International Journal of Advanced Technology in Engineering and Science
Vol. No.4, Issue No. 12, December 2016
www.ijates.com
ijates
ISSN 2348 - 7550

STUDY OF POLYETHERIMIDE (PEI)-POLYETHYLENE TEREPHTHLATE BLENDS (PET)

Qayssar M. Alobaidi

Department of Polymer Engineering, Maharashtra Institute of Technology (MIT), (India)

ABSTRACT

The objective of this work it is to develop the blends based on polyetherimide(PEI) with thermoplastic polyester polyethylene terephthlate (PET) blends suitable for various automotive application such as under hood components, lighting etc. Optimization of the blend composition will be made for improved processibilty, thermal properties and better machining characteristics. The optimized blend composition will have acceptable heat deflection temperature for under the hood application and better processibility (improved processing window higher flow ration for injection moulding, lower escrow torque during plasticizing).The optimized blend composition will have better machining characteristics (machinability index) and better metalizing properties. Polymer blends is one of the significantly important option to develop various polymeric materials required for wide range applications. Especially studies on the blends of engineering thermoplastics such as polyimide and its derivatives have been gradually increased in recent years. The blends find promising, application in automotive sector which use engineering thermoplastics to a large content .This study will be beneficial for this sector, considering the cost effectiveness of proposed blend along with promising desired properties.

Keywords: Polyester, Polyimide, Polymer Blends, Processibility, Thermoplastic

I INTRODUCTION

Polymers have been combined with other plastics to form blends, mixed with various fillers to give filled systems, and extruded and molded with fibers and other anisotropic reinforcements to give composite and hybrid materials. This “mix and match” approach has allowed plastics engineers to utilize a small library of polymers to produce bewildering array of useful products capable of possessing extremes of property values. Polymer blends is an excellent way of developing new materials with desired properties for various applications. The studies on the blends containing super engineering plastics such as polyimide and its derivatives have been gradually increased in last few decades.[1]

Polyetherimide (PEI) is an amorphous high performance polymer with a high Tg of 215°C and has excellent physical properties such as tensile, flexural and compressive strength, toughness and good electrical insulation. But, due to amorphous nature it has poor chemical resistance. The key performance attribute of PEI is its ability to maintain properties at elevated temperatures up to 180°C.

However, although this polymer has been developed to improve the processibility of wholly aromatic polyimides, its processing temperature is still high, about 380°C. Polyethylene terephthalate (PET) on the contrary is semi-crystalline polymer with Tg about 78°C and melting temperature of about 255°C. It has processing temperature of about 270°C for unmodified neat PET. Hence, blending of PEI with PET will provide wider processing window which will be beneficial for processor / component manufacturer.[2]

Heat Distortion temperature (HDT) of PET is about 220°C while that of PEI is about 190°C. Therefore, it can be expected that blending of PEI with PET will improve its HDT and make it more useful for high temperature application. Better % elongational characteristics of PEI/PET blend will be helpful in automotive lighting (tail lamp) applications as these facilitates the required machining operations (drilling hole for fitting, fixing screws etc.) Also, it is not possible to carry out metalizing operation on PET while PEI can be metalized with relative ease. Hence, it can be expected that PEI/PET blend will provide a new window for metalized component applications.

The blending of PEI with PET can also reduce the cost (cost of PEI is about Rs. 1400-1500 per kg while PET is about Rs. 200-250 per kg) and thus extending its application window for high thermal and chemical. PEI/PET blends may offer an interesting route to combine the complementary properties of both the polymers. [2]

II POLYTHERIMIDE THERMOPLASTIC

2.1 General Description

Polyetherimide (PEI) is an amorphous thermoplastic (5), which is based on repeating aromatic imides and ether units, PEI is known for its high strength and rigidity, especially under long-term heat exposure. The rigid aromatic imides units provide PEI with its high performance properties at elevated temperatures, while the ether linkages provide it with the chain flexibility necessary to have good melt flow and processability. Currently, the largest area of growth of PEI is in metal replacement applications, mostly involving the replacement of aluminium and brass automotive parts. Specially grades available include glass, mineral, and carbons reinforced, low wear and low friction, improved chemical resistance and PC/PEI blends, as well as commercial aircraft interior and medically rated grades. **[3]**

