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PEEL STRENGTH IMPACT OF FUNCTIONALIZED POLYETHYLENE TO THERMOPLASTIC POLYURETHANE CALENDERED ON A POLYESTER FABRIC

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Abstract:

Many producers use the extrusion calendering process for thermoplastic polyurethane coated fabrics. However, a technological lock is the ability to get a good adhesion of the coating with the fabric. Producers could increase the coating extrusion temperatures but TPU have a narrow extrusion temperatures range making it difficult to extrude. One solution is to make a blend with another polymer which has a higher extrusion temperature range. In the present work, the studies of the addition of LDPE and LLDPE-g-Ma in polyurethane coating on the tensile strength of the sheet and on the peel strength with a polyester fabric have been studied as well as the influence of the extrusion temperature. SEM observations and viscosity measurements have been performed to understand the behaviour of the different blends. Results show that extrusion temperature and penetration depth of the coating in the fabric have an influence on the peel strength.

Keywords: Polyurethane, Fabric, Peel strength, Calendering, Polymer

INTRODUCTION

Coated technical textiles are widely used for several applications like paragliders or inflatable boats and the demand is still on the rise. These light technical textiles are usually manufactured with a polyester fabric and a PolyVinyl Chloride (PVC) matrix. However, environmental constraints force the manufacturers to find a substitute material for the PVC which is harmful and difficult to recycle. For this purpose Thermoplastic PolyUrethane (TPU) is a good substitute material for the PVC. Depending on its formulation and the components, TPU can have good properties such as UV resistance (1) and abrasion resistance (2). Due to these properties, TPU is widely used for coated textiles and to achieve the quality of natural leather. Despite all these good properties, there is still a technological lock. Some industrials report that they are recalcitrant to use polyurethane sheets for coated textile because of very low peel strength of the sheet on the polyester fabric after calendering.

Six theories have been proposed to explain the different mechanism of adhesion: mechanical interlocking (3), wetting (4), diffusion (5), electrostatic (6), chemical (7) and weak boundary layer (8). All these theories show that the adhesion between two materials is linked with the interface as outlined by Mittal (9). Further studies explain that the quality of adhesion between the fabric and the matrix is a key parameter to obtain good mechanical performances of the composite (10). As a consequence, several treatments have been developed to enhance the quality of the interface. Previous research used different treatments for the fabric such as atmospheric air treatments to modify the surface energy of the fabric and increase the adhesion of the coating. For example, Leroux et al. showed that the adhesion of a silicon resin on a polyester fabric after atmospheric air plasma treatment has been multiplied by two (11). However Novak et al. showed that the shelf-life of this treatment for a polypropylene material with polyvinyl acetate was only about 50 days due to the loose of the surface oxidation (12). Other research used corona treatments (13) or chemical treatments (14) to increase the wettability of the fabrics.

There is less research about the plastic sheet that is calendered on the fabric. A solution to enhance the adhesion of the sheet on the fabric could be to modify the sheet that is extruded before being calendered. One possibility is to increase the extrusion temperature in order to modify the viscosity and the surface energy. The problem is that TPU has a narrow range of extrusion temperatures and an increase of only 5°C can generate a drop in the viscosity of the polymer making it impossible to calender on a fabric. The objective of the present study is to propose a blend with Low Density PolyEthylene (LDPE) with the aim to extrude the sheet at higher temperatures. Because of their high difference of polarities and their high interfacial tension, Polyurethane and Polyethylene are two immiscible materials. However, previous researches explain that it is possible to have a compatibility if the PE is grafted with maleic anhydride (LLDPE-g-Ma) (15). These compatibilizers are capable to stay at the interface and entangling with both sides. The final material is then prepared by calendering the sheet of TPU/LDPE blend on a polyester fabric. According to the literature, there is no previous research about the influence of this blend on the adhesion on a polyester fabric.

The value of the adhesion of the sheet on the fabric is the main proof of the influence of the blend. For this study, the sheet viscosity, the miscibility of the LDPE and PE-g-Ma in the TPU, the penetration depth of the coating in the yarns of the fabrics and the FTIR analysis are used to analyse and explain the results of the adhesion.

MATERIALS AND METHODS

1. Materials Characterization

The coating has been calendered on the polyester fabric described in the Table 1.

