

News



The Society of Plastics Engineers

ENGINEERING PROPERTIES
& STRUCTURE DIVISION

August 2008

Past and Present EPSDIV Chairs



The present and past chairs of the Engineering Properties & Structure Division were part of the very successful ANTEC 2008, held in May in Cincinnati. Seen left to right are: xxxxxxxxxxxxx, Brian Grady, Don Witenhafer, and Jeffrey Gillmor, the chair person for 2008-2009

PLAN TO ATTEND...

EPSDIV's TOPCON

New Approaches in
Polymer Characterization
Nanocomposites,
Block Copolymers, and
other Structured Materials

October 13-14, 2008
Wilmington Delaware

*See page 5
for more information.*

September 1st deadline for ANTEC 2009 submissions

— Submitted by TCP Chairs
Shing-Chung Josh Wong and Kevin Kit

The TPCs for EPSDIV would like to report that at the time of this writing, keynote speakers for ANTEC 2009 have been confirmed. EPSDIV would like to solicit active participation and call for papers in the following areas:

- New Materials
- Recent developments in Polyolefins
- Structure & Property Relationships in Engineering


- Resins and Blends
- Polymer Nanocomposites
- Films and Packaging
- Fracture Mechanics of Polymers
- Tissue Engineering Polymers
- Materials for Biological Applications
- Polymers Useful in Alternate Energy Sources
- Smart Materials
- Nanotechnology in Electronic and Biological Devices
- Renewable Resources in Engineering Polymers

The abstract submission deadline is September 1, 2008. Yes, it is September 1!!!

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Awards for EPSDIV at ANTEC and TOPCON Plans

In-coming chair,  Jeffery Gillmor, seen at left, presents a plaque to Brian Grady, Chair for 2007-2008.



In this month's chair report, I want to report on some of the awards that were presented at the past ANTEC in Milwaukee and some information on our upcoming TOPCON in October.

ANTEC 2008

EPSDIV had another great technical program at ANTEC this year, which included fifty eight papers and three posters in eleven sessions. I want to thank our program chairs Pierre Moulinie and Murali Rajagoplan for running a great program this past year.

We have again won the silver level of the Pinnacle award again this past

year, which was presented, to Brian Grady our past chair during the awards luncheon. We had two society awards for: Fellow of the Society — Sadhan Jana and Honored Service Member — Don Witenhafer. Brian Grady our past chair has now joined the Executive Committee for SPE.

TOPCON

The preparations for our TOPCON entitled *New Approaches in Polymer Characterization: Nanocomposites, Block Copolymers, and other Structured Materials* to be held October 13-14, 2008 at Wilmington Delaware is well under way.

We have six invited speakers that are experts in this field of study and many contributed talks, which should provide any attendee with a great depth of technical knowledge. We are also looking for sponsors/exhibitors, which would be a great opportunity for companies providing products and services related to this field. Please check the SPE website for further information.

— Jeffery Gillmor

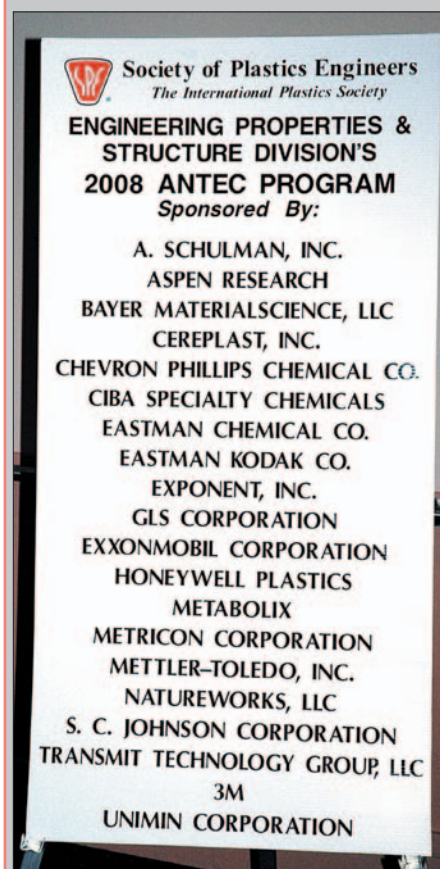


Sadhan Jana was awarded Fellow of the Society at ANTEC 2008.