2.2 General Properties

Polytherimide is a high performance plastic, (6) which is well suited for extreme service environments. At room temperature, its mechanical properties exceed those of most thermoplastics, and it displays an impressive retention of these properties at temperatures as high as 375^0F (191^0C). PEI also performs extremely well at elevated temperatures. For example, Ultem 1000 has a glass transition temperature of 419^0F (215^0C), heat deflection temperature at 264 psi (1.82MPa) of 392^0F (200^0C), and continuous service temperature of 338^0F (170^0C). PEI exhibits excellent impact strength and ductility, but does display notch sensitivity when subjected to high stress rates. PEI has an exceptionally high flame resistance, and when it does burn, it generates very low levels of smoke. It is an excellent electrical insulator, has a low dissipation factor, a high volume resistivity, a high arc resistance, and is extraordinarily free of ionic contaminations. Not only does PEI have excellent hydrolytic stability, UV stability, and radiation resistance, but also it is also extremely well suited for repeated stream, hot air, ethylene oxide gas and cold chemical sterilizations .[3]

III POLYESTER BLENDS WITH AMORPHOUS ENGINEERING THERMOPLASTIC

The majority of engineering polymer blends used in automotive applications. The most common reason for choosing an amorphous engineering polymer in polyester blends is to reduce mold shrinkage and to increase dimensional stability.(6) In the case of glass fiber-reinforced grades, the blending approach helps to reduce war page. The amorphous engineering polymers, such as PEI, PC, Par and ABS, provide excellent impact strength in addition to the dimensional stability and freedom from shrinkage or war page, whereas the thermoplastic polyesters, being crystalline, offer excellent chemical resistance, good mechanical properties, and ease of melt processing due to low viscosity above their melting points. Therefore, blending of crystalline thermoplastic polyester with an amorphous engineering polymer offers good dimensional stability, ease of processing, and chemical resistance, as well as mechanical properties that can be tailored to suit particular application needs.[4]

IV POLYETHERIMIDE BLENDS

4.1 Literature Review

4.1.1 -Jonghan Choi and Miko Cakmak (2016) have studied the effect of blend composition and deformation history on the development of structural hierarchy in crystallizable PET rich blends of PET/PEI from amorphous PET/PEI films are melt miscible consequently the addition of PEI to PET increases the glass transition temperature while slightly broadening it as a result of partial micro phase separation. The crystallization in these films was found to be affected by two main factors: i) blend composition ii) the extent of deformation imposed on the films leading to preferential orientation. In the absence of orientation, the increase of PEI fraction leads to decrease of crystallizability as expected from the dilution effect that spatially prevents the crystallizable polymer chains from coming together. Stretching process and resulting stress levels and crystallinity depends on the blend concentration. The increase of PEI at a given temperature increases the stress levels during stretching due to both the increase of Tg and its breadth. The increase PEI concentration leads to increase in highly defective crystallites due to intrusion of bulky PEI chains into the PET crystalline regions. Above 30% PEI, the blend crystallizatibility disappears.[5]

4.1.2-Robert R. Gallucci and Mark A. Sanner (2011) have studied the Blends of polyetherimide (PEI) with polyethylene terphthalate (PET) were re-examined. Previous blends with high (>25%) PEI content and those with very low (<10%) PEI shows a single Tg giving clear films indicative of a miscible blend. The samples stayed clear on heating and did not crystallize or phase separate. Use of lower Mw PEI:PET resin opened the window of miscibility allowing clear blends to be formed with as much as 50% PET. These clear yellow resins have high strength, 145-160^{0}C Tg, good practical toughness and high flow. PEI performance. Use of a low Mw

PET:PEI pairs, with 40-50% PET, allows formation of clear, miscible blends with low haze and a 145 to 160^{0}C Tg. The blends have high strength and stiffness, as well as good practical toughness.[6]

4.1.3-J. Ramiro, J.L. Equiazabal, and J. Nazabal (2006) have studied the compatibilized poly (ether imide) / amorphous polyamide (PEI/a-PA) blends in the melt state by substitution of 20% PEI by PET. The two amorphous phased of the blends comprised both a miscibilized 80/20 PEI/PET blend. The presence of PET in the two phases of most of the blends was the main reason for the clear decrease in the particle size that indicated compatibilization .(24) The clear increases in ductility and impact strength after PET addition further demonstrated the compatibilization effect of PET. The addition of PET led to a clear increase in the processability of the blends and to decrease in the dispersed phase size throughout the composition range.[7]