Table 1: Main properties of the fabric

| Composition | Polyester |
|---------------------------------------|-----------|
| Weaving | Plain |
| Additives on the surface | No |
| Number of yarns per cm : weft | 18 |
| Number of yarns per cm: warp | 18 |
| Thickness (μm) | 170 |
| Number of filaments per yarn | 48 |
| Filament diameter (μm) | 23 |
| Yarn count (g/km) | 280 |
| Mechanical properties: weft (daN/5cm) | 155 |
| Mechanical properties: warp (daN/5cm) | 155 |

This fabric has been woven without the use of any additives like sizings on the surface of the yarns to avoid a decrease of the wetting ability.

Several blends have been realized with Low Density Polyethylene (LDPE), Thermoplastic PolyUrethane (TPU) and Linear Low Density Polyethylene Grafted Maleic Anhydride (LLDPE-g-Ma). References and properties of LDPE, LLDPE-g-Ma and TPU are gathered in Table 2.

Table 2: Main properties of LDPE (Low Density Polyethylene) and LLDPE-g-Ma (Linear Low Density Polyethylene grafted maleic anhydride) and TPU (Thermoplastic Polyurethane)

| | LDPE LD 171 BA | LLDPE-g-Ma OREVAC OE825 | TPU IROGRAN A 90 P 5055 DP (Aromatic Polyether) |
|--------------|------------------------|----------------------------|---|
| Manufacturer | EXXON MOBILE® | OREVAC® by ARKEMA | HUNTSMAN® |
| Density | 0.929g/cm ³ | 0.913g/cm ³ | 1.2g/cm ³ |
| Additives | no | Maleic anhydride | no |

All the blends prepared are presented in the Table 3.

Table 3: Composition of the prepared blends

| Sample number | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
|---------------|------|----|----|------|----|----|------|------|----|-----|-----|-----|----|----|-----|
| % TPU | 83.4 | 81 | 81 | 77.5 | 85 | 71 | 77.5 | 83.4 | 85 | 100 | 0 | 0 | 71 | 71 | 0 |
| % LDPE | 16.6 | 19 | 19 | 22.5 | 15 | 29 | 22.5 | 16.6 | 15 | 0 | 100 | 100 | 0 | 26 | 0 |
| % PE-g-Ma | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 29 | 3 | 100 |

2. Experimental Methods

2.1 Blend Preparation

The PE/TPU blend has been prepared in two steps. The pellets of TPU and LDPE (and/or LLDPE-g-Ma) have been well mixed in a container.

2.2 Extrusion – Calendering Process

Extrusion has been performed with a laboratory-scale extruder Polylab system composed of a HAAKE RheoDrive4 motor coupled with a HAAKE Rheomex 19/25 OS single screw extruder with a Maddock mixer. The system was piloted by PolySoft OS software to set and control temperature zones and screw speed. The extruder unit was equipped with a fish-tail designed die of 100mm wide and 450 μ m thick to process the molten polymer into a film. The extruder was connected to the air network which provides ambient temperature air to cool the hopper zone. The calendering was performed on only one face of the fabric using a 3-roll laboratory calender from THERMO SCIENTIFIC. The rolls were 200mm wide and were cooled with a HAAKE Phoenix II P1 thermostat (THERMO SCIENTIFIC) with oil and regulation pump speed. All the process parameters and extrusion temperatures for extrusion-calendering are respectively given in Table 4 and Table 5.

In the purpose to test separately the mechanical performance of the film that was calendered on the fabric, the same film was prepared with the same parameters.

Table 4: Process parameters

| Parameters | Value |
|--|---------------------------------------|
| Die gap | 450 μ m |
| Die temperature | [174°C; 209°C] (+/-1°C) (see Table 5) |
| Extrusion speed | 60 rpm |
| Calendering speed | 6 rpm |
| Temperature of the thermoregulated rolls | 40°C |
| Distance between the die and the rolls | 20mm |

Table 5: Extrusion temperatures of the prepared films (in °C)

| Sample number | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
|---------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Hopper zone | 211 | 180 | 202 | 196 | 202 | 196 | 216 | 187 | 180 | 170 | 180 | 196 | 196 | 196 | 170 |
| Zone 1 | 211 | 180 | 202 | 196 | 202 | 196 | 216 | 187 | 180 | 172 | 180 | 196 | 196 | 196 | 172 |
| Zone 2 | 206 | 175 | 197 | 191 | 197 | 191 | 211 | 182 | 175 | 174 | 175 | 191 | 191 | 191 | 174 |
| Die | 199 | 174 | 196 | 190 | 196 | 190 | 210 | 181 | 174 | 175 | 174 | 190 | 190 | 190 | 175 |

The different heating zones of the extruder are presented in Figure 1.