Best Paper, ANTEC 2008 is
reprinted in this issue.

See page 6

THANK YOU



Our ANTEC sponsors, listed above, contributed greatly to EPSDIV's success at ANTEC 2008. We thank them and appreciated their support.

EPSDIV was recognized with a Silver Pinnacle Award at ANTEC 2008



Update on changes at SPE



Don Witenhafer

There have been some changes to the top leadership of SPE. First, I would like to recognize **Brian Grady** our Division Chairman of last year for his appointment as a Vice President and member of the Executive Committee of SPE. Brian will no doubt add valuable leadership to the society.

Barbara Arnold-Feret and **James Griffing** have been elected Vice President-Treasurer and Vice President-Secretary respectively. I know them both well and have no doubt they will be excellent officers in these positions. Congratulations.

SPE has just completed an update of the website and if you have not visited it recently you should do so.


One new member benefit of the new website is that back issues of SPE journals are available online

to all members. They are key word searchable. Also SPE has completed a publishing agreement with Wiley Publishing for *Plastics Engineering*. It is anticipated that this will substantially reduce the cost of publishing this magazine. *Plastics Engineering* has had many supporters and detractors over the years. Hopefully this change will be able to please both sides of this issue.

One benefit from this deal I understand is the SPE members will have limited access to all Wiley journals online. You may download a limited number of articles each year.

Don't forget that the due date for ANTEC submissions has been moved forward this year. Don't miss out because of not knowing the deadline. I assume that this is because of the added complication of meeting with NPE.

— Don Witenhafer, Division Councilor



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ANNUAL FINANCIAL REPORT: JULY 1, 2007 TO JUNE 30 2008



*Emmett Crawford is the EPSDIV
Treasurer for 2008/2009*

STARTING BALANCE as of July 1, 2007

XXXXXXX

INCOME	Actual	Budget	Variance
Interest & Dividends	1310.827	500.00	810.82
Newsletter Ads/Sponsorships	750.00	4500.00	(3750.00)
Scholarship Contributions	1000.00	1000.00	—
SPE Rebate	4970.99	4200.00	770.99
ANTEC Sponsorships	4000.00	6500.00	(2500.00)
Refund from Meeting (Hyatt)	44.42	0.00	44.42

Total Income **4668.19**

EXPENSES

General Office Expenses	0.00	150.00	(150.00)
Teleconferences	1106.19	1000.00	106.19
Board Meetings	1508.27	1000.00	508.27
TOPCON	566.87	1000.00	(433.13)
Newsletter Printing/Mailing	1070.00	1000.00	70.00
Awards	2301.00	2000.00	301.00
TPC	3198.26	4000.00	(801.74)
Councilor Travel	1872.97	1500.00	372.97
BOD Travel	603.28	2000.00	(1396.72)
Student Travel Fund	-	500.00	(500.00)
Membership	0.00	300.00	(300.00)
ANTEC	485.00	1000.00	(515.00)

Total Expense **(2438.16)**

Allocation of Funds

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TOPICON 2008

New Approaches in Polymer Characterization: Nanocomposites, Block Copolymers and Other Structured Materials

October 13-14, 2008
Wilmington Delaware USA

Invited Keynote Speakers

Professor Wesley Burghardt, Northwestern University
Flow-induced Alignment in Polymer Nanocomposites

Professor Ben Hsiao, SUNY Stony Brook
Control of Nanostructures

Professor Richard Register, Princeton University
Crystallizable Block Copolymers

Dr. Richard Vaia, Air Force Research Laboratory
Responsivity in Polymer Nanomaterials: Structure-Property Relationships

Professor Karen Winey, University of Pennsylvania
Combining STEM and SAXS to Probe the Morphologies in Ion-Containing Nanocomposites

Peter Lillehei, NASA Langley Research Center
New Methods for Imaging Nanocomposites

Additional contributed-paper topics include:

- ♦ Nanoscale Imaging
- ♦ Block Copolymers
- ♦ Electrochromism
- ♦ Polymer Nanocomposites
- ♦ Electron Tomography