4.1.4-Lee JK, Choi WS, Kwon YK, Lee KH (2002) have carried out the work on the liquid-liquid (L-L) phase separation an crystallization behaviour of poly (ethylene terephthalate) (PET) / Poly (either imide) (PEI) blend were investigated with optical microscopy, light scattering, and small angle X-ray scattering (SAXS). The thermal analysis showed that the concentration fluctuation between separated phases was controllable by changing the time spent for remixing before crystallization. The L-L phase-separated time (ts). The slow crystallization for a long t_s implied that the growth path of crystals was highly distorted by the rearrangement of the spinodal domains associated with coarsening. The characteristic morphological parameters at the lamellar level were determined by the correlation function analysis on SAXS data. The blend had a larger amorphous layer thickness than the pure PET, indicating that PEI molecules in the PET-rich phase were incorporated into the interlamellar regions during crystallization.[8] .

4.1.5-Adhemar Ruvolo and Adriana de Fatima Barros (2001) have studied the miscibility, crytstallization and melting behavior in blends of virgin and recycled poly (ethylene erephthalate) (PET) with poly (ether imide) (PEI) have been examined using DSC. The blends exhibit single, composition dependent T_g's over the entire composition range and a negative value of the Flory interaction parameter, indicating miscibility of the amorphous polymers. The crystallization of the PET/PEI blends was dramatically slowed by PEI contents higher than 20%. A linear correlation between a structure –dependent parameter R_T (from FTIR measurements) and the enthalpy of fusion (from DSC measurements) for both types of PET samples used in the blends, make the determination of the degree of crystallinity of the PET in any PET/PEI blend possible. In agreement with the melting behaviour of the PET/PEI blends.[9]

V PEI-PET BLEND

Polytherimide is an amorphous, high performance polymer with a Tg of 215^{0}C. The key performance attribute of PEI is its ability to maintain properties at elevated temperatures up to 180^{0}C. The blending of PEI with PET is aimed at lowering the cost of using PEI and thus extending its applications window in end users requiring high thermal and chemical resistances. Typical applications of PET/PEI blends are those requiring higher thermal stability and durability. Since the products from PET/PEI blends are transparent and can withstand temperatures as high as 160^{0}C, cookware and food bowls, which move from the refrigerator or the freezer to the microwave and to the dishwasher, constitute a potential application area. A commercial example of PET/PEI blends is Ultem 1285 from General Electric Co. It has the US Food and Drug Administration approval and is being used by Tupperware, a well-known food service manufacturer.[4]

PEI is known to form a miscible blend with PET in the melt. However, when PET/PEI blends are cooled to temperatures below the melting point, a liquid-liquid phase separation occurs via the spinodal decomposition mechanism, and the resultant modulated (interconnected) structure is preserved owing to the rapid crystallization of PET. In a recent paper, the phase diagram of a PET/PEI blend has been reported. An upper critical solution temperature (UCST) phase diagram below 240^0C is identified for the system. The range of temperature and composition over which liquid-liquid phase separation and crystallization occur simultaneously is rather wide. The effect of spinodal decomposition on the growth of PET spherulites was investigated by small-angle light scattering (SALS). Non-linear growths were observed at higher crystallization temperatures of 210 and 220^0C, whereas the growths were linear below 210^0C. At a crystallization temperature below 210^0C, the growth rate locked in without a chance to rearrange. Thus, the growth rate remained nearly constant. When the crystallization temperature was increased, the growth rate was depressed much more than the spinodal decomposition rate. As a result, the spinodal domains could undergo some rearrangement before being locked in by crystallization, and the radial growth rate was altered. The final morphological formation in the blend will thus be influenced by the crystallization temperature. The increase in PEI content decreases the bulk crystallization rate and the degree of crystallinity. These studies indicate the criticality of controlling the injection molding conditions such as melt and mold temperature, which influence the quench rate achieved during the manufacture of products from PET/PEI blends.[4]