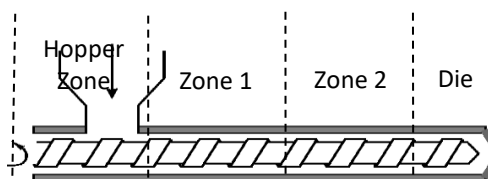


Figure 1: Scheme of the extruder and temperature zones

2.3 Analysis of the Peel strength

The peel strength of the coating sheet on the fabric has been determined by a peel test carried on a Zwick Z010 tensile machine according to the standard NF EN ISO 2411. The 50 mm width coating sheet and the fabric were clamped separately on the machine with a distance of 50 mm between grips. A crosshead speed of 100 mm/min and a 0.5 kN cell were chosen. During the test the force was recorded as a function of displacement thanks to TestXpert® II software (Zwick). Reported data are the average of five samples.

2.4 Analysis of the mechanical properties of the sheet

Tensile strength of blends sheets was measured during a tensile test on a Zwick Z010 with a crosshead speed of 500 mm/min and a 0.5 kN cell. Strip-shaped samples were prepared with a cutting press. The

length between grips and width of samples is 40 mm and 10 mm respectively and the thickness is measured for each sample and varies between 180-220 μ m.

During the test, the force was recorded as a function of the displacement thanks to TestXpert® II software (Zwick). Reported data are the average of 10 samples. The tensile strength was obtained by dividing the force applied at the breaking by the initial section of the sample.

2.5 Analysis of the shear viscosity of the blend

The dynamical rheological measurements were performed on disks using a strain controlled rheometer ARES (TA Instrument) equipped with a 25 mm parallel plates geometry in continuous shear mode at the same temperatures than those used for the different calendering tests. According to prior experiments consisting in determining the linear viscoelastic domain for which the behavior of the polymer does not depend on the strain, the frequency sweep at strain was kept at $\epsilon=3\%$ and the pulsation ω was in the range of 0.1 to 100 rad/s. Nitrogen was used to decrease the ageing of blends. Disk samples of 1.8 mm thick and 25mm wide were prepared by injection. The gap was set at 1.5mm. The result is the average value of three samples.

2.6 Analysis of the coated textile sections

The section of the coated textile has been analyzed with a Scanning Electron Microscope using the detection of backscattered electrons and a magnification of x500.

The penetration of the coating on the fabric was measured as following. Red arrows in Figure 2 indicate the depth of coating penetration ($\pm 2\mu$ m):

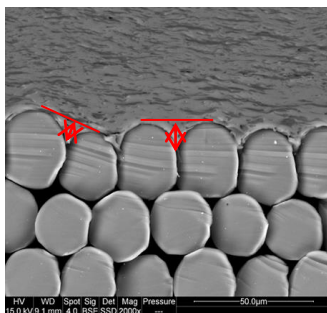


Figure 2: Measurement method of the coating penetration on the fabric

2.7 Analysis of the Chemical Composition

Infrared measurements at room temperature were performed on a Perkin–Elmer Spectrum One FT-IR (Fourier Transformed Infrared) Spectrometer with 32 scans and a resolution of 2 cm^{-1} in the absorption mode to determine the chemical composition of the different blends.

RESULTS

1. Peel Strength of TPU/LDPE Blends

According to Table 6, peel strengths of neat TPU do not exceed 7N/50mm. For the blends given in Table 6, an increase between 200% can be highlighted for experiment number 9 (15% of LDPE and extrusion temperature 174°C) and 430% for experiments 4 and 6 (respectively 23% and 29% of LDPE and extrusion temperature 190°C). However neat LDPE (at both 174°C and 175°C) also exhibit a very low peel strength which means that the increase of the peel strength of the blends is not only due to the LDPE.

