New Method for Evaluating Adhesive Property of Fabrics and TPU: The Rigid-Body Pendulum Rheometer Approach

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ABSTRACT: Coating and laminating processes play an important role in textile industry. They are frequently used to produce fabric laminates during which the physical properties and appearance of textile fabrics are modified and enhanced. Currently, the adhesive property testing of a fabric laminate is done so by the use of pulling test machines such as Universal Tensile Tester, which measures the strength required to peel the tested materials apart. The adhesive test to date has not yet been performed using a newly developed machine, Rigid-Body Pendulum Rheometer (RPR). This study was to establish a more effective method for fast-evaluating adhesive properties of fabric laminates by assessing the performance of RPR. Specifically, RPR and Universal Tensile Tester were used to measure, respectively, the viscosity and peeling strength of PET/TPU (thermoplastic polyurethane) and nylon/TPU in response to UV exposure and water immersion. RPR can continuously observe and record viscosity behavior of tested samples in various temperature condition including from low temperature to high temperature, it not only measures viscosity speed, it also detects the differences in crosslinking and measures data generated during the softening process when the balanced time was achieved during the oscillations procedure. J Appl Polym Sci 103: 2855–2863, 2007

Key words: adhesives; viscosity; thermoplastics; elastomers; fibers

INTRODUCTION

Thermoplastic polyurethanes (TPU) are flexible compounds with excellent chemical, mechanical, and physical properties, which have been used for wide applications.[1–7] TPU have superior physical, mechanical, and process forming properties because of their durable strength, excellent moisture permeability, ability to resist impact, water, and aging.[8–12] When used as raw material, TPU can bond with diverse substrates, and are therefore used for making adhesives in place of traditional PU coating and PVC adhesive.[13]

Coating and laminating processes play an important role in textile industry. They are frequently used to produce fabric laminates during which the physical properties and appearance of textile fabrics are modified and enhanced. To broaden the application of fabric laminates, considerable attention has been invested in the development of fiber coating and laminating processes to improve material's fire resistance,[14,15] waterproof ability, and moisture permeability.[16,17] Nonsolvent hot melts adhesive technology has also been developed, which is a superior method than the traditional hot melts method in the production of fiber laminates.[18–26] Testing the adhesive property such as peeling strength of fiber laminates, which have been improved with the aforementioned technologies under different temperatures, pressures, and time periods, has also been investigated extensively.[18–27]

Currently, the adhesive property of fabric laminates is tested using a pulling test machine, which measures the strength required to peel the fabric laminates apart.[8,23] To the best of our knowledge, fabric laminate adhesive test has not yet been performed using Rigid-Body Pendulum Rheometer (RPR), which is newly developed by Tanaka.[28–32] RPR, however, has been used to successfully evaluate physical and mechanical properties of many other materials. For instance, RPR was used to study the curing behaviors of coating resin for optical fiber, adhesive properties of UV curable pressure-sensitive adhesive tape for semiconductor processing, and curing behavior of silicone rubber/PEL polymer electrolyte.[27,33–36]

The aim of this research was to establish a more effective method for fast-evaluating adhesive properties of fabric laminates. Specifically, RPR and Universal Tensile Tester were used to measure, respectively, the viscosity and peeling strength of polyester (PET)/TPU and nylon/TPU in response to UV exposure and water immersion. RPR has been shown to produce reliable

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results with less testing time and sample as data obtained from RPR were 84.95% comparable with data obtained from Universal Tensile Tester. We have hence, concluded that the use of RPR is a superior method in the testing of adhesive property of PET/TPU and nylon/TPU samples.

EXPERIMENTAL

Materials

Elastollan 685A thermoplastic polyurethane (TPU) film, 105 μm in thickness was purchased from Elastogran (Uberlingen, Germany). Polyethylene terephthalate (PET, 100% polyester woven fabric, 84 density of warp ×74 welt ×58″ fabric width, 280 g/y unit weight), and nylon (polyamide, 100% woven fabric, 147 density of warp ×72 welt ×59″ fabric width, 125 g/y unit weight) were bought from Lipeng Enterprise (Taipei, Taiwan).

Microscopic viscosity study by RPR

Instrument

A cylinder-type Rigid-Body Pendulum Rheometer as shown in Figure 1 (model RPR α-100 with FSM-300 a specimen mount, SFM-100 cylindrical edge and FRB-400 Rigid-Body Pendulum, Tohoku Electronic Industrial, Tokyo, Japan) was used to measure viscosity of tested samples in this study. The use of RPR in this study was based on the principle of RPR described as follows:

The principle of RPR has been described by principle of Rigid-Body Pendulum Rheometer.[26–32] Figure 2 shows the typical curing curve, as measured with the rigid-body pendulum instrument with theoretical analysis as follows. In curve C, the reaction begins after point 1, and molecular weight is increased; therefore, viscosity increases as well. The increasing slope of viscosity between points 3 and 4 shows the velocity of increased viscosity. The first point of inflection is called the gel point, and its corresponding time is the gel time. After point 5, the last point of inflection is called the cure point, and its corresponding time is the cure time, which results in a balanced curve.

