., and British Patent Specification 1,228,173 to Jervis et al.

30 SUMMARY OF THE INVENTION

The present invention provides an improvement in a process for the production of polyester yarn involving spinning and drawing steps. The present invention further provides polyester yarn treated in
35 accordance with the process.

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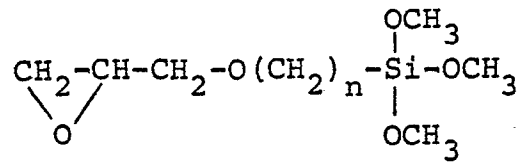
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decaglycerol tetraoleate, about 5 to 12 weight percent of ethoxylated tall oil fatty acid, about 5 to 15 weight percent of sulfonated glycerol trioleate, about 1 to 10 weight percent of
5 ethoxylated alkyl amine, and about 2 to 45 weight percent of the silane. The preferred silane is gamma-glycidoxypropyltrimethoxysilane. The yarn is preferably treated with a sufficient amount of the overfinish composition that (a) about 0.4 to
10 1.0 weight percent based on the weight of the yarn of the non-aqueous portion of the overfinish composition is added, and (b) about 0.05 to 0.2 weight percent based on the weight of the yarn of the silane is added. Alternatively, the overfinish
15 composition can comprise about 7 to 50 weight percent of the silane, about 5 to 20 weight percent of dimethyl polysiloxane emulsion, and the balance water.

The present invention also provides, in a
20 process for the production of polyester yarn involving spinning and drawing steps, the improvement which comprises exposing the yarn subsequent to the drawing step for a time period of at least about 0.3 second to ultraviolet radiation having a wave
25 length between about 200 and 400 nanometers; and treating the yarn with an overfinish composition comprising about 60 to 90 weight percent of water and about 10 to 40 weight percent of an oil portion, the oil portion comprising about 50 to 70 weight
30 percent of hexadecyl stearate, about 3 to 9 weight percent of glycerol monooleate, about 2 to 8 weight percent of decaglycerol tetraoleate, about 5 to 12 weight percent of ethoxylated tall oil fatty acid, about 5 to 15 weight percent of sulfonated
35 glycerol trioleate, about 1 to 10 weight percent of

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10 having about 19 to 30 carboxyl end groups (meq./kg.).

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DESCRIPTION OF THE PREFERRED EMBODIMENT

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The yarns of this invention can be processed by any spin draw process or spinning and separately

drawing process available to the art and the patent and technical literature, using any suitable polyester.

The preferred polyesters are the linear
5 terephthalate polyesters, i.e., polyesters of a glycol containing from 2 to 20 carbon atoms and a dicarboxylic acid component containing at least about 75 percent terephthalic acid. The remainder, if any, of the dicarboxylic acid component may be
10 any suitable dicarboxylic acid such as sebacic acid, adipic acid, isophthalic acid, sulfonyl-4,4'-dibenzoic acid, or 2,8-di-benzofuran-dicarboxylic acid. The glycol may contain more than two carbon atoms in the chain, e.g., diethylene glycol, butylene
15 glycol, decamethylene glycol, and bis-1,4-(hydroxymethyl)cyclohexane. Examples of linear terephthalate polyesters which may be employed include poly(ethylene terephthalate), poly(butylene terephthalate), poly(ethylene terephthalate/5-chloroisophthalate) (85/15),
20 poly(ethylene terephthalate/5-[sodium sulfo]isophthalate) (97/3), poly(cyclohexane-1,4-dimethylene terephthalate), and poly(cyclohexane-1,4-dimethylene terephthalate/hexahydroterephthalate) (75/25).

EXAMPLE 1 (COMPARATIVE)

25 For comparative testing, a polyethylene terephthalate yarn having $11\frac{1}{2}$ carboxyl end groups was prepared substantially in accordance with one procedure described in U.S. Patent 3,672,977 to Dardoufas, hereby incorporated by reference, i.e.,
30 a 1000 denier 192 filament yarn was prepared comprised of polyethylene terephthalate filaments treated with about 0.45 percent based on the weight of the yarn of a liquid spin finish identified as spin finish A in Table I. Drawing performance of
35 the yarn was excellent. Finish oil on the fiber was

about 0.15 to 0.25 percent.

