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[54] **TREATMENT OF POLYPROPYLENE SURFACE TO PROMOTE ADHESIVE BONDING**

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[58] **Field of Search**.....161/67, 92, 175, 254, 253; 156/72, 334

[56] **References Cited**

UNITED STATES PATENTS

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[57] **ABSTRACT**

The adhesion of carboxylated SBR adhesive to polypropylene carpet backings is improved by a coating on the polypropylene of chlorinated polypropylene having a chlorine content of 10–20%.

2 Claims, No Drawings

TREATMENT OF POLYPROPYLENE SURFACE TO PROMOTE ADHESIVE BONDING

The present invention relates to a tufted carpet and more specifically to a tufted carpet using woven polypropylene as the primary and/or secondary backing and carboxylated SBR adhesive. The present invention provides improved adhesion between the polypropylene backing and the carboxylated SBR by means of a thin layer of chlorinated polypropylene containing about 10 to 20 percent chlorine pre-applied to the polypropylene backing.

Tufted carpet comprises a primary backing layer through which tufted face yarns are inserted, an adhesive being applied against the rear or lower surface of the primary backing to hold the tufts and primary backing together. In better grades of carpet, a secondary backing is applied over the adhesive to improve dimensional stability which is held to the primary backing and tuft yarns by means of the same adhesive.

A variety of yarns have been used for tufting, including particularly wool, cotton, acrylics, nylon and polypropylene. However, until quite recently, the primary and secondary backing materials have generally been woven jute. Many materials have been proposed as substitutes for jute, but it has continued to be used because of its strength and low cost. Nevertheless, it has been recognized for some time that a synthetic backing would be desirable because jute, being a natural fiber, fluctuates widely in price and availability. In particular, polypropylene has been recognized as a potential synthetic substitute for jute because its fibers are very strong, and it is one of the least expensive fiber materials.

One difficulty associated with polypropylene fibers which has tended to reduce their usefulness for this purpose is their poor adhesion to other materials, partly because of their smooth, hydrophobic surfaces. In particular, there has been a problem of poor adhesion between the primary and secondary backings when one or both are woven polypropylene and the adhesive is carboxylated SBR latex.

Another difficulty has been the tendency of the primary backing to ravel when a carpet is cut during installation to fit an irregular area. A principal object of the present invention is to improve the adhesion of polypropylene carpet backings and reduce the aforesaid raveling problem, and therefore to increase the usefulness of polypropylene as a primary and/or secondary carpet backing. Briefly stated, this object is achieved by applying a thin layer of a chlorinated polypropylene containing about 10 to 20 percent chlorine to the polypropylene carpet backing before applying carboxylated SBR adhesive. Surprisingly, it has been found that extremely small add-on of this material, e.g., only 0.0073 oz./sq. yd., greatly increases adhesion.

The invention is described in detail below and is illustrated in the drawing which illustrates the use of the fabric of the invention as a secondary carpet backing.

By "polypropylene fibers" it is intended to refer to filaments, fibers and fibrillated and slit films composed of fiber-forming polymer of propylene, which has stereospecific molecular structure and especially to isotactic polypropylene. As will be recognized, the fiber forming polymer may contain small amounts of other monomers, such as olefins, and especially

ethylene, which do not substantially alter the polymer's characteristics. By "chlorinated polypropylene," I refer to a solid product derived by introducing chlorine into polypropylene such that the product contains about 10–20 percent chlorine by weight of the chlorinated material. In this material, the polypropylene may be as defined above, except that it need not be a stereospecific fiber-forming polypropylene. The product is quite soluble in xylene and may be dissolved to concentrations of 50 percent or more. Typical molecular weight distribution for the chlorinated polypropylene is 2,000 to 10,000 with a number average of about 4,000. Typical chlorine content of the chlorinated polypropylene is 10 to 20 percent based on the dry weight of the polymer.