Period of oscillation (T) and logarithmic damping ratio (Δ) can be obtained by the following equation.

$$T = (P_1 + P_2 + \cdots\cdots + P_n)/n$$

$$\Delta = [\ln(A_1/A_2) + \ln(A_2/A_3) + \cdots\cdots + \ln(A_n/A_{n+1})]$$

where P is the wavelength, n is the number of oscillations, and A is the amplitude.

Substances have viscosity (η). The viscosity varies depending on the molecular weight (M), ambient temperature (t), and each k, θ, and β is the constant, curing, or drying changes the molecular weight. Viscosity of substance has a function of stopping oscillation of the oscillated body, which is in contact with

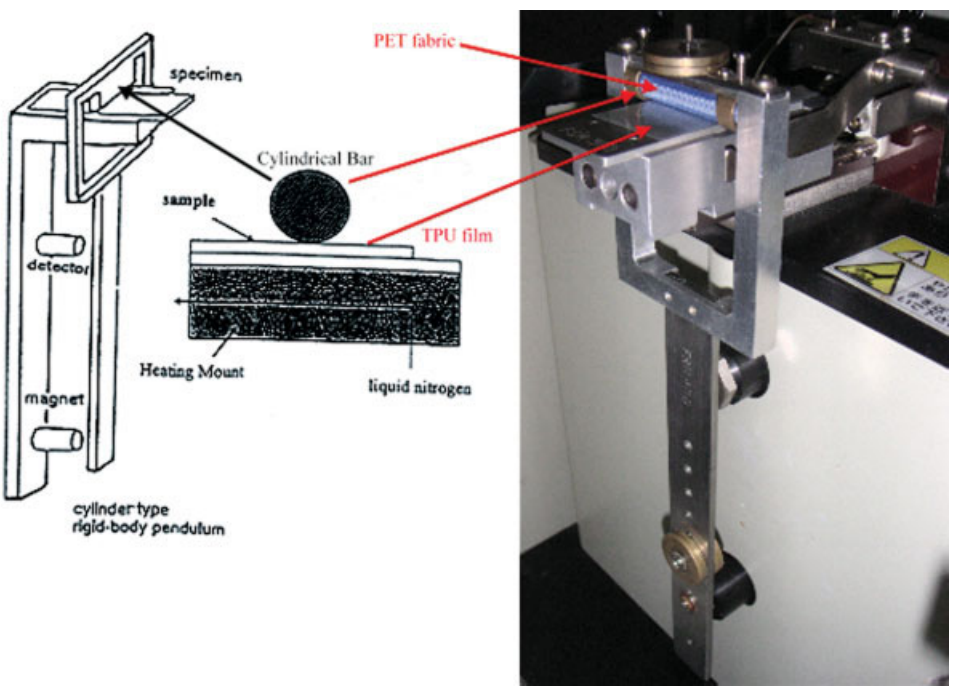

Figure 1 Set-up of Rigid-Body Pendulum Rheometer (model α-100). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

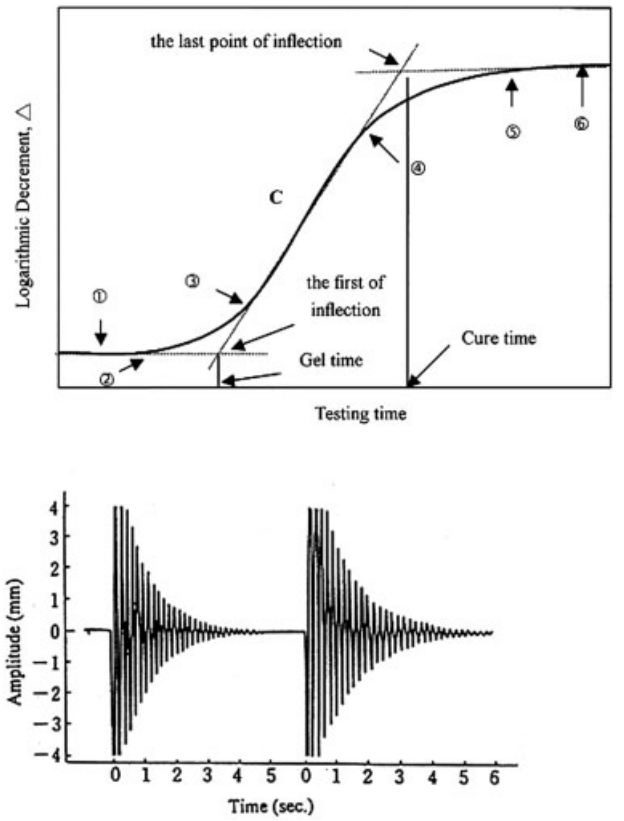

Figure 2 Curing behavior by rigid-body pendulum rheometry (RPR).

the substance. Therefore, as viscosity becomes greater, logarithmic decrement increases. Viscosity is evaluated from the following equations:

$$\eta = kM^{\theta}$$

$$\eta = k \ \ln \ \exp\left[\frac{\beta}{t}\right]$$

For a practical test of the curing process, the testing method made use of a damping pendulum system in compliance with the specifications of ISO 1522. To measure the curing process, a rigid-body pendulum equipped with a knife (or cylindrical) edge was provided.