The yarn was passed through a chamber enclosing two 4-tube layers with off-set centers of high intensity lamps of the germicidal type having wave length of about 200 to 300 nanometers, with peak wave length at 253 nanometers. The lamps were approximately 83.8 centimeters (33 inches) in length and can be obtained from the Ultradynamics Corporation, Santa Monica, California. The yarn made six passes through the chamber, each pass being centered at a distance of about 1 centimeter between three of the tubes so that high intensity ultraviolet radiation fell on all sides of the yarn. The lamps were found to provide maximum efficiency at a temperature near 37.8°C. (100°F.). The yarn was exposed to the ultraviolet radiation for a total of 7 seconds.

To this yarn a secondary finish, or overfinish, identified as overfinish A of Table II, was applied at a 3 percent total wet pickup to achieve a total oil on yarn level of about 1.0 percent. The yarn was then twisted into 3-ply cords having 9x9 twists per inch. Each cord was treated with a conventional non-ammoniated resorcinol-formaldehyde-latex dip comprising vinyl pyrridine latex, resorcinol, formaldehyde, sodium hydroxide and water, at about 4.5 percent total solids pickup based on the weight of the cord. The cords were then cured at treating condition 3 of Table III. The treated cords were subjected to the strip adhesion test, results of which are presented in Table IV.

EXAMPLE 2 (COMPARATIVE)

The procedure of Example 1 was repeated with the following changes: The yarn was not exposed to ultraviolet radiation; and the yarn was overfinished

at room temperature with overfinish B of Table II. Results of adhesion testing are presented in Table IV.

EXAMPLES 3-5

5 The procedure of Example 1 was repeated in each of Examples 3 through 5 with the following changes: The yarn was exposed to the ultraviolet radiation for a period of 0.3, 3 and 7 seconds, respectively, in Examples 3 through 5; and the
10 yarn was overfinished at room temperature with overfinish B of Table II. Results of adhesion testing are presented in Table IV.

EXAMPLES 6 and 7

15 The procedure of Example 1 was repeated with the following changes: The yarn was not exposed to ultraviolet radiation in Example 6 while in Example 7, the yarn was exposed to ultraviolet radiation for a period of three seconds; and in
20 both examples, the yarn was overfinished at room temperatures with overfinish C of Table II. Results of adhesion testing are presented in Table IV.

EXAMPLES 8-13

 Polyethylene terephthalate yarn having 24^{+1} carboxyl end groups was prepared in accordance
25 with the procedure of Example 1 in Examples 8 through 13, utilizing the spin finishes, overfinishes, ultraviolet treatment and treating conditions specified in Table IV. Results of adhesion testing are also presented in Table IV.

EXAMPLES 14-19

30 Polyethylene terephthalate yarn having 24^{+1} carboxyl end groups was prepared in accordance with the procedure of Example 1 in Examples 14 through 19, utilizing the spin finishes, overfinishes,
35 ultraviolet treatment and treating conditions

specified in Table IV; however, the step of
exposing the yarn to ultraviolet radiation followed
the application of overfinish to the yarn in
those examples (15, 16, 18 and 19) which included
5 both of these steps. Results of adhesion testing
are presented in Table IV.