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Society of Plastics Engineers

and the SPE Engineering Properties & Structure Division and SPE Philadelphia Section

ADHESION OF ELASTOMERIC COPOLYMERS TO POLYOLEFINS

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Abstract

The effectiveness of elastomeric copolymers as adhesives for polypropylene and polyethylene was studied using coextruded microlayered tapes. The olefinic and styrenic block copolymers were the most effective adhesives of all the elastomeric copolymers used in this study, and they delaminated adhesively from the polypropylene-tie-layer interface during T-peel experiments. The effect of tie-layer thickness, peel temperature, and peel rate on the adhesive properties were probed. Relationships between tie-layer deformation, damage zone structure, interfacial morphology, and delamination toughness were established, and a structural mechanism is provided.

Introduction

Polypropylene (PP) and high-density polyethylene (HDPE) are amongst the most abundant polymers in use world-wide due to their low cost, mechanical robustness, and melt processability. Blending the two materials might produce synergistic properties, and would definitely ameliorate the challenge of materials sorting during plastics waste recycling. Unfortunately, these polymers are incompatible, and their blends show extremely poor toughness and tensile elongation properties.

Incompatibility has been attributed to poor interfacial adhesion between the PP and HDPE phases resulting in fracture during elongation or impact. A well-known method for improving interfacial interactions in polymer blends is the addition of a suitable compatibilizer. The compatibilizer lowers the interfacial tension and promotes adhesion between the two phases.

In a recent study, it has been shown that the addition of a linear olefinic multi-block copolymer (OBC) synthesized using a novel chain shuttling technology [1,2] can effectively compatibilize a PP/HDPE blend [3]. The OBC significantly reduced the domain size by concentrating at the interface between PP and HDPE and

resulted in improved interfacial adhesion and mechanical properties.

In the present study, we test the adhesion of five different compatibilizers to polypropylene and polyethylene using coextruded microlayered tapes. Microlayered systems with many alternating layers of two or more polymers can be thought of as one-dimensional blends where the layers can be readily controlled to thicknesses on similar size scales as domains in commercial blends. Adhesion is measured as delamination toughness and compared for the different compatibilizers. The effects of tie-layer thickness and peel rate on the adhesive properties of the different compatibilizers were examined.

Experimental

Microlayer tapes were coextruded using the three-component layer multiplying process described previously [4,5]. The tapes were about 2 mm thick and 12 mm wide, and consisted of alternating layers of PP and a Ziegler-Natta catalyzed HDPE separated by a tie layer. The adherents and tie-layers used in this study are tabulated in **Table 1**. The tie-layers include: (1) a styrene-ethylene-butadiene-styrene (SEBS) triblock copolymer, KRATON[®] G1652M (Kraton Polymers, TX) having a 30/70 styrene/rubber ratio and is referred to as a styrenic block copolymer (SBC) in this paper; (2) an ethylene-octene linear multi-block copolymer, OBC (The Dow Chemical Company), and is referred to as an olefinic block copolymer (OBC) in this paper; (3) an ethylene-octene random copolymer, ENGAGE[™] 8842 (The Dow Chemical Company) and; (4) two propylene-ethylene copolymers, VERSIFY[™] Plastomer 2200 and VERSIFY[™] Elastomer 2400 (The Dow Chemical Company) having different comonomer contents. Microlayer tapes with 65 layers (16 PP layers and 17 HDPE layers separated by 32 tie layers) were produced. Different tie-layer thicknesses were produced by varying the extruder feed ratios. The adherent PP and HDPE layers had thicknesses of 50 μm each, and the tie-layer

thicknesses ranged from 2, 4, 10 and 14 μm . A microlayered tape of PP and HDPE with no tie-layer was also produced as a control. The tapes were collected on a conveyor-belt take off unit and quenched in cold water.

The microlayered tapes were microtomed through the thickness direction at -70°C and the cross-section surface was observed under an Olympus BH-2 optical microscope. The layer uniformity and tie-layer thicknesses were measured.