The primary backing used in the present invention is composed of woven polypropylene yarn, which may be spun staple fiber or filament yarn or fibrillated film, or unfibrillated slit film typically 1,100 to 2,200 denier; the fabric ordinarily has 12 to 30 warp ends per inch and 12 to 24 filling ends per inch and a weight of 3.5 to 15 ounce per square yard. The secondary backing may be a woven fabric made of the same yarns, typically about 550 to 2200 denier; the fabric ordinarily has 12 to 30 warp ends per inch and 8 to 24 filling ends per inch and a weight of 1.4 to 15 ounce per square yard.

Tufts are inserted and the tufts may be cut off in conventional manner. As tufting yarns useful in the present invention, mention may be made e.g., of wool, cotton and man-made fibers. The latter include rayon, acrylics (e.g., polyacrylonitrile), nylon, i.e., polamides of the type wherein repeating units in a polymer chain are connected together by amide linkages (e.g., polyhexamethylene adipamide or polycaproamide), polyester of the type wherein repeating units in a polymer chain are connected to each other by ester linkages (e.g., polyethylene terephthalate and copolymers thereof) and polypropylene. The tufting yarns may be 3,000 to 13,000 denier and may provide a weight of 20 to 45 ounces per square yard.

The adhesive used in the present invention is carboxylated SBR. SBR is a copolymer of butadiene and styrene containing, for example, 60–80 percent budadiene. The carboxylated SBR adhesives used in the practice of this invention are themselves known, and may be produced by methods described in the Encyclopedia of Chemical Technology, 2d. Ed., Volume 17, pages 657 and 658. They may be furnished as adhesives in the form of aqueous latices or dissolved in organic solvents.

The chlorinated polypropylene may be applied to the polypropylene carpet backing by any means. However, two techniques have been found to be especially useful. In one method the chlorinated material is first dissolved in xylene to a concentration of 50 percent by weight and then diluted to spray consistency with e.g., xylene or a chlorinated non-flammable solvent such as perchloroethylene. Concentration of chlorinated polypropylene in the spray solution may be 0.5–10 percent by weight for conventional spray equipment although other concentrations may be useful, depending on the equipment and spray conditions. The diluted solution is sprayed onto the fabric, which is then dried by evaporating the solvent. Results are about the same regardless of whether this is at room temperature or an

elevated temperature, although the latter is faster. This method of application has the advantage of carefully controlling the amount applied so that an extremely small amount of chlorinated polypropylene may be put on. Alternatively a solution of the chlorinated polypropylene, e.g., 50 percent in xylene, may be emulsified in water at a solids concentration of, e.g., 0.5 to 2.5 percent by weight. The emulsion thus obtained may be applied by padding, kiss roll applications or knife coating. If necessary, an emulsifying agent may be included in the formulation to stabilize it. If an emulsifier is used it may also be a lubricant for the fabric. Surprisingly, the result is the full equivalent of that obtained using a solution.

Present information indicates that the amount of chlorinated polypropylene applied to the fabric should be at least about 0.004 ounces per square yard for fabrics having a weight of about 2 to 6 ounces per square yard. At this application rate peel adhesion was found to be about **10.5** lbs./3 inch, which is slightly higher than the desired minimum of 10 lbs./3 inch. However, even at this application rate, the peel adhesion was substantially greater then untreated polypropylene, for which the peel adhesion is 1 lb./3 inch. In general one will use no more than necessary of the chlorinated polyolefin, for economic reasons. For example, one would not use more than 1 ounce per square yard, unless the fabric were exceedingly heavy, and generally the amount will be less than 0.2 ounce per square yard. Quite good results are obtained when the add-on is less than 0.025 ounces per square yard but a preferred range is 0.05 to 0.16 oz/sq. yd. for secondary carpet backings. For any particular fabric, the optimum add-on can be determined by making up samples with a range of add-ons and measuring peel adhesion.

The following examples illustrate the invention, in which all parts and percentages are by weight. The chlorine content of the chlorinated polypropylene used may be 18.0 percent total chlorine base on the dry weight of the polymer.