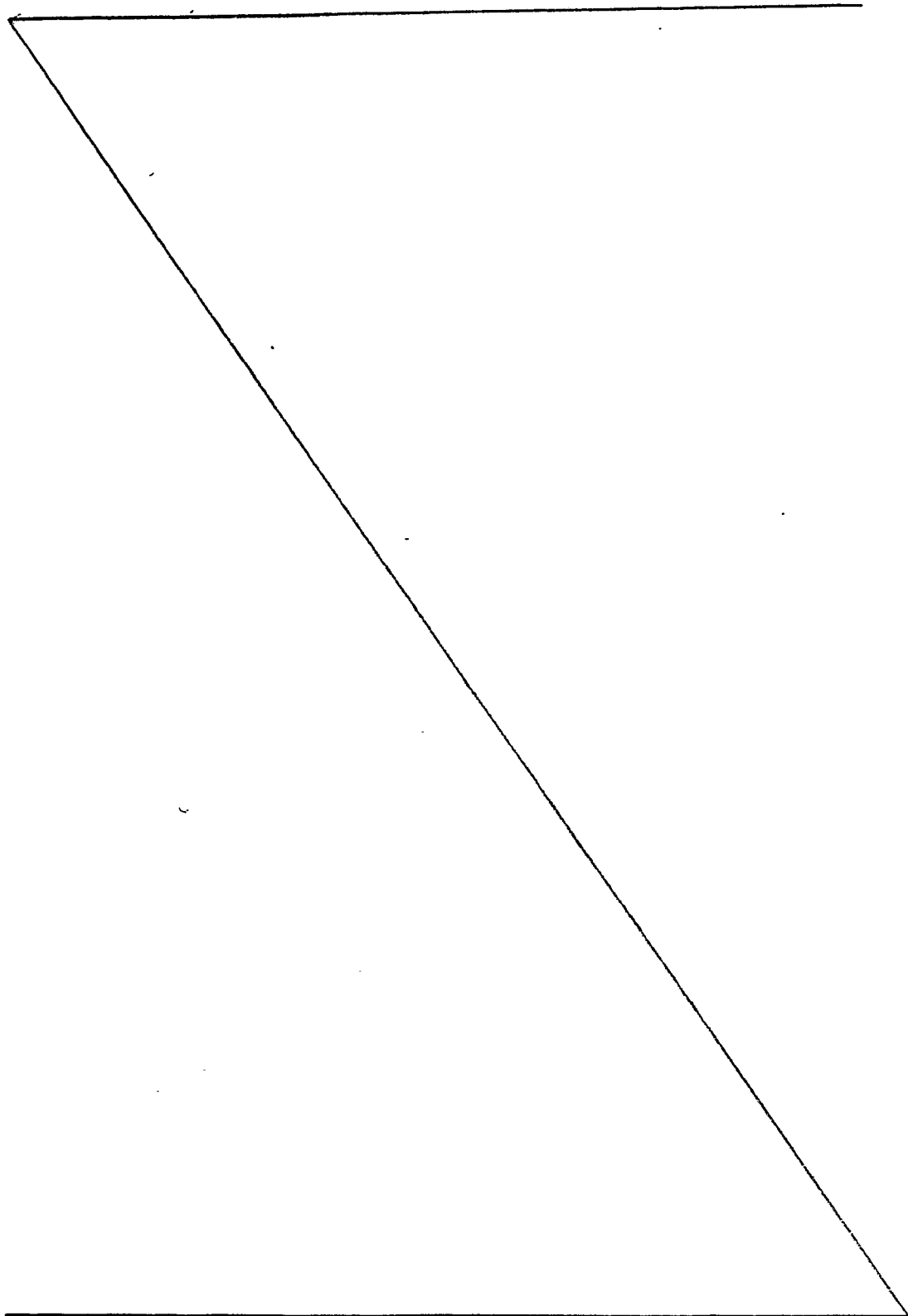


TABLE I

SPIN FINISH COMPOSITION (PERCENT BY WEIGHT)

	Components	Finish Identities			
		A	B	C	D
5	Isohexadecyl stearate	15	-	30	-
	Refined coconut oil	15	30	-	-
	POE(4)* lauryl ether	13	13	13	10
	Sodium salt of alkylarylsulfonate	10	10	10	10
10	POE(20)* tallow amine	5	5	5	5
	Nekal WS-25**	2	2	2	-
	Mineral oil	40	40	40	40
	C ₈ -C ₁₀ blended alcohol ester of pentaerythritol	-	-	-	30
15	Aerosol OT-S***	-	-	-	5

*Moles of ethylene oxide per mole of base material.

**GAF's trade name for solution consisting of 75 percent sodium dinonyl sulfosuccinate, 10 percent isopropanol, and 15 percent water.

20 ***American Cyanamid's trade name for solution consisting of 70 percent sodium dioctyl sulfosuccinate and 30 percent petroleum distillate.