Briefly, RPR was set up with the pendulum on the specimen plate as shown in Figure 1. The cylindrical surface of a cylindrical bar is put on the solid material. The test piece, coated or set on a plate, was placed on a heating mount, and the pendulum was set so that the edge, or the fulcrum of the swing, came vertically in contact with the coated surface.

Determination of optimal testing temperature for TPU film and effect of temperature on adhesive property of PET/TPU and nylon/TPU

To determine the optimal testing temperature of TPU film, PET fabric (2×2 cm^2) was wrapped around the cylinder part roll and a piece of TPU (2×2 cm^2) was placed on the metal plate and heated on the specimen mount to 90, 100, and 110°C. The viscosity measured by the log-damping ratio was recorded every 1.5 min for the curve becomes balanced.

Concurrently, this study also recorded the effect of temperatures on the viscosity of PET/TPU sample while the optimal testing temperature of TPU film was tested. Following the determination of optimal testing temperature of TPU at 100°C, a piece of nylon (2×2 cm^2) was wrapped around the cylinder edge and a new piece of TPU film (2×2 cm^2) was heated on the specimen mount to 80, 90, and 100°C. The viscosity produced by these two materials was then recorded every 1.5 min for the curve becomes balanced.

The cylindrical edge rolling rates on the surface of the test piece of TPU is in accordance with the swing of pendulum. And the resistance between cylindrical edge and TPU test piece reduces the swing of pendulum. From the logarithmic damping ration (Δ), the viscoelastic property of TPU test piece surface and the holding layer can be obtained.

Effect of environmental factors on adhesive property of PET/TPU and nylon/TPU

Samples (PET, nylon, and TPU films, width × length; 25×100 mm^2) were exposed to UV lights at 340 nm (UltraViolet/Condensation Weathering Device) for various durations (100, 200, or 300 h). In a separate study, a new set of samples with the same dimensions was treated with water (100 g) containing household washing powder (Bailan laundry concentrate powder with Unilever Taiwan Company Limited). Main ingredients: LAS-Na (n-$RC_6H_4SO_3Na$), Nonionic Surfactant, Sodium tripolyphosphate, Proteases. Water to powder ratio, 100:15) for various durations (100, 200, or 300 h). The samples were air-dried before. Treated-samples were then prepared as previously described and the viscosity of the fabric samples to TPU films measured by RPR at 100°C.

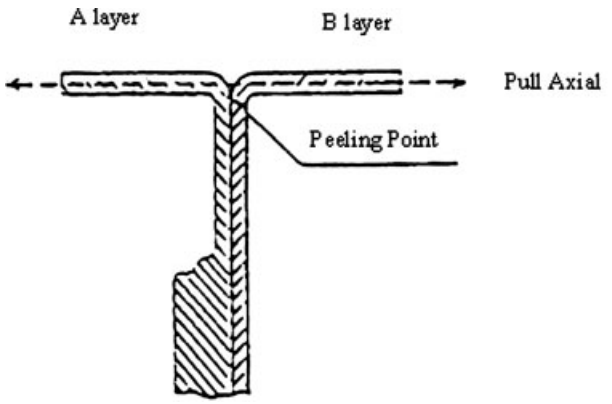

Figure 3 Set-up Universal Tensile Tester for peeling strength test.

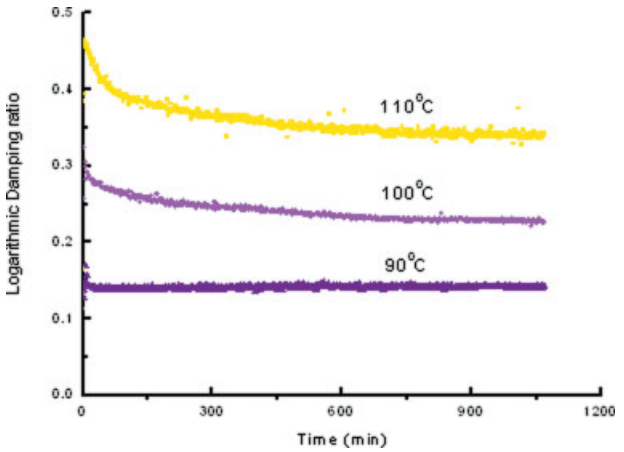

Figure 4 Effect of temperatures on the viscosity of PET/TPU samples. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