Delamination was carried out with a modified T-peel test (ASTM D1876). Strips 6.4 mm wide and 20 cm long were cut from the center of the tape and notched by pushing a fresh razor blade into the midplane of the tape. The notch was examined with an optical microscope to ensure that the crack started along a single interface [6]. Specimens were peeled at ambient temperature at 10 mm min^{-1} in an MTS Alliance RT/30 electromechanical testing frame. Three specimens of each composition were tested.

Composition of the peeled surfaces was determined by a Nicolet 800 FTIR spectrometer in the ATR mode. Five different areas for each surface were tested.

Results and Discussion

Optical micrographs of microlayered tapes

The cross-section of the microtomed tapes was observed under the optical microscope (OM). The alternating and uniform layered structures of PP and HDPE separated by a tie-layer were observed, **Figure 1**. Two tie-layer thicknesses, a 4 μm and a 10 μm thick tie-layer are shown as examples.

Comparison of peel data for various adhesives

Figure 2 shows typical peel force vs. deflection curves of the three different microlayered tapes having a tie-layer thickness of 2 μm . For all tapes, the arms bent into the T-peel configuration as the load was applied, and the crack propagated at a relatively constant load, P_{cr} , from which the peel force, $F = P_{\text{cr}}/W$, was obtained for a specimen[s] of width W [7]. The peel force of the PP/HDPE control with no tie layer was $45 \pm 10\text{ N/m}$. The incorporation of a relatively thin 2 μm adhesive tie-layer vastly improved the adhesion of PP to HDPE. At this thickness, OBC and SBC were the most effective adhesives with peel forces about an order of magnitude more than the control at $500 \pm 30\text{ N/m}$ and $460 \pm 30\text{ N/m}$ respectively. The EO, P/E 876 and P/E 859 also improved the adhesion between PP and HDPE and had peel forces of $320 \pm 20\text{ N/m}$, $360 \pm 30\text{ N/m}$ and $350 \pm 20\text{ N/m}$ respectively.

The effect of layer thickness on peel force is shown in **Figure 3**. Almost no dependence on layer thickness was observed in the system with P/E 876 tie-layers, suggesting that adhesive failure occurred with little deformation of the adhesive layer. In contrast, the other four tie-layers had significant, linear dependencies on layer thickness. The dependence was strongest in the SBC,

OBC, and P/E 859 systems, suggesting that deformation occurred through the thickness of the tie-layer. With thick 14 μm tie-layers, these three adhesives had similar performance with peel forces of 1000 and 1200 N/m. The peel forces of EO 855 and P/E 876 were approximately 50 and 25% of these values, respectively. Interestingly, the regression lines extrapolate to similar values, suggesting that the purely adhesive components of failure are similar amongst these tie-layers.

The viscoelastic contribution to the peel force was probed with ambient temperature peel rate experiments, **Figure 4**. Very little dependence on peel rate was observed in all the tie-layers except for SBC, which had pronounced time dependence.

Surface composition of peeled surfaces

The composition of the delaminated surfaces was determined by ATR-FTIR. The adherent and adhesive tie-layers have unique FTIR peaks that allow for their differentiation. For the HDPE/SBC/PP tapes, one surface exhibited strong peaks at 698, 1453, 1463 and 1494 cm^{-1} , characteristic of the SBC and very weak intensity HDPE peaks at 719 and 1473 cm^{-1} , **Figure 5(a)**. This surface was characterized as the SBC surface. It was speculated and later confirmed using atomic force microscopy (AFM) that the weak HDPE peaks were due to the migration of low molecular weight fractions of the HDPE into the tie-layer. The matching surface showed strong PP peaks at 1376, 1456, 2839, 2866, 2918 and 2950 cm^{-1} , and the surface was characterized as being the PP surface. This identification confirmed that the delamination occurred adhesively at the PP/SBC interface, and that failure occurred adhesively during the peel test.