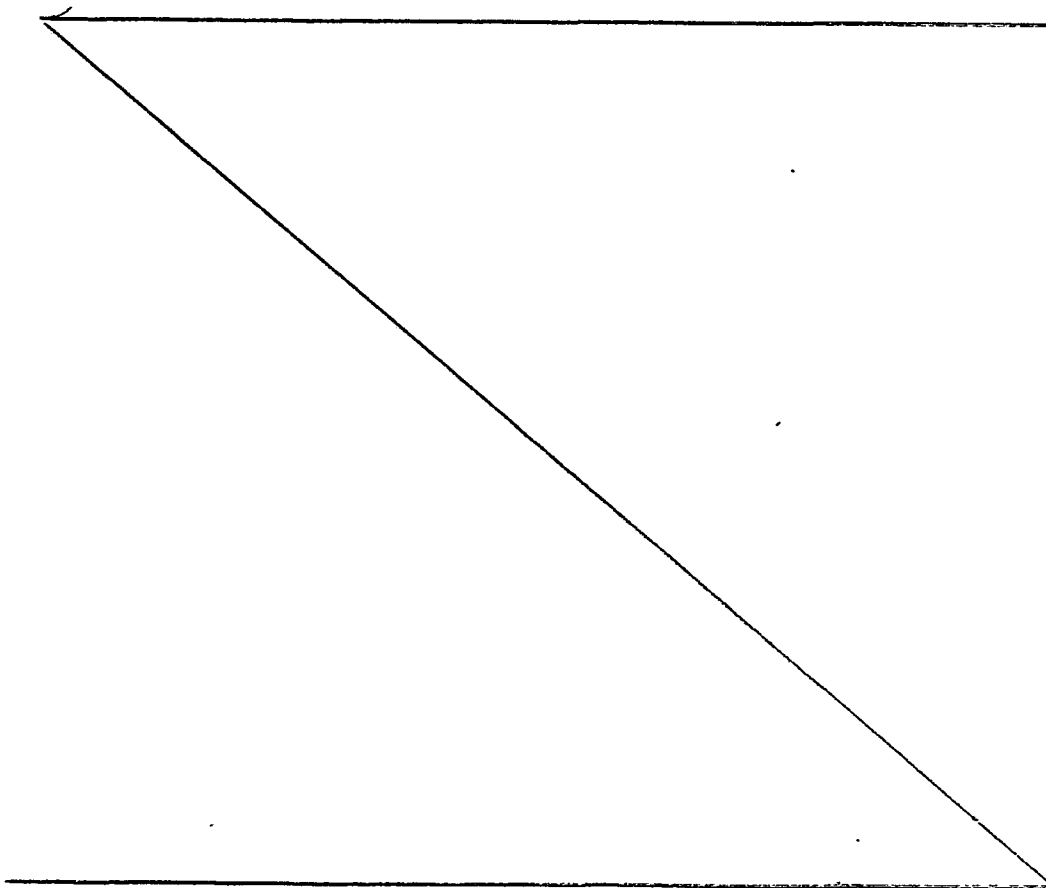


TABLE II

OVERFINISH COMPOSITIONS (PERCENT BY WEIGHT)

<u>Component</u>		<u>Finish Identities</u>		
		<u>A</u>	<u>B</u>	<u>C</u>
5	Isohexadecyl stearate	19.5	18.7	-
	Glycerol monooleate	1.8	1.7	-
	Decaglycerol tetraoleate	1.5	1.4	-
	POE(15)* tall oil fatty acid	2.4	2.3	-
	Sulfonated glycerol trioleate	3.6	3.5	-
10	POE(20)* tallow amine	1.2	1.2	-
	Gamma-glycidoxypropyl- trimethoxysilane	-	4.0	10
	Water	70.0	67.2	80
	Dimethyl polysiloxane emulsion**	-	-	10

15 *Moles of ethylene oxide per mole of base material.

**SWS 232, obtainable from SWS Corporation, Adrian, Michigan 49221.

TABLE IIISINGLE DIP* TREATING CONDITIONS

5	<u>Condition</u>	<u>Drying</u>			<u>Curing</u>		
		<u>Temperature</u> <u>(°C.)</u>	<u>Temperature</u> <u>(°F.)</u>	<u>Exposure</u> <u>(seconds)</u>	<u>Temperature</u> <u>(°C.)</u>	<u>Temperature</u> <u>(°F.)</u>	<u>Exposure</u> <u>(seconds)</u>
	1	149	300	80	204	400	60
	2	149	300	80	218	425	60
	3	149	300	80	241	465	60

10 *Non-ammoniated RFL dip at 4.0 to 5.0 percent total solids
pickup based on the weight of the cord, and 1 percent stretch.