In the recent years, the study of polyester blends with other polymers has been directed towards a deeper insight in the miscibility and compatibility of such systems. If the polymer pair is miscible, the blend between an amorphous and a semi crystalline polymer is more interesting. It is the case of poly(ethyleneterephthalate)-poly(ether imide) (PET/PEI) blend. As being an amorphous polymer, PEI presents low resistance to solvents and because of its high glass temperature, PEI has a high resistance to solvents, but its mechanical properties are not so good. The effects of blending on glass transition behaviour and on the melting temperature were analyzed and reported in this paper. This analysis is also interesting as long as the melt behaviour of PET is not yet well understood. One of the possible partners for blending with PEI is poly(ethy1ene terephthalate) (PET). In the case of the PEI/PET blends and with respect to PET-rich blends, for instance, the presence of PEI should both decrease the crystallization ability of PET and also increase its glass transition temperature if the polymers are miscible. Both of these effects are positive in some of the applications of PET, such as bottle production, because they give rise to increased melt strength and also reduce crystallization tendency, the single Tg values increase monotonically as the PEI content in the blends increases. These results indicate the miscibility of the PEI /PET blends.[4]

Gallucci and Sanner in 2011 conducted some tests on PET-PEI blends to observe the effect of MW on the miscibility of the two polymers. The PEI used was Ultem 1010 and both high and low Mw grades were used.

The Mw of low MW PEI was 32000. PET used were high IV=0.83(Mw=92400) and Low IV=0.56(Mw=56200). The Polymers were melt extruded at 290 C and then dried at about 125oC. Then they were moulded into specimens at 300C-340C.[6]

DSC scans showed that the for 50/50 composition of High Mw PEI with High Mw PET, hazy films were obtained for high IV PET but gave lower Tg. On the other hand clear films were obtained for low IV PET. proportion of Low Mw PEI and low Mw PET gave clear films with good mechanical properties which may be due to the higher modulus of PEI.fig.1

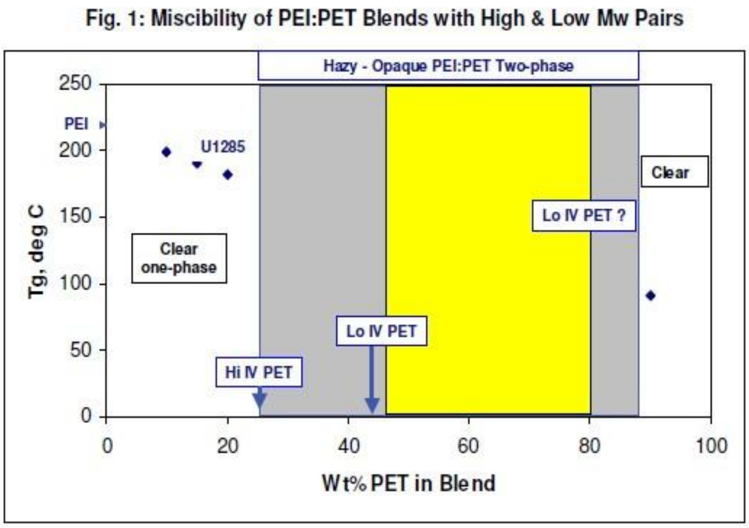

Fig. 1*: Miscibility of PEI: PET blends with high and low mw pairs*

DMA studies showed that the blend provided higher impact strength and could not be broken even after dropping from 6ft above the ground on a concrete floor. The relatively higher PET content in PEI gave it better flowability during processing. The viscosity v/s shear rate plot showed that the blend had good melt flow rate and its melt viscosity was comparable to standard polycarbonates. TGA tests showed that the blend exhibited better thermal stability than PET. The change in viscosity was less than 10% when heated at 300oC for 30 min. This shows that the blend gives a wider processing window for injection moulding purposes. [6]

Miscibility, crystallization and melting behavior in blends of PEI and PET have been examined using DSC by [8]. It was found that the blends exhibit single and composition- dependent Tg's over the entire composition range, indicating the miscibility of the PEI/PET blends in the amorphous region. Also, the temperature interval available for crystallization of PET in the blends, Tm - Tg, are decreased with the addition of PEI.fig.2

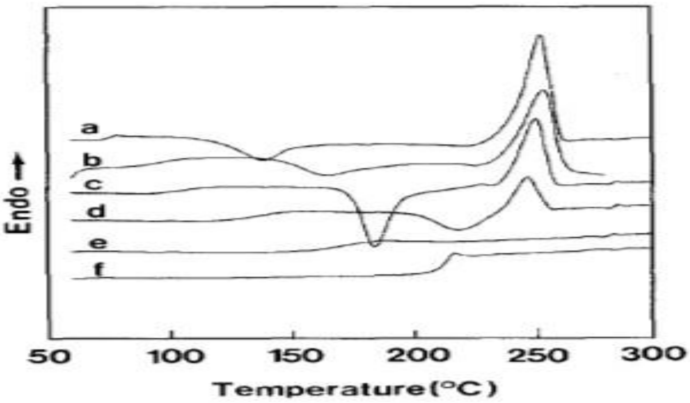

Fig. 2: DSC thermograms of PET/PEI Blends: a-100, b-80, c-60, d-40, e-20,f-0 wt% of PET