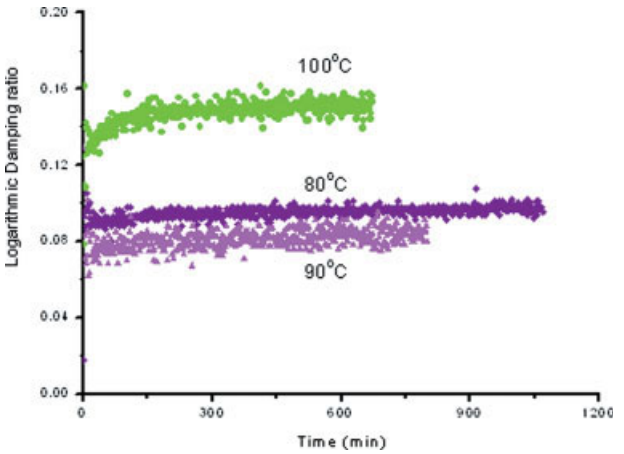

Figure 5 Effect of temperatures on the viscosity nylon/TPU samples. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Macroscopic peeling strength study by universal tensile tester

Effect of environmental factors on peeling strength of PET/TPU and nylon/TPU

New sets of samples were cut into size and treated with UV and water as detailed in ''Microscopic Viscosity Study''. Following the treatments, a thermal compression machine (Hydraulic Industrial, Taiwan) was used to laminate PET/TPU and Nylon/TPU samples for 10 min at 100°C, 100 psi. In accordance to Chinese National Standards (CNS 3557, method of test for adhesion of vulcanized rubber; equivalent to ISO 36 and ISO 6133), the peeling strength of laminated samples was measured with Universal Tensile Tester (unit: kgf/2.5 cm at a 180 angle and peel rate of 50 mm/min, Orientec, Tokyo, Japan) as illustrated in Figure 3.

RESULTS AND DISCUSSION

Currently, a pulling machine such as Universal Tensile Tester is currently used to measure the adhesive property of fabric laminates. Universal Tensile Tester measures the peeling strength required to separate the hot-melt treated materials from each other.[1,18] To evaluate the effectiveness of RPR as a new testing method, Universal Tensile Tester was used as a comparison to measure the effects of UV exposure and water immersion on both PET/TPU and nylon/TPU samples.

In textile industry, TPU are used in the process of coating and laminating with fabrics to form a fabric laminate. To test the adhesive property of the fabric laminate under various environmental influences is very important.[18–27] Traditionally, Universal Tensile Tester is used to evaluate the adhesive property of TPU and fabrics. Recently, a newly developed testing machine known as RPR has been used to test the curing behavior of various materials in response to UV effects.[27,33–36] To the best of our knowledge, fabric laminate adhesive test has not yet been performed using RPR. By using RPR to measure the adhesive strength of TPU to PET and nylon in response to various environmental factors, and compare the results with the traditional testing method this study aimed to assess the performance of RPR in the hope to establish a more effective method for fast-evaluating adhesive property of fabric laminates.

As a latest developed testing machine, RPR measures the adhesive property of fabric laminates differently from traditional testing machine such as Universal Tensile Tester. Specifically, the traditional method employs a hot-melt process in which TPU is melted and glued to sample fabric. Universal Tensile Tester is then used to measure the physical force

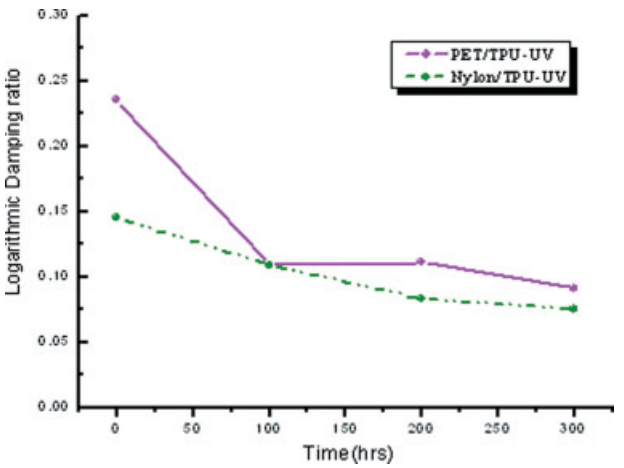

Figure 6 Effect of UV exposure on the viscosity of PET/TPU and nylon/TPU samples. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

