Adhesion Between Poly(ethylene terephthalate) Silk and Chlorohydrin Rubber for Aerial Film

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ABSTRACT: The surface adhesion activation of poly(ethylene terephthalate) silk (PET silk) was accomplished through dipping in a reaction product of a water-soluble modified epoxide resin (SJR-2) and a precondensed resorcinol-formaldehyde (SJR-1), and by partial curing of the dip. Then, the adhesion activated sample was coated with a single resorcinol-formaldehyde latex (RFL) dip prepared from SJR-1. The effects of heat treatment conditions and formulation of impregnation solution as well as RFL prepared with water-soluble SJR-1 resin on the adhesion between PET silk and chlorohydrin rubber for aerial film were studied. High adhesion between PET silk and chlorohydrin rubber was obtained by using optimized RFL formulation and heat treatment condition, and the high levels of adhesion were maintained after oil impregnating test, dynamic fatigue and storage stability test. The PET silk possessed good adhesion to natural rubber (NR), chloroprene rubber (CR), hydrogenated nitrile rubber (HNBR), acrylonitrile-butadiene and rubber (NBR), and chlorohydrin rubber (CHR). RFL filled with diffused carbon black improved the fatigue endurance and oil resistance of adhesion. The adhesion activator is nontoxic. This method of adhesion activation and RFL preparation for adhesion treatment of poly(ethylene terephthalate) silk is a practical technique with excellent technological, economical, and safety effects. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 88: 2097–2099, 2003

Key words: polyesters; adhesion; rubber

INTRODUCTION

Adhesion between fiber textiles and rubber is a very important matter in the rubber industry. Classical textile reinforcing materials such as rayon and nylon possess polar surfaces; sufficient adhesion between such materials and rubber is usually obtained by the dipping of textiles in a resorcinol-formaldehyde latex (RFL) suspension. In the case of polyester textiles, where surface reactivity is much lower, a pretreatment of these materials is required (often with epoxy reagents or isocyanate compound subcoats) before standard RFL dipping.[1–3] These pretreatment methods have their disadvantages; that is, isocyanate is toxic, subcoats need organic solvents harmful to personal health and pollute the environment, and uncured epoxy on the surface of textiles is harmful to personal health, too. In comparison to the *in situ* RFL used in our previous study,[4,5] we improved the adhesion of aramid fiber and nylon fiber to rubber by preparing an RFL with precondensed resorcinol-formaldehyde (PRF). To avoid using harmful organic solvents, in this study we pretreated the surface of PET silk with a water-soluble modified epoxide resin (SJR-2), and such a technology of RFL prepared with PRF was adapted. The RFL dip recipe and heat treatment technology for the PET silk dipping were optimized so that good adhesion between the PET silk and NR, CR, NBR, HNBR, and CHR was obtained.

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EXPERIMENTAL

Materials

PET silk was used as a reinforcing material for aerial films. Its thickness was 0.14 mm.

SJR-1 and SJR-2[6] resins were used to prepare the adhesion activator and RFL. The aqueous SJR-1 resin was a precondensate of resorcinol and formaldehyde in a water solution containing an acid or base catalyst under certain reactive conditions. The aqueous SJR-2 resin was a highly active and water-soluble epoxide resin.

The rubber stocks used in the adhesion tests were provided by Chinese North-West Rubber Plant (Xianyang, China) and Shenyang Fourth Rubber Factory (Shenyang, China), respectively. They were as follows:

CHR: rubber stock of aerial film.
HNBR: rubber stock of aerial film.
NBR: tuber rubber stock of high-pressure hydraulic hose.
CR: cover rubber stock of high-pressure hydraulic hose.
NR: rubber stock of tire.

TABLE I
Effect of Pretreatment of the Subcoat on Adhesion of the PET Silk

Rubber Stock	Peel strength (kN m^{-1})
CHR	2.7–3.3
NBR	2.6

The latex types used in the tests were chloroprene latex (LDR-403, Chongqing Changshou Chemical Factory, Chongqing, China), vinylpyridine latex (VP; Nipol 2518FS, Nippon Zeon Co. Ltd., Japan), and NBR-26 latex (Lanzhou Chemical, Lanzhou, China).