The reciprocal of the crystallization half-time corresponding to the rate of crystallization decreases with increasing the content of PEI in the blends. This results from an increase of the Tg's of the blends with the addition of PEI The equilibrium melting temperatures of PET in the blends, which are determined from the Hoffman-Weeks plot, are depressed with increasing the PEI content.

Miscibility, crystallization and melting behavior in the blends of virgin and recycled PET with PEI have been examined using DSC data in the blends exhibit single, composition- dependent Tgs over the entire composition range and a negative value of the Flory interaction parameter, indicating miscibility of the blends in the amorphous region. The crystallization of the PET/PEI blends became dramatically affected by PEI contents higher than 20%.

A linear correlation between the structure-dependent parameter RT (from FTIR measurements) and the enthalpy of fusion (from DSC measurements) for both types of PET samples used in the blends, make the determination of the degree of crystallinity of the PET in any PET/PEI blend possible. Corroborating the melting behavior of the PET/PEI blends, the average values of the activation energy for transconformer formation confirm the negative effect of the PEI content on the cold crystallization of the PET. [3]

The primary and secondary crystallization rate constants at various Tcs were obtained from the analysis for different PET/PEI blend compositions in [4]. It was found that the rates of both crystallization stages decreased with increasing PEI composition. Because the temperature at which the maximum crystallization rate was located, Tmax was approximately the same for primary and secondary crystallization at a given blend composition, this suggested that the average melt compositions from which these two crystallization stages proceeded were about the same.

This project aims at optimization of the material used in manufacturing of automotive components that are largely dominated by PEI by increasing its melt processability and give a higher processing window and higher flow rate for injection stage and reduced screw torque during plasticization stage in injection moulding. It will also increase the heat distortion temperature of PEI and give good machinability index. Also metallizability of PET is less as compared to PEI.

This project aims at increasing the ease of metalizing of PET to increase applications in metalized articles. Finally the blends will be able to reduce the cost of virgin PEI markedly.

VI EXPERIMENTAL WORK

6.1 Materials

The PEI used in this work was (SABIC Ultem 1000) , It has relative viscosity of 0.5 dL/g as measured at 0.2 % and 25C in chloroform and its melt flow index is 8.5 g/10 min, measured at 337°C and with a 6.7 kg load (ASTM D-1238). PET was Reliance 0.8 IV. Three compositions of blends has been used, 70% PET-30% PEI, 70%PEI-30%PET and 50%PET-50%PEI.

6.2 Blending Process

Blending and compounding process of PET-PEI blends prepared by twin roll mills ,at 315-320°C. the gap between the rolls was 1.5mm.and the friction ratio (the difference in speed between the back roll and the front) was around 1.25.the friction ratio allows a shearing action (friction) at the nip to diperse the ingredients and to force the compound to stay on one roll. Soften and breaking of the materials bulk produced from twin roll by scrap granulator machine to obtain compounded pellets.

6.3 Molding

The compounded pellets obtained molded in an injection molding machine to obtain Tensile (ASTM D-638) and impact (ASTM D-256) specimens. the screw of the injection molding machine had a diameter of 18mm, a L/D ratio of 17.8, a compression ratio of 4 and helix angle of 17.8*. the barrel temperature was 340°C and the mold temperature was 24°C.the injection speed 8.5 cm3/s and the injection pressure 2250 bar.

VII TESTING AND CHARACHTERIZATION

Testing of injection molded specimen is an important part of the experimental work ,since it shows compatibility, feasibility and enhancement of the blend. Mechanical and thermal properties were tested in this work.

7.1 Mechanical Properties

7.1.1 .Tensile Test: Tensile test were carried out in an Instron 4301 at 23°C according to (ASTM-D-638) and at a crosshead speed 10mm/min with 10KN weight load.

7.1.2. Izod Impact Test: Impact properties of the polymeric materials are directly related to the overall toughness of the material which is defined as the ability of the polymer to absorb applied energy without failure. Izod impact test were carried out according to (ASTM-D-256) using a Ceast 6548/000 pendulum on notched specimens (notch depth 2.54mm,notch radius 0.25mm.)