For the HDPE/OBC/PP tapes, one of the fracture surfaces showed strong OBC peaks at 720, 731, 1462, 1473, 2849, 2917 and 2956 cm^{-1} and the matching surface showed PP peaks at peak positions described previously, **Figure 5(b)**. The presence of OBC peaks on one surface and PP peaks on the matching surface confirmed that the delamination occurred at the PP/OBC interface. For the HDPE/EO/PP tapes, strong EO peaks at 720, 1457 and 1466 cm^{-1} and weak HDPE peaks at 730 and 1473 cm^{-1} peaks were identified on one surface, while the other surface showed characteristic PP peaks, **Figure 5(c)**. The HDPE peaks were postulated to be the result of the diffusion of low molecular weight HDPE fractions into the EO tie-layer.

For the HDPE/(P/E)/PP tapes, one surface exhibited strong P/E peaks at 1377, 1461, 2842, 2869, 2920 and 2952 cm^{-1} and weak HDPE peaks at 2849 and 2917 cm^{-1} , **Figure 5(d)**. The matching surface showed strong HDPE peaks and a very weak P/E shoulder at 2951 cm^{-1} . The presence of primarily HDPE on one fracture surface and primarily P/E on the other fracture surface confirmed that the delamination occurred at the HDPE/(P/E) interface.

Conclusions

The effectiveness of different adhesives on polypropylene and high density polyethylene was successfully tested using coextruded microlayer tapes. It was found that the SBC and OBC were more effective in improving the adhesion of HDPE to PP. The delamination for the SBC, OBC and EO adhesives occurred at the PP/tie-layer interface, while the delamination for the P/E adhesives occurred at the HDPE/tie-layer interface during the T-peel test. This was also confirmed by measuring the surface composition of the peeled surfaces. SBC exhibited higher strain rate dependence as compared to the other adhesives.

Acknowledgments

The project was made possible through the generous financial and technical support of The Dow Chemical Company.

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Best Paper ANTEC 2008 continued on page 10

REMEMBER:
September 1st deadline
ANTEC 2009 Submissions

Upcoming Events

2008 Thermoforming Conference:

September 20-23, Minneapolis Convention Center
Minneapolis, Minnesota USA

EUROTECTM 2009 Call for Papers

Submit your abstract by October 15th

SPE Automotive Composites Conference (ACCE):

September 16-18, 2008, Management Education Center, Michigan State University, Troy, Michigan

New Approaches in Polymer Characterization:

October 13-14, 2008, Holiday Inn Select, Wilmington, Delaware USA

SPE 2008 New Technology Symposium:

November 11-12, 2008, Renaissance Philadelphia Hotel Airport, Philadelphia, Pennsylvania USA

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Table 1. Characteristics of adherent and adhesive tie-layers

Adherent Layers			Adhesive Tie Layers		
Material	Grade	Density (g/cc)	Material	Grade	Density (g/cc)
PP	H105-03	0.900	SBC	KRATON [®] G1652 Styrene/Rubber Ratio: 70/30	0.910
ZN-HDPE	DMDH 6400	0.961	EO855	ENGAGE [™] 8842 Octene content = 17 mol%	0.855
			OBC	M.I. = 1.2 g/10 min Hard Segment = 24 wt%; Octene content in Hard Segment = 0.4 mol%; Octene content in Soft Segment = 16 mol%	0.880
			P/E 876	VERSIFY [™] 2200 Ethylene content = 13 mol%	0.876
			P/E 859	VERSIFY [™] 2400 Ethylene content = 20 mol%	0.859

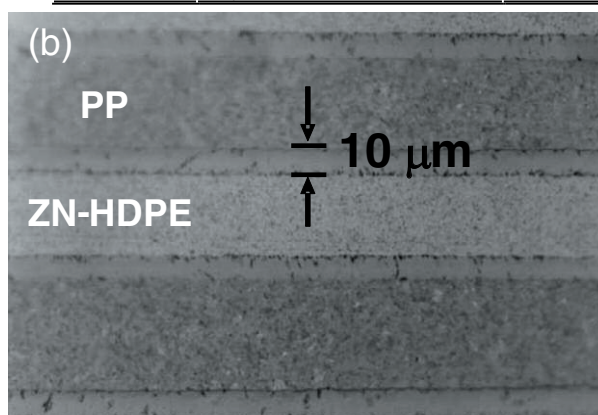
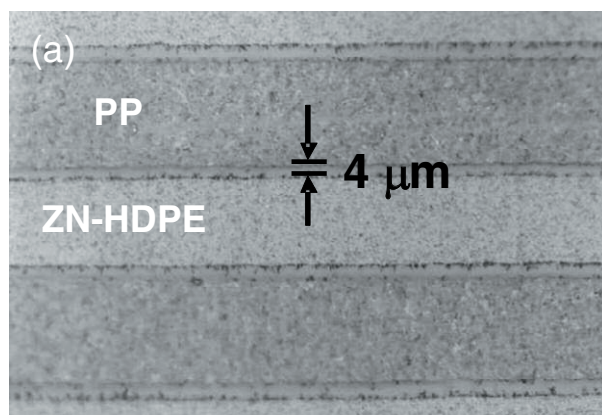