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TABLE IV

		COOH	Spin Finish	Over- finish	UV Treat- ment	Treating Con- ditions*	Adhesion**	
Example	End Groups	Pounds Pull					Visual Rating	
5	1	11 ⁺ -1	A	A	7 sec.	3	22	2.5
	2	11 ⁺ -1	A	B	no	3	24	3.0
	3	11 ⁺ -1	A	B	0.3 sec.	3	26	3.9
	4	11 ⁺ -1	A	B	3 sec.	3	26	4.3
	5	11 ⁺ -1	A	B	7 sec.	3	26	4.4
10	6	11 ⁺ -1	A	C	no	3	22	2.5
	7	11 ⁺ -1	A	C	3 sec.	3	25	4.5
	8	24 ⁺ -1	A	B	no	1	24	3.0
	9	24 ⁺ -1	A	B	3 sec.	1	26	4.2
	10	24 ⁺ -1	A	B	no	2	25	3.5
15	11	24 ⁺ -1	A	B	3 sec.	2	29	4.4
	12	24 ⁺ -1	A	B	no	3	26	4.6
	13	24 ⁺ -1	A	B	3 sec.	3	29	4.9
	14	24 ⁺ -1	A	B	no	1	22	3.0
	15	24 ⁺ -1	A	B	1 sec.	1	22	4.2
20	16	24 ⁺ -1	A	B	2 sec.	1	22	4.4
	17	24 ⁺ -1	A	B	no	2	24	3.8
	18	24 ⁺ -1	A	B	1 sec.	2	22	4.3
	19	24 ⁺ -1	A	B	2 sec.	2	24	4.6

*See Table III.

**Tested at 250°F. (121°C.).

CONCLUSIONS

5 A comparison of Examples 1 through 7 shows the critical importance to adhesion for low carboxyl polyethylene terephthalate yarn of both exposing the yarn to ultraviolet radiation and treating the yarn with an aqueous finish composition containing an epoxy silane as previously described. The adhesion ratings were especially excellent for Example 5.

10 A comparison of Examples 8 through 19 shows the critical importance to adhesion for regular carboxyl polyethylene terephthalate yarn of both exposing the yarn to ultraviolet radiation and treating the yarn with an aqueous finish composition containing an epoxy silane as previously described. In Examples 9, 11 and 13, the polyethylene terephthalate yarn was exposed to ultraviolet radiation prior to treatment with an aqueous overfinish composition containing the epoxy silane, while in Examples 15, 16, 18 and 19, the polyethylene terephthalate yarn was treated with an aqueous overfinish composition containing the epoxy silane prior to exposure to ultraviolet radiation. The adhesion ratings were good in both instances. Further, it can be seen that substantially lower curing temperatures (treating conditions of Table III) can be utilized with the regular carboxyl yarn; this results in both energy savings and reduced plant emissions.

30 Alternative sources of ultraviolet radiation can be used. For example, medium wave length (280 to 400 nanometers) fluorescent sun lamp Model FS-40 available from Westinghouse Corporation is suitable. The intensity of ultraviolet radiation is expressed in micro-watts per square centimeter

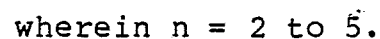
at a given distance. The total ultraviolet energy emitted from all sides of the ultraviolet lamp is expressed in watts. The total exposure is a product of energy, time and area which is expressed as

5 ultrads (microwatt seconds per square centimeter). The same number of ultrads can be achieved with a short exposure time at a high intensity of ultraviolet radiation, or a long exposure at a low intensity of ultraviolet radiation. Exposures of

10 the polyethylene terephthalate yarn to ultraviolet radiation for a period of up to 15 minutes showed virtually no loss of yarn breaking strength and other yarn physical properties, but did show

15 improvement in adhesion to rubber when treated with the overfinish system as described in this specification.

1. In a process for the production of polyester yarn involving spinning and drawing steps, the improvement which comprises: exposing the yarn subsequent to the drawing step to ultraviolet radiation; and treating the yarn with a finish composition which comprises water and a silane having the structure formula



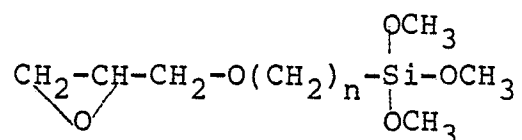
3. The process of claim 1 wherein the step
15 of exposing the yarn to ultraviolet radiation occurs
prior to the step of treating the yarn with the finish
composition.

5. The process of claim 1 wherein the silane is gamma-glycidoxypropyltrimethoxysilane.

7. The process of claim 1 wherein the finish composition is an overfinish composition which further comprises a dimethyl polysiloxane emulsion.

exposing the yarn subsequent to the drawing step for a time period of at least about 0.3 second to ultraviolet radiation having a wave length between 200 to 400 nanometers; and

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