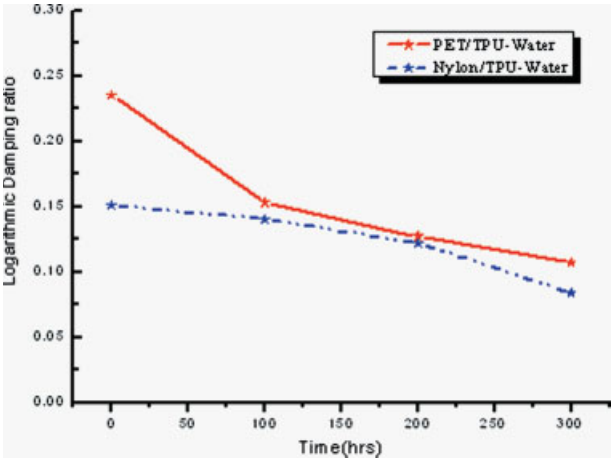

Figure 7 Effect of water immersion on the viscosity of PET/TPU and nylon/TPU samples. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

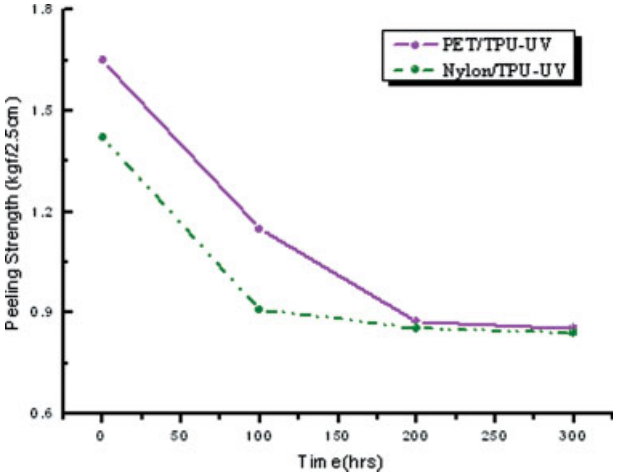

Figure 8 Effect of UV exposure on peeling strength of PET/TPU and nylon/TPU samples. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

required to pull the two materials apart. RPR, in contrast, does not require TPU to be melted and glued to the sample fabric. Instead, Rigid-Body Pendulum vibrates to create resistance between the interfaces of TPU film and sample fabrics. RPR is then able to measure the maximum viscosity of the tested materials under various temperatures and conditions without physically damaging the fabrics during testing while using minimal amounts of samples.

Effect of temperature on microscopic adhesive property of PET/TPU and nylon/TPU

To determine the effect of temperature on microscopic viscosity between TPU and fabrics, PET/TPU fabric laminate was heated to 90, 100, and 110°C on the RPR specimen mount. It was observed from Figure 4 that the logarithmic damping ratio of PET/TPU sample increased with temperature. At 90°C, the viscosity of PET/TPU is quite low. PET/TPU fabric laminate achieved its maximum logarithmic damping ratio at 110°C but PET/TPU become melting flow. Therefore, the optimal testing temperature in this study was set at 100°C. To find out the optimal temperature for the testing process PET/TPU was neared at tree temperatures, 90, 100, and 110°C. The viscosity between PET/TPU increased as the temperature increased with the maximum viscosity achieved at 110°C. TPU film, however, had a melting flow at 110°C. Thus, 100°C was chosen to be a more appropriate testing temperature.

Following the determination of maximum testing temperature for TPU at 100°C, a second sample, nylon/TPU fabric laminate was heated to 80, 90, and 100°C. Its viscosity was then measured (Fig. 5). In contrast to PET/TPU sample, the viscosity between nylon and TPU was higher at 80°C than at 90°C with the maximal viscosity achieved at 100°C. This slight fluctuation of viscosity may be due to the higher moisture content of nylon in comparison to PET. At 80°C, the interaction between water molecules of nylon and TPU film resulted in a stronger viscosity than at 90°C. When the temperature reached 90°C, the moisture content of nylon began to evaporate reducing the fraction between water molecules and TPU film; hence the slight decrease in viscosity. As the temperature reached 100°C, the influence of high moisture content of nylon on the viscosity diminished as TPU film reached its optimal temperature creating strong interaction with nylon achieving in the maximal viscosity. These observations suggest that the moisture content of the fabrics should be taken into account when the adhesive property of materials is tested by RPR.

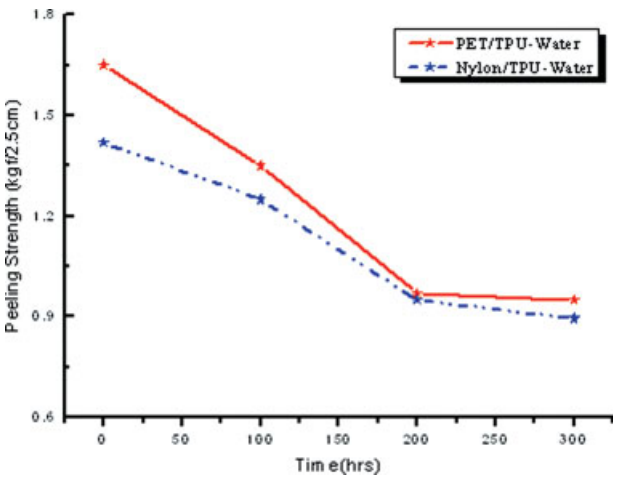

Figure 9 Effect of water immersion on peeling strength of PET/TPU and nylon/TPU samples. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