For a same amount of LDPE, an increase of the extrusion temperature seems to increase the peel strength. For example, for a same amount of 19%wt of LDPE (samples 2 and 3) but different extrusion temperatures (respectively 174°C and 196°C), the peel strength is doubled (13.0N/50mm and 27.4N/50mm respectively). The same trend is observed with samples 1 and 8, while the opposite trend is observed with samples 4 and 7. This can be explained by the very high die temperature employed for sample 7 that may cause a degradation of the blend. This is further correlated to an important decrease of the tensile strength.

Conversely and for a same extrusion temperature the amount of LDPE seems to have slight influence on the peel strength. For example, experiments 2 and 9 were both performed at 174°C with respectively 19%wt and 15%wt of LDPE but the peel strengths are identical. This observation is also true for experiments 4 and 6

and experiments 3 and 5. The complete substitution of LDPE by LLDPE-g-Ma leads to a decrease of the peel strength (experiment 13). The substitution of only 3% of LDPE by LLDPE-g-Ma does not have a significant influence on the peel strength (experiment 14).

Table 6: Peel strength of neat TPU, neat LDPE and TPU/LDPE blends

| Sample number | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
|------------------------|------|----|------|----|------|----|------|----|----|----|-----|-----|------|------|-----|
| Peel strength (N/50mm) | 22.3 | 13 | 27.4 | 30 | 29.1 | 30 | 21.2 | 17 | 13 | 7 | 1.8 | 4.5 | 16.5 | 30.8 | 4.9 |

2. Tensile Strength of TPU/LDPE Blends

Tensile strengths of the different blends are displayed in Table 7. Tensile strengths of neat TPU extruded at 175°C is about 23 MPa while the tensile strength of neat LDPE depends of the process temperature (24.6MPa and 42.6 MPa for extrusion temperatures of, respectively, 174°C and 190°C. The difference can be due to the partial fusion of LDPE pellets at a temperature of 174°C while the fusion is complete at 190°C). Excepted for experiment 9, the tensile strength of all blends is lower than the tensile strength of neat TPU and neat LDPE which means that there is an incompatibility between TPU and LDPE. This is consistent with the literature (15). For an extrusion temperature of 210°C, the film seems to be degraded. The corresponding tensile strength is only 11.6 MPa while it is more than 14 MPa for all other blends. It is important to note that the tensile strength of TPU/LDPE blends seems to depend on temperature. Indeed, except for experiment 3, an increase of the extrusion temperature leads to a decrease of the tensile strength. The addition of only 3% of LLDPE-g-Ma in the blend leads to an increase of the tensile strength (experiment 14). With the complete substitution of LDPE by LLDPE-g-Ma (experiment 14), the tensile strength reaches 37.9MPa which is a strong increase in comparison (at the same extrusion temperature) with the uncompatibilized blend (experiment 6).

Table 7: Peel strength of neat TPU, neat LDPE and TPU/LDPE blends

| Sample number | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
|------------------------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|
| Tensile Strength (Mpa) | 14.3 | 22.5 | 17.9 | 13.9 | 17.8 | 18.6 | 11.6 | 18.7 | 25.5 | 23.1 | 24.6 | 42.6 | 37.9 | 23.5 | 28.1 |

3. Analysis of The Chemical Composition

The Ma group can be seen between around 1700 cm^{-1} and 1800 cm^{-1} as explained on previous research on PP-g-Ma (16). Figure 3 shows the FTIR spectrum of samples 2 and 6. Although the peel strength is very different (sample 6 displayed a peel strength almost 3 times higher than sample 2), the two spectrums are similar. No difference was observed on the FTIR spectrums of the samples 1 to 9. The FTIR spectrum of experiments 6, 13 and 14 is presented in Figure 4 and the same conclusion can be made. The percentage of Ma in the blends 13 and 14 is so weak that it is almost not visible on the FTIR experiment except for the peak around 1730 cm^{-1} . The large peak around 1700 cm^{-1} is a peak from TPU corresponding to the urethane group (C=O) (17) and cannot be attributed to Ma.

In conclusion to these analyses, no significant difference on the FTIR spectrum has been observed between all the blends. It may be that there is no new bonds creation by mixing TPU and LLDPE or LLDPE-g-Ma so the noted better adhesion is not due to a chemical link between the fabric and the coating.