Experimental procedure

The PET silk was dipped in a subcoat (surface adhesion activator), dried and partial cured, dipped in RFL prepared from SJR-1, dried and cured, and vulcanized. This was followed by adhesion testing.

Test methods

Adhesion samples were prepared to be a sandwich in which one-ply rubber stock was clamped between two-ply textiles. The thickness of the interlayer sample was about 1.0 mm.

Adhesion of the dipped PET silk to different rubber stocks was measured according to the two-ply strip peel adhesion test GB532-91.

Fatigue test conditions

The distance between the upper and lower lampers in the fatigue tester was 70 mm, the reciprocating distance of the lower lamper was 50 mm, the test speed was 300 rpm, and the samples were not stretched.

RESULTS AND DISCUSSION

Surface adhesion activation (A-A) of PET silk

At present, a pretreatment of the PET silk is required (often with epoxy reagents or isocyanate compound subcoats) before standard RFL dipping.[1–3] This procedure is called surface A-A. We synthesized a completely water-soluble and highly active modified epoxide resin. This resin could self-condensate and cocondensate with SJR-1 resin. The PET silk was dipped in a 3% aqueous solution of such a resin and an SJR-1 resin; then, the dip was partially cured. A highly active and firm film (ca. 3% of the silk weight) was formed on the surface of the PET silk. The data in Table I show that the PET silk met the adhesion level of CHR stock (in practical production, the peel strength desired is more than 2.0 kN/m), even without an RFL topcoat. In this study, the pretreatment method did not use any organic solvent, and after cocondensation partially cured, no uncured epoxide harmful to personal health remained on the surface of the PET silk.

The control of heat treatment conditions of the subcoat directly affected the adhesion effect of surface A-A. The data in Table II show that different drying times affected the adhesion effect of the subcoat at 150°C. The pickup of the subcoat was about 5.0%; the heat treatment effect was optimal when the drying time was 4 min at 150°C.

TABLE II
Effect of Heat Treatment Conditions of the Subcoat on Adhesion of the PET Silk to CHR Stock

	Time (min)		
	3	4	5
Peel strength (kN m^{-1})	1.8	3.3	2.8

TABLE III
Latices that Were Selected by Different Stocks

	Stock				
	CHR	CR	HNBR	NBR	NR
Latex	LDR-403	LDR-403	NBR-26	NBR-26	VP

Effect of RFL preparation and heat treatment conditions on the adhesion of the PET silk

The latex used in RFL must match the rubber stock. Therefore, the adhesion treatment of the PET textile to different rubber stocks needed different latices. Table III lists the latices used in different rubber stocks.

There were many factors that influenced the adhesion strength of the textile dipped in RFL, such as the preparation methods of RFL, the formulation of the RFL dip, the pickup of the adhesive on the silk, and heat treatment conditions. The data in Table IV show the effect of the weight ratio of the resin/latex (RF/L)

TABLE IV
Effect of the RF/L Weight Ratio on the Adhesion of the PET Silk to CHR Stock

	RF/L weight ratio		
	1/4	1/5	1/6
Peel strength (kN m^{-1})	4.1	3.8	3.4

TABLE V
Effect of Heat Treatment Time at 160°C on the Adhesion of PET Silk to CHR Stock

	Time (min)		
	4	5	6
Peel strength (kN m^{-1})	3.1	3.1	4.3

TABLE VI
Adhesion Results of the PET Silk to Different Rubber Stocks

	Stock					
	NR	CR	NBR	HNBR	CHR	CHR
Peel strength (kN m^{-1})	4.5	5.0	5.0	4.9	4.0–5.6	4.1[a]

[a] This datum was the result of Shenyang Fourth Rubber Factory stock with methylenediphenyl di-isocyanate (MDI) treatment.

on the adhesion of the PET silk. The data in Table V show the effect of heat treatment conditions on PET silk adhesion.