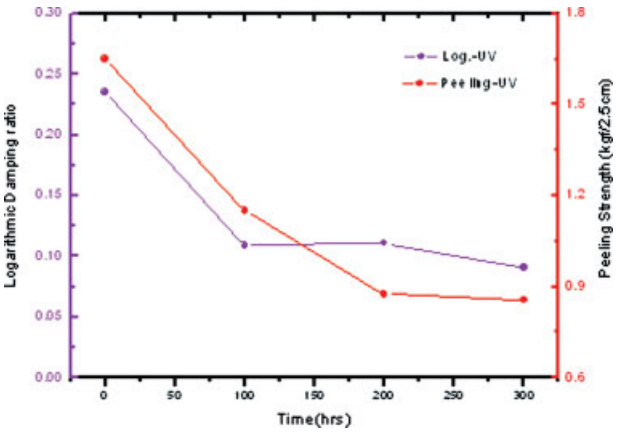

Figure 10 Correlation analysis of the viscosity and peeling strength (UV-exposed PET/TPU samples). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

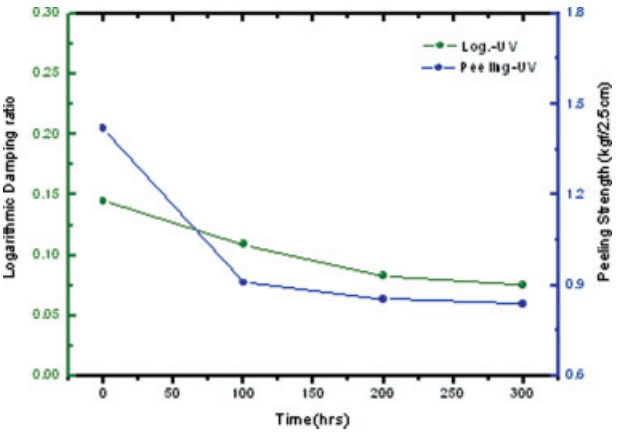

Figure 11 Correlation analysis of the viscosity and peeling strength (UV-exposed nylon/TPU samples). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Effect of UV exposure on microscopic adhesive property of PET/TPU and nylon/TPU

To study the possible profound effect of UV lights on fabric laminates, PET/TPU and nylon/TPU fabric laminates were exposed to UV lights for 100, 200, and 300 h. Figure 6 shows that at 0 h, the viscosity between PET and TPU film was stronger than that of nylon, and TPU film indicated by higher logarithmic damping ratio of PET/TPU fabric laminate. This suggests that TPU film adhered better to PET than to nylon at the initial stage of the study. One possible explanation to this is that the two fabrics had different surface moisture contents resulting in stronger friction force of PET to TPU film creating stronger viscosity.[30–35] This advantage in adhesion of PET to TPU film, however, diminished after UV exposure as the viscosity of both UV-treated samples was reduced sharply. This reduction in viscosity was particularly notable in PET/TPU fabric laminate as its logarithmic damping ratio reduced by 53.6% after 100 h of UV exposure, while nylon/TPU fabric laminate reduced by 24.8%. After 300 h of UV exposure, logarithmic damping ratio of PET/TPU sample diminished by 61.3% and nylon/TPU sample reduced by 48.3%. This suggests that nylon/TPU sample resisted UV exposure better than PET/TPU sample. The possible explanation to this may be that UV exposure caused more damage to PET than nylon at the molecular level leading to larger cracking and interface decay.

Effect of water immersion on microscopic adhesive property of PET/TPU and nylon/TPU

Water immersion and UV exposure cause interface decay similar trends on the viscosity of PET and nylon fabrics to TPU film. Figure 7 shows that after being immersed in water for 100 h, logarithmic damping ratio of PET/TPU fabric laminate had a small reduction of 34.9% compared with nylon/TPU fabric laminate, which had a much greater reduction of 57.0%. After 300 h of water immersion, PET/TPU sample had greater decrease in logarithmic damping ratio (54.5%) compared with nylon/TPU sample (44.4%). Thus, the viscosity of PET/TPU and nylon/TPU samples did not differ greatly after being immersed in water for more than 100 h.