EXAMPLE 1

Solutions are prepared as follows, the compositions being given in percent by weight:

	Sample Number	
	1	2
Chlorinated polypropylene	2.5%	5%
Xylene	97.5%	95%

The samples were sprayed onto swatches of woven polypropylene secondary carpet backing of a tenter frame to give the following wet pickups:

Sample 1	0.23 oz./yd.^{2}
Sample 2	0.30 oz./yd.^{2}

The fabrics were heat set and dried for 2 minutes in an oven maintained at 305° F. Peel strengths to determine the degree of adhesion of the secondary backing were then made on these samples using the following test method. The method was developed to closely simulate the actual conditions that exist when carpet manufacturers apply adhesive and secondary backing in the fabrication of carpets.

METHOD OF DETERMINATION OF PEEL ADHESION

1. Cut a piece of tufted carpeting 4 × 12 inches.
2. Apply 39.3 grams of carboxylated SBR adhesive to the underside of the tufted carpeting with a spatula using slight pressure to assure filling of the voids between tufts. This amount of adhesive is equivalent to 41 oz./sq. yd. wet weight.
3. After applying the adhesive, lay the secondary backing onto the adhesive and pat slightly by hand to remove any entrapped air.
4. Next turn the sample over so that the tufted side is upright and again pat slightly to assure uniform contact between the secondary backing and the adhesive.
5. Place the sample, tufted side up, in and air-blown oven and heat for approximately 30 minutes at 250° F.
6. Remove sample from oven when appropriate and allow the sample to cool to room temperature.
7. Cut a 3 inch wide strip from sample approximately 30 minutes after removal from oven.
8. Insert sample in jaws of Scott tester, Model J–2, 3 inch clamps, 12 **inch** /minute pull rate, so that the tufted carpeting is held by the upper jaw and the secondary backing is held by the lower jaw of the tester. This procedure is known as the 180° peel adhesion test.
9. Engage the tester and peel approximately 6 inches of the sample, recording the peel strength on appropriate charts.
10. Remainder of the sample is retained for testing after a 24 hour period or such time as is required for the sample to reach equilibrium at testing room conditions.
11. Report results obtained as pounds peel adhesion per 3 inch strip.

The test results were as follows:

	Peel Strength/3 inches	
	30 minutes	24 hours
Sample 1	12.5 lbs.	14 lbs.
Sample 2	12.0 lbs.	13 lbs.

EXAMPLE 2

Example 1 was repeated using the following solutions:

	Sample 3	Sample 4
Chlorinated polypropylene as in Example 1	2.5%	1.25%
Xylene	97.5%	98.75%

Application rates and peel strengths (lbs./3 inch) were as follows:

		Peel Strength	
	Wet Pickup	30 minutes	24 hours

Sample 3	0.18 oz./yd.²	9.5	13.5
Sample 4	0.16 oz./yd.²	7.5(wet)	13.0

EXAMPLE 3

In this example, the polypropylene fabric was heat set on a tenter frame for 2 minutes in an oven at 305° F. and then the solutions were sprayed on while the fabric was still hot. The fabric quickly air dried. Drying was too rapid for wet pickups to be measured, and dry add-ons were too small to be measured. The solutions used were as follows:

	Sample 5	Sample 6
Chlorinated polypropylene as in Example 1	1%	0.5%
Xylene	99%	99.5%

Peel strengths (lbs./3 inch) were as follows:

	30 minutes	24 hours
Sample 5	9.0	11.0
Sample 6	8.5	11.0

EXAMPLE 4

Eight samples were prepared using the following solutions:

	Sample 7	Sample 8
Chlorinated polypropylene as in Example 1	0.5	1.0%
Xylene	99.5%	99%

Application rates and peel strengths were as follows:

	Add-on of Chlorinated Polypropylene	Peel Strengths (lbs./3 inch)	
		30 minutes	24 hours
Sample 7A	0.0086 oz./sq.yd.	9.5	13.5
Sample 7B	0.0071	10.0	13.0
Sample 7C	0.0075	9.5	12.5
Sample 7D	0.0059	9.5	13.5
Sample 8E	0.0113	11.5	15.0
Sample 8F	0.0090	8.5(wet)	13.0
Sample 8G	0.0145	3.0(wet)	13.0
Sample 8H	0.0087	6.5	13.0

The following examples show the possibility of including other materials in the bath for the purpose of additional weight, stiffness, lubrication, etc. without interfering excessively with the adhesion properties of the chlorinated polypropylene.