Figure 1. Optical micrographs of thin and thick tie layers

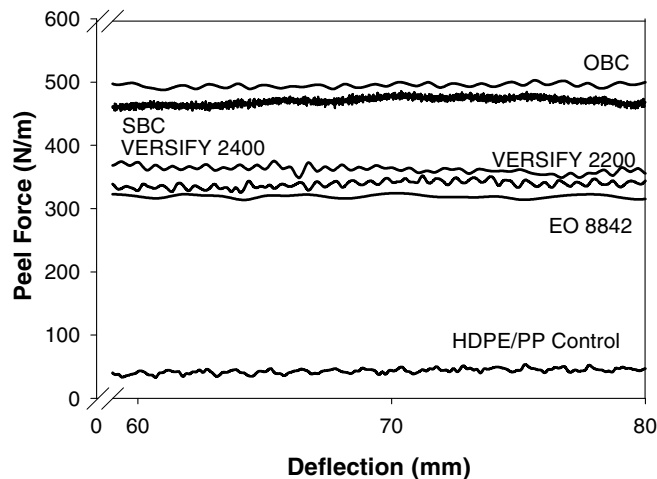


Figure 2. Comparison of peel forces for different tie-layers (Tie-layer thickness = 2.2 μm)

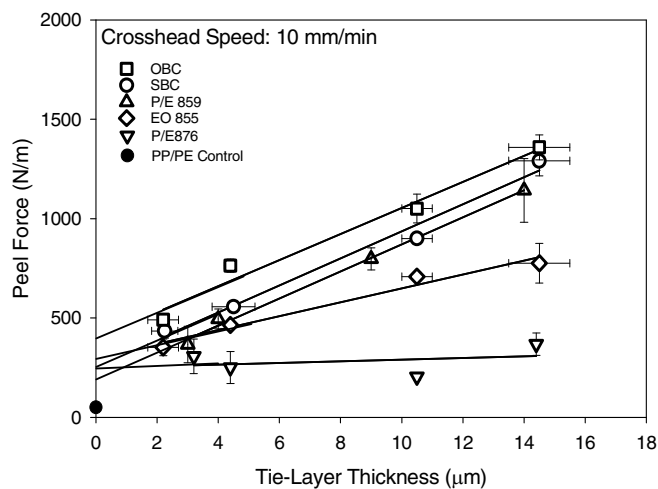


Figure 3. Effect of tie-layer thickness on the delamination toughness of different tie-layers

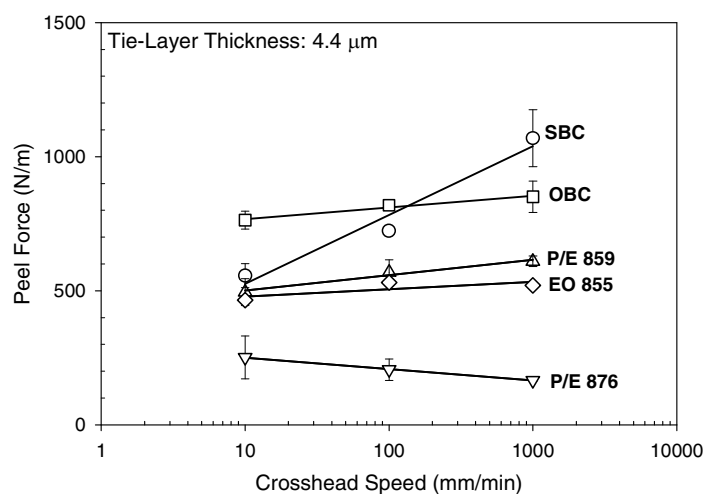


Figure 4. Effect of crosshead speed on the delamination toughness of different tie-layers