Effect of UV exposure on macroscopic peeling strength of PET/TPU and nylon/TPU

Figure 8 indicates that the peeling strength required peeling the fabrics off the TPU film was reduced as the UV exposure increased. After 100 h of UV treatment, the peeling strength required to separate PET/TPU sample decreased by 30.3%, while the peeling strength needed for nylon/TPU sample decreased

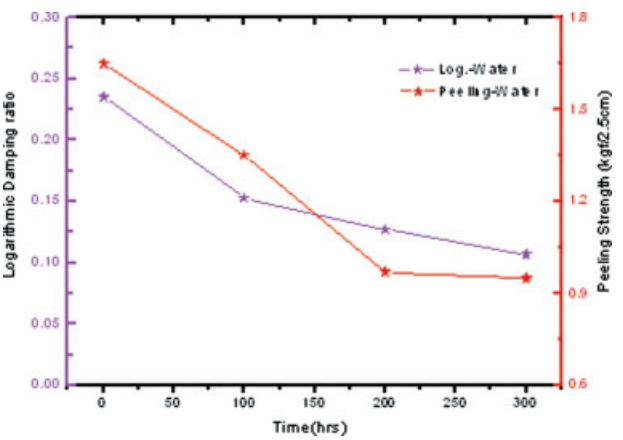

Figure 12 Correlation analysis of the viscosity and peeling strength (water-treated PET/TPU samples). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

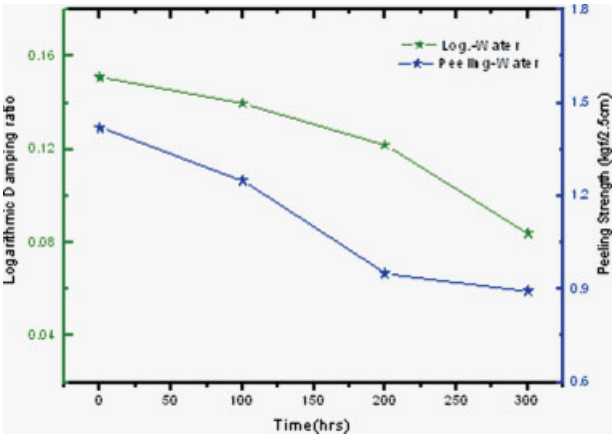

Figure 13 Correlation analysis of the viscosity and peeling strength (water-treated nylon/TPU samples). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

by 35.9%. The peeling strength required varied little after 200 h of UV exposure and as a whole; the reduction in peeling strength after 300 h of UV exposure was 47.0 and 39.8% for PET/TPU and nylon/TPU samples, respectively.

Effect of water immersion on macroscopic peeling strength of PET/TPU and nylon/TPU

Water immersion produced a similar decay trend, as the UV exposure had on the peeling strength required separating PET or nylon fabric from TPU film. As shown in Figure 9, after 100 h of water immersion, the peeling strength required to separate PET or nylon from TPU film was reduced by 12.0 and 18.2%, respectively. The reduction in peeling strength reached a plateau after 200 h. As a whole, water treatment of 300 h resulted in decreased peeling strength (PET/TPU by 41.2% and nylon/TPU by 33.1%).

Correlation analysis

Tensile test is to evaluate adhesion fracture from macroscopic perspective. There are three well-known modes for adhesion fracture: interfacial mode, cohesion mode, and mixed mode. The present study employed the interfacial mode to perform peeling test on PET/TPU and TPU/nylon. The interfacial mode adhesion fracture behavior of PET/TPU and TPU/nylon is associated with miscibility and wettablity of the tested materials. Based on the intermolecular interaction between TPU film and tested fabric, the above adhesion fracture behavior can be explained through microscopic Rheological behavior.

RPR evaluates viscosity behavior from a microscopic point of view. The present study tested adhesive property of PET/TPU and nylon/TPU using both tensile test and PRP. A strong correlation was established between the two test methods, suggesting PRP may be used to assess adhesive property of PET/TPU and nylon/TPU in additional to the more traditional tensile test method.

The goal of this study was to investigate RPR as an effective method in testing fabric laminate properties, in these case adhesive properties, in comparison to traditional testing method such as Universal Tensile Tester. In contrast, Universal Tensile Tester uses the peeling strength to separate samples as the means of assessing adhesive property. Since the two machines employ different methods of testing, a direct comparison of the data generated by these two testing machines was not possible; instead a correlation analysis was performed. Figures 10 and 11 respectively, summarize the adhesive property of PET/TPU and nylon/TPU in response to UV exposure measured by both testing machines. Although a direct comparison was not possible, RPR was able to produce a trend showing viscosity degradation of samples in response to UV exposure. This trend was similar to the trend that Universal Tensile Tester produced to show a reduction in peeling strength required to separate tested fabric laminates. This similarity in viscosity/peeling strength reduction of the samples was particularly pronounced after 100 h of UV exposure. In regards to the effect of water immersion on PET/TPU and nylon/TPU fabric laminates, results produced by RPR were also in a similar decay trend compared with those of produced by Universal Tensile Tester (Figs. 12 and 13).