In this study, the subcoat was prepared with SJR-1 resin and SJR-2 resin. The topcoat was prepared with SJR-1 resin, and the latex was a variable parameter. After the optimization of a series of RFL formulations and treatment conditions, good adhesion of the polyester textile to different rubber stocks was obtained. The results are listed in Table VI.

Fatigue and oil resistance of the adhesion of the PET silk to CHR

Fatigue endurance and oil resistance

The data in the Table VII show that the maintenance percentage of the adhesion strength was more than 60% after a fatigue of 10^5 cycles; the samples had no stripping-off phenomenon. The maintenance percentage of the adhesion strength was more than 80% when the sample was dipped with #2 kerosene after 24 h at room temperature.

Effect of carbon black (CB) filler in RFL on the fatigue and oil resistance of adhesion

CB filler dispersed in an RFL dip can improve the adaptability of a textile to rubber stocks and therefore, improve the fatigue and oil resistance of adhesion. The ratio of CB to latex was selected as 15/100 by many experiments. CB soluble dispersion is composed of

TABLE VII
Effect of RFL Filled with CB on Textile Adhesion

1	2	3	4
3.8	2.6 (68.4%)	3.7 (97.4%)	2.0 (52.6%)
3.5	3.6 (100%)	3.5 (100%)	2.6 (74.3%)

1, 2, 3, and 4 represent the peel strength (kN m^{-1}) of direct measurement, a fatigue of 10^5 cycles, dipped with #2 kerosene, and a fatigue of 10^5 cycles after dipping with #2 kerosene, respectively. The data in parentheses are the maintenance percentages of adhesion strength.

TABLE VIII
Storage Stability of the Treated PET Silk

Compound	Direct peel strength (kN m^{-1})	Peel strength (kN m^{-1}) after 2 months of storage	Peel strength (kN m^{-1}) after 2 months of storage
CHR	4.5	4.3	—
CHR[#]	3.8	—	3.8

The two types of rubber stocks were all CHR stocks. Only the formulation was different.

high abrasion CB and the condensate of sodium lauryl sulfonate to formaldehyde, CB particle diameter is 0.4 mum, it is ground in a global-stone muller. The results of adhesion are shown in Table VII.

Storage stability of dipped PET silk

A large number of the PET silk samples were treated in the laboratory. Some of them were used for the testing of aerial film product adhesion in Shenyang Fourth Rubber Factory. The adhesion of the samples to two types of different CHR stocks was tested after storage of 2 and 4 months, respectively. The results are shown in Table VIII. Compared with the original adhesion strength data measured in the laboratory, the adhesion property showed no significant change. The results show that the storage stability of the dipped PET silk is good.

CONCLUSIONS

A desirable A-A effect of PET silk was obtained by treatment with an aqueous solution of modified epoxide resin. Only a single RFL-dipped textile had good adhesion to rubber. Compared with the two-step dipping method, this treatment technology of a PET silk for adhesion to rubber does not use any organic solvents, and there is no poison of an uncured epoxide on the surface of the treated PET silk by partial curing of the dips. It is a practical technique with excellent technological, economical, and safety effects.

References

1. Weening W. E. Proceedings from the International Rubber Conference, 1986; Vol. 2, Swedish Inst. of Rubber Technology: Varamo, Sweden, 1986, p 298.
2. Takeyama, T.; Matsui, J. Rubber Chem Technol 1969, 42, 229.
3. Solomon, T. S. Rubber Chem Technol 1985, 58, 566.
4. Jing, Y. Master's Thesis, Xi'an Jiaotong University, 1989.
5. Yuexian, S. Master's Thesis, Xi'an Jiaotong University, 1991.
6. Youdao, W.; Bihe, W.; Yuansuo, Z.; Yuexian, S. Chin. Pat. CN1088156A (2000).