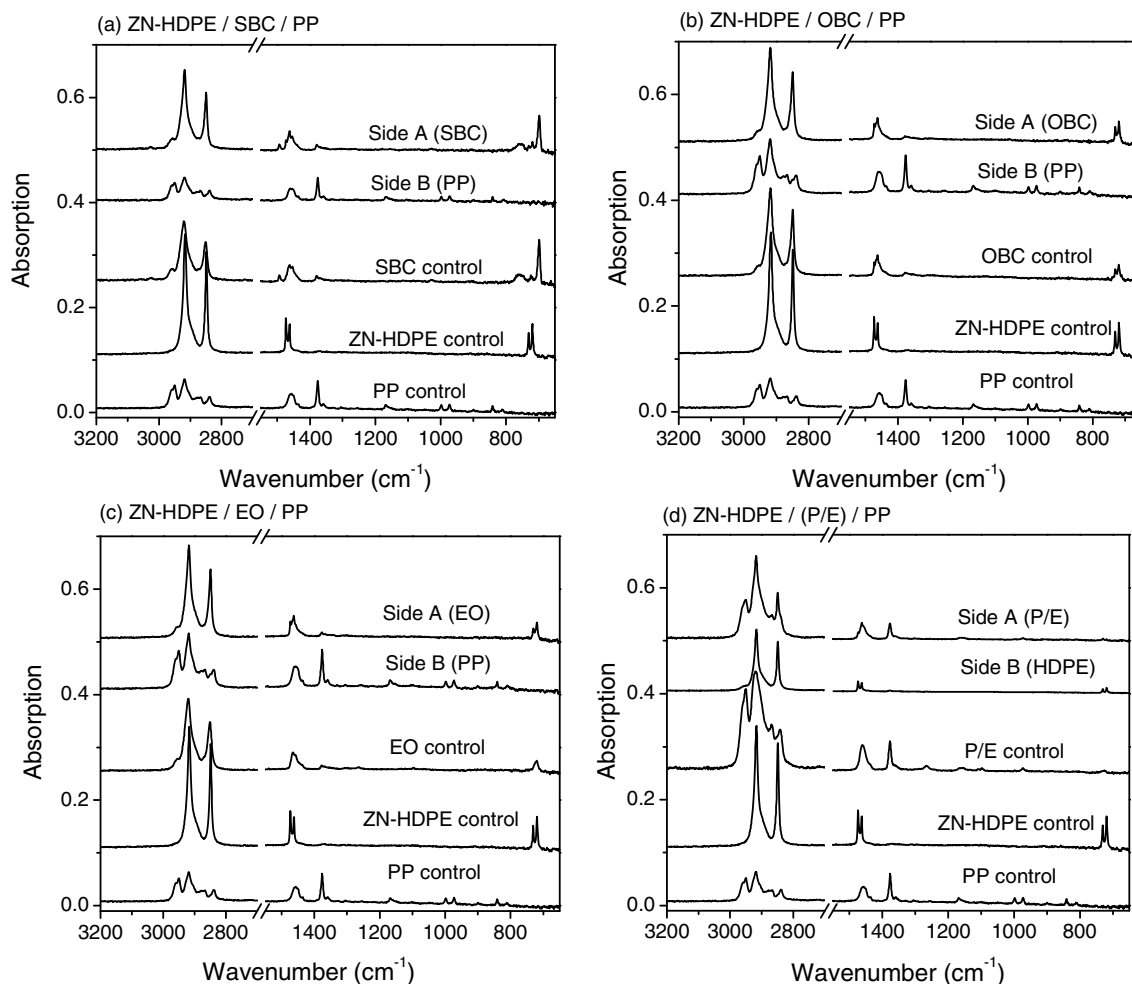


Figure 5. Surface composition of the peeled microlayered tapes using ATR-FTIR

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