TABLE I
Correlation Analysis of UV-Exposed Samples[a]

	Logarithmic damping ratio (Δ)		Peeling strength (kgf/2.5 cm)	
UV exposure time (h)	PET/TPU	Nylon/TPU	PET/TPU	Nylon/TPU
0	0.235	0.145	1.650	1.420
100	0.109	0.109	1.150	0.910
200	0.111	0.083	0.875	0.875
300	0.091	0.075	0.855	0.855

[a] $r = 0.9325$ for LDR vs. Peeling Strength of Nylon/TPU-UV; $r = 0.9464$ for LDR vs. Peeling Strength of PET/TPU-UV.

TABLE II
Correlation Analysis of Water-Immersion Samples[a]

	Logarithmic damping ratio (Δ)		Peeling strength (kgf/2.5 cm)	
Water immersion time (h)	PET/TPU	Nylon/TPU	PET/TPU	Nylon/TPU
0	0.235	0.151	1.650	1.420
100	0.153	0.140	1.350	1.250
200	0.127	0.122	0.970	0.950
300	0.107	0.084	0.950	0.895

[a] $r = 0.9565$ for LDR vs. Peeling Strength of PET/TPU-Water; $r = 0.8889$ for LDR vs. Peeling Strength of Nylon/TPU-Water.

To further investigate the relationship between the data obtained from these two testing machines, correlation coefficient was calculated. When the logarithmic damping ratios and peeling strength curves of the UV-treated PET/TPU and nylon/TPU samples were compared, the correlation coefficient was 0.9464 and 0.9325, respectively, (Table I). The correlation coefficient of PET/TPU and nylon/TPU samples, which have undergone the water immersion was 0.9565 and 0.8889, respectively, (Table II). These analyses have demonstrated that results obtained from RPR were positively correlated with the results obtained by Universal Tensile Tester in a linear relationship. Based on the linear regression shown in Figure 14 an equation $Y = 5.6194X + 0.3949$ was derived. With the goodness of fit being 84.95%, we speculated that this linear equation summarizes the relationship between the data produced by RPR and Universal Tensile Tester. Potentially, this equation may be used to estimate the expected values, based on the experimentally obtained data from either machine.

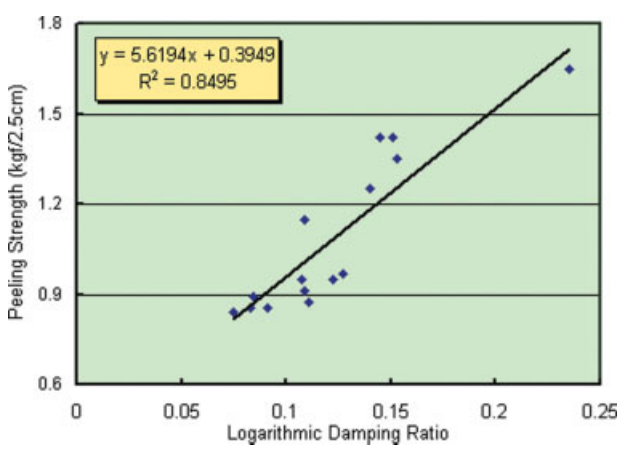

Figure 14 A linear relationship between logarithmic damping ratio and peeling strength measured by RPR and Universal Tensile Tester respectively. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

CONCLUSIONS

In this study, RPR and Universal Tensile Tester were used to assess the adhesive property of two fabric laminates, PET/TPU and nylon/TPU in response to UV exposure and water immersion. Data measured by both machines indicate a reduction in viscosity/peeling strength of the samples tested after UV and water treatments. Based on the findings, it has been concluded that firstly the data measured by RPR were correlated with the data measured by Universal Tensile Tester, showing a strong positive correlation relationship eventuated in the deriving of a linear equation. The equation may potentially use to estimate the expected values, based on the experimentally obtained data from either machine.

Secondly, RPR may be an easier testing method to evaluate the adhesive property of fabric laminates in comparison to the traditional method, Universal Tensile Tester. With a more advanced design, RPR is able to determine the optimal testing temperature of the procedure in less time, which will help to improve the precision and reliability of a study. RPR utilizes smaller amount of samples and does not physically damage the fabrics as Universal Tensile Tester does by melting and gluing TPU film onto tested fabrics. RPR also is capable of measuring and recording continuously the impact that temperature variation has on the adhesive property of tested samples.

In conclusions, RPR can continuously observe and record viscosity behavior form low temperature to high temperature, not only were the differences in reaction speed observed, but also the differences in crosslinking and other data in the softening process when the balanced time was achieved during the oscillations procedure. With a shorter testing time and less sample required, data obtained from RPR were 84.95% comparable with data obtained from Universal Tensile Tester. Hence, RPR has been shown to be a new and fast method to test adhesive property of PET/TPU and nylon/TPU samples. Future studies, however, are needed to evaluate if RPR produces reliable results when measuring other aspects of fabric laminate properties.

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