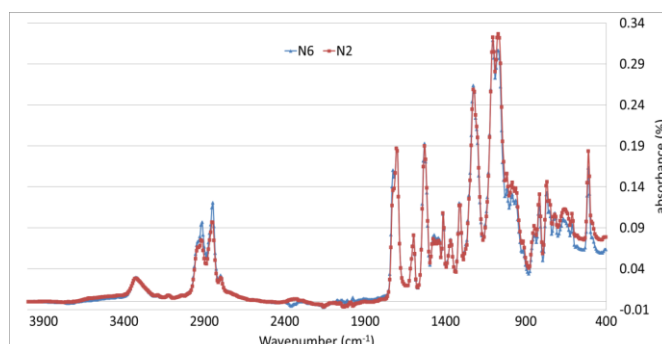


Figure 3: FTIR of experiment 2 and 6

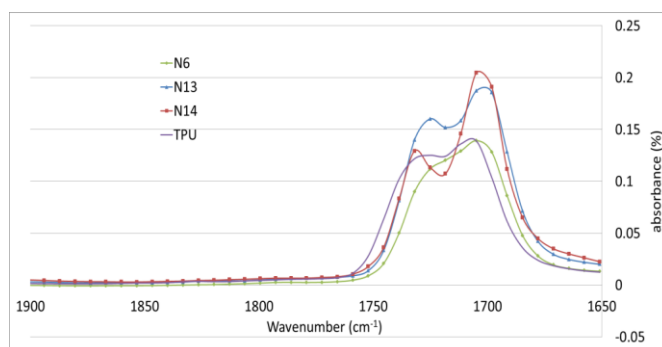


Figure 4: FTIR spectrum (1650-1900cm⁻¹) of neat TPU and experiment 6, 13 and 14

4. Analysis of The Shear Viscosity of The Blend

The viscosity of the sheet is important because the ability of the polymer to penetrate inside the yarn depends of this viscosity. The shear viscosity given in Table 8 is the viscosity of the different blends at the corresponding extrusion temperatures for a shear rate between 10s⁻¹ and 100s⁻¹. These shear rates are those corresponding to the calendering process according to the literature (18).

At the same die temperature, the viscosity of neat LDPE and neat LLDPE-g-Ma is 7 times higher than the viscosity of neat TPU. Although the viscosity of LLDPE-g-Ma is lower than LDPE ones, the blend of LLDPE-g-Ma/LDPE/TPU (experiment 14) has a viscosity twice higher than that of experiment 6 for a same temperature of process. This must be linked with the miscibility of the different materials and it should be compared with the mechanical performance and the morphology of the blends. For the same LDPE/TPU blends, the higher the process temperature is, the lower is the viscosity. For example for an amount of 22.5%wt of LDPE and a shear rate of 10s⁻¹, the viscosity is about 421 Pa.s for a temperature of 190°C and 47 Pa.s for a temperature of 210°C.

Table 8: Shear rate viscosities of neat TPU, neat LDPE, neat LLDPE-g-Ma and of the different blends at the corresponding processing temperatures and for a shear rate between 10s⁻¹ and 100s⁻¹

| Sample number | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
|------------------|-----|-----|-----|-----|-----|-----|----|-----|-----|-----|------|------|-----|-----|------|
| Shear rate | 110 | 481 | 119 | 222 | 112 | 421 | 47 | 447 | 487 | 237 | 3800 | 2900 | 445 | 884 | 2980 |
| viscosity (Pa.s) | 93 | 284 | 96 | 166 | 99 | 267 | 43 | 276 | 335 | 188 | 960 | 700 | 248 | 485 | 1300 |

5. Analysis of the coated textile sections

The analysis of the coated textile section gives important information about the depth of penetration of the polymer between the filaments of the yarns that compose the fabric. Table 9 gives the depth of penetration for each experiment. The coating has a better penetration when extruded at high temperature especially if the temperature is above 190°C (experiments 1, 3, 5 and 7). It could be due to a difference of surface energy or viscosity. However it is important to notice that the diameter of a filament is 23 µm so the coating never penetrates inside the fabric but always keeps on the surface.