EXAMPLE 5

Solutions are prepared as follows:

	Sample 9	Number 10
Chlorinated Polypropylene as in Example 1	2.5%	5%
Ethylene-vinyl acetate copolymer containing about 20% vinyl acetate	5%	10%
Xylene	95%	90%

The samples were sprayed onto swatches of woven polypropylene secondary backing as described in Example 1 to give the following wet pickups:

Sample 9	0.77
Sample 10	0.61

Peel strengths were as follows:

	Peel strength/3 inches	
	30 minutes	24 hours
Sample 9	15.0	14.0
Sample 10	12.0	13.5

EXAMPLE 6

The procedure of Example 2 was repeated, using, as the coating solution, the following mixtures:

	Sample Number 11	12
Chlorinated Polypropylene as in Example 1	2.5%	1.25%
Copolymer as in Example 5	5%	2.5%
Xylene	95%	97.5%

Application rates and peel strengths were as follows:

Sample No.	Wet Pickup	Peel Strength 30 minutes	24 hours
11	0.17	8.5(wet)	12.5
12	0.25	9.5	12.5

EXAMPLE 7

The procedure of Example 3 was repeated, using the following solutions:

	Sample 13	Sample 14
Chlorinated Polypropylene as in Example 1	1%	0.5%
Copolymer as in Example 5	1%	0.5%
Xylene	98.5%	0.75%

Peel strengths were as follows:

Sample No.	Peel Strength 30 minutes	24 hours
13	11.5	12.0
14	8.0	11.5

EXAMPLE 8

Eight samples were prepared using the following solutions:

	Sample 15	Sample 16
Chlorinated polypropylene as in Example 1	0.5%	1
Copolymer as in Example 5	0.5%	1%
Xylene	99%	98%

Application rates and peel strengths were as follows:

	Add-on, oz./yd.²	Peel Strength lbs./3 inches	
		30 minutes	24 hours
Sample 15A	0.0085	9.5	13.5
Sample 15B	0.0030	11.0	13.5
Sample 15C	0.0126	9.0	12.5
Sample 15D	0.0142	9.0(wet)	13.0
Sample 16A	0.0219	5.5(wet)	13.5
Sample 16B	0.0247	3.5(wet)	12.5
Sample 16C	0.0235	8.0	13.0
Sample 16D	0.0283	12.0	13.5

The following examples illustrate the use of aqueous emulsions:

EXAMPLE 9

An aqueous emulsion was prepared from the following materials:

10 grams of the chlorinated polypropylene of Example 1
30 grams xylene
3.5 grams sodium lauryl sulfate
2.5 grams of the sodium lauryl sulfate was dissolved in the xylene with the chlorinated polypropylene. The additional gram was dissolved in 355 grams of warm water. The water was added slowly to the xylene solution with agitation. Inversion took place and a stable emulsion was obtained.

EXAMPLE 10

The following materials were mixed to form a solution:

20 grams of the chlorinated polypropylene of Example 1
50 grams xylene
1 gram sodium lauryl sulfate

A water phase was separately prepared by dissolving 16 grams of Elvanol 92–60 (polyvinyl alcohol) and 1 gram sodium lauryl sulfate in water. The water phase was added slowly with agitation to the solution and a stable emulsion was obtained.

EXAMPLE 11

The following materials were formed into a stable emulsion by the method of Example 10:

5 grams of the chlorinated polypropylene of Example 1
15 grams xylene
1 gram Renex 20 (polyoxyethylene ester of mixed fatty and resin acids)
1 gram Renex + (polyoxyethylene ether alcohol)
10 grams water

EXAMPLE 12

Samples of the emulsions of Examples 9, 10 and 11 were diluted with water to a solids content of 2.5 percent of chlorinated polypropylene. Each was padded onto secondary carpet backing of polypropylene having 24×8 WXF construction using black fibrillated polypropylene film as the fill yarn. Padded swatches were dried on a pin tenter frame for 2 minutes in a 305° F. oven. The results were as follows:

Emulsion used	Add-on oz./sq.yd.	Peel Adhesion 30 minutes	24 hours
Example 9	.16	10.3	13.0
Example 10	.11	8.5	14.0
Example 11	.05	11.8	13.8

EXAMPLE 13

Samples of the emulsions of Examples 9, 10 and 11 were diluted with water to concentrations of 1.25 percent and 0.625 percent solids of chlorinated polypropylene. The diluted emulsions were applied and tested as in Example 8 with the following results:

Emulsion used	Solids in emulsion	Add-on oz./sq.yd.	Peel adhesion 30 minutes	24 hours
Example 9	1.25%	0.04	9.0	13.0
Example 10	1.25%	0.04	10.5	12.5
Example 11	1.25%	0.09	9.5	10.5
Example 9	0.625%	.15	9.0	11.0
Example 10	0.625%		8.5	10.5
Example 11	0.625%	.01	8.3	10.5

EXAMPLE 14

The following example illustrates the advantages of applying the chlorinated polypropylene as respects antiraveling properties. An emulsion of the type shown in Examples 9, 10 and 11 are applied to primary carpet backing of woven polypropylene with a construction of 24 warp ends and 11 fill yarns in the same manner shown in the examples for application to secondary carpet backing. The add-on in oz./sq.yd. of material was comparable to that obtained on secondary backing, namely 0.05 to 0.16 oz./sq.yd. This primary backing material was then tufted along with an untreated control fabric of the same construction. These two samples were then given a precoat of carboxylated SBR adhesive on a carpet coating unit for the purpose of determining the resistance to raveling of the primary backing materials. This precoat is essentially the same composition and weight normally used in making up a complete carpet but for test purposes a secondary fabric backing is not joined to the structure with the adhesive. After drying, the precoated samples were tested by the following method to determine ravel resistance:

A 6×6 inch coated sample is cut parallel to a course of tufted yarn. A hook is then embedded between the second and third courses of yarn and the resistance to pull measured on the Instron tester at a 12 inch/minute pull rate. Recorded value is the peak or maximum resistance obtained.

For the examples cited the edge pull resistance results were as follows:

Control Sample: 18 lbs.
Treated Sample: 29 lbs.

This improvement in ravel resistance is believed to be the result of contact between the carboxylated SBR adhesive and the primed or treated surface of the primary backing.

The product of this invention is illustrated in the attached drawing.

I claim:

1. In a tufted carpet comprising tufted yarns inserted through a primary carpet backing and having a secondary backing adhered to its lower surface by means of an adhesive;

the improvement wherein the adhesive is a carboxylated D styrene-butadiene copolymer rubber, and at least one of said backings has a weight of 1.4 to 15 ounces per square yard, comprises polypropylene fibers, and is pre-coated with chlorinated polypropylene having a chlorine content of 10–20% by weight, the amount of said chlorinated polypropylene being 0.04 to 0.16 ounces/square yard.

2. In a tufted carpet comprising tufted yarns inserted through a primary carpet backing and adhered thereto by means of an adhesive;

the improvement wherein the adhesive is a carboxylated styrene-butadiene copolymer rubber, and

said backing has a weight of 1.4 to 15 ounces per square yard, comprises polypropylene fibers and is coated with chlorinated polypropylene having a chlorine content of 10 to 20 percent by weight, the amount of said chlorinated polypropylene being 0.04 to 0.16 ounces/square yard.

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