Table 9: Penetration depth of the coating for each blend in the fabric

| Sample number | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
|------------------------|----|---|----|----|---|---|----|---|---|----|----|----|----|----|----|
| Penetration depth (µm) | 13 | 6 | 11 | 10 | 9 | 7 | 14 | 8 | 7 | 2 | 5 | 6 | 8 | 6 | 2 |

ANALYSIS

1. Analysis of The Peel Strength Increase

FTIR shows that no new chemical bonds have been created in any of the different blends. Indeed, despite the large difference of peel strength, all FTIR spectrums are identical. It means that the peel strength difference is not due to the creation of new chemical bonds between the fabric and the coating.

Regarding the theories of wetting and mechanical interlocking, the observations of the coating penetration depth in the fabric give good information. As expected, the lower is the viscosity, the better is the penetration. According to the results, peel strength seemed to increase when coating penetration was higher than 7µm. For example, on sample 7, the penetration is about 14µm and the peel strength is 21.2 N/50mm while penetration is only 2µm for sample 10 and the corresponding peel strength 1.8 N/50mm. But this trend could not be generalized; in fact sample 6 displayed 30 N/50 mm peel strength with a penetration depth of only 7µm. The conclusion is that the coating penetration depth in the fabric, and the related viscosity, has a strong influence on the peel strength but is not the only parameter involved.

Actually, the temperature seems to have a strong impact on the peel strength. The temperature dependence of surface energy has been shown by previous papers (19). This modification of surface energy could lead to a better affinity between the fabric and the coating. As said previously, for a same amount of LDPE, an increase of the extrusion temperature leads to an increase of the peel strength until a maximum value for a temperature of 190°C. For higher temperatures (experiments 1, 3, 5 and 7), the peel strength decreases as a consequence of the polymer degradation, which could be observed by the decrease of the tensile strength.

2. Maleic Anhydride Influence

As observed previously, the complete substitution of LDPE by LLDPE-g-Ma has a negative impact on the peel strength, but a substitution of only 3%wt of LDPE by LLDPE-g-Ma did not degrade this property while it increases its tensile strength. The LLDPE-g-Ma is needed to get good peel strength and also good tensile properties.

One interesting point is the difference of viscosity between experiment 6, 13 and 14 which have the same amount of LDPE or LLDPE-g-Ma. At the same temperature, LLDPE-g-Ma has a lower viscosity than LDPE. But also for a same temperature, the viscosity of the blend 14 made of 26%wt of LDPE and 3%wt of LLDPE-g-Ma is 2 times higher than the viscosity of the blend 6 made with 29%wt of LDPE and blend 13 with 29%wt of LLDPE-g-Ma. This increase of viscosity means that there is a good compatibility between LDPE and LLDPE-g-Ma. The complete substitution of LDPE by LLDPE-g-Ma have no impact on the viscosity of the blend at 190°C as it was found by comparing experiment 6 and 13.

CONCLUSION

In the present paper, the impact on peel strength of the addition of low density polyethylene and linear low density polyethylene to a thermoplastic polyurethane sheet calendered on a polyester fabric has been studied.

In the first part, the study has shown that the addition of LDPE in the TPU coating has no direct impact on the peel strength while the die temperature has a strong influence. Because of the short sheet extrusion range of TPU, the LDPE is essential to extrude the coating at temperature above 175°C. However it is important to note that the best peel strength is obtained for an extrusion temperature of 190°C which is not the highest temperature. This must be due to a degradation of the film at higher temperature as shown by analyzing the tensile strength. The increase of the peel strength can be attributed to several phenomena among which the penetration of the coating in the fabric which creates a mechanical interlocking, and the extrusion temperature which create a different surface energy of the coating resulting in a better affinity with the fabric. In fact, it has been proved in previous research that for liquids (19) or for polymer films (20) the temperature of the material has a high impact on the surface energy. This theory will have to be proved for our study in a future work by using a pendant drop experiment as previously realized by Kwok et al (20).

In a second part, the influence of maleic anhydride as a compatibiliser between TPU and LDPE has been studied with the addition of LLDPE-g-Ma. It has been shown that the substitution of LDPE by LLDPE-g-Ma has a negative impact on the peel strength but hugely increases the tensile strength. However the

substitution of only 3%wt of LDPE (among 29%wt) by LLDPE-g-Ma has no impact on the peel strength but still increases the tensile strength.

In future work, this experimental investigation will be continued with a study of the surface energy of the extruded coating sheet depending on the die temperature. The effect of the temperature on the surface energy will be helpful to confirm the theory proposed in our conclusion to explain the better adhesion.

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