VIII THERMAL ANALYSIS

8.1 Differential scanning calorimetric (DSC)

Calorimetric analysis was performed using a Perkin-Elmer DSC-7 differential scanning calorimetry. All the samples heated under Nitrogen atmosphere. Pure PET sample heated from (30°C-300°C),Pure PEI,(30%PEI-70%PET),(50%PEI-50%PET),(70%PEI-30%PET) samples heated from (30°C-350°C).and all the samples were under heating rate 10°C/min. polymer crystallinity ,polymer crystalline temperature(Tc),the area of maximum peak for the melting of the polymer and Delta H which is the heat given off by the part of polymer sample which was already in the crystalline state before we heated the polymer above Tc. were carried out.

8.2 Thermo Gravimetric Analysis (TGA)

TGA analysis carried out using Perkin-Elmer thermo gravimetric analysis system TGA-7. .all the samples heated from (30°C-900°C) under Nitrogen atmosphere. Loss of mass and degradation point were determined.

IX RESULTS AND INTERPRETATION

9.1 Mechanical Properties

Table 1: Tensile and Impact Strengths Obtained For Varying Compositions Of PEI In PET Blends

Sr. No.	**Sample Name**	**PEI**	**PET**	**Tensile Strength (N/mm^2)**	**IZOD Impact Strength (KJ/m^2)**
1	ERM-PET-100	0	100	108.09	7.15
2	ERM-PEI-30	30	70	0.66	1.47
3	ERM-PEI-50	50	50	14.61	2.08
4	ERM-PEI-70	70	30	74	2.034

The results show that as the concentration of PEI in PET goes on increasing, the tensile strength and impact strength of the materials increasing (table.1) and to understand the reason behind this behavior we have to understand that the PEI is an amorphous thermoplastic has high Tg (217°C). amorphous polymers are stiff, brittle, tough and clear in the virgin state. The temperature and stress state have a profound effect on the molecular arrangement and hence the properties of a polymer. Under the action of sufficient stress, the polymer chains can uncoil and align over a period of time. At elevated temperatures, polymer chains have enough energy to rotate and coil up further. Below a temperature known as the glass transition temperature, Tg, the structure of amorphous polymers is termed 'glassy', with a random arrangement of the polymer chains, similar to the random molecular arrangement found in glass. As the temperature increases to Tg, the polymer chains have sufficient thermal energy to rotate, resulting in a drop in modulus. Amorphous thermoplastics are generally used at temperatures below their Tg, and requires a large amount of energy to fracture below Tg. so PEI is considered tough, finding use in applications requiring impact resistance, e.g. safety helmets and bullet proof glazing. Amorphous polymers possess a wide softening range (with no distinct melting temperature), moderate heat resistance, good impact resistance, and low shrinkage.

From other side PET is a semi-crystalline thermoplastic has law Tg (78°C). The elasticity modulus E (stiffness) of a semi-crystalline thermoplastic decreases significantly when its temperature exceeds Tg, i.e. much lower than Tm. It is particularly advantageous to reinforce a semi-crystalline thermoplastic material with an amorphous thermoplastic, thus to form a blends, as this allows a considerable increase of E above Tg, enabling therefore the material to be used under high stress (as a rigid material) at a higher temperature.

The most common reason for choosing an amorphous engineering polymer in polyester blends is to reduce mold shrinkage and to increase dimensional stability. The amorphous engineering polymers, such as PC, PAr, ABS and PEI, provide excellent impact strength in addition to the dimensional stability and freedom from shrinkage or war page.

X THERMAL ANALYSIS

10.1 Differential scanning calorimetry (DSC)

Calorimetric analysis was performed using a Perkin-Elmer DSC-7 differential scanning calorimetry. All the samples heated under Nitrogen atmosphere. Pure PET sample heated from (30°C -300°C),Pure PEI ,(30%PEI-70%PET),(50%PEI-50%PET),(70%PEI-30%PET) samples heated from (30°C -350°C) and all the samples were under heating rate was10°C /min.

Degree of crystallinity of the samples determined using the following equation:

% Crystallinity = (□ Hf /□ □ □ Hf100%) x 100%

Where □ □ □ □ Hf is the heat given off by the part of polymer sample which was already in the crystalline state before we heated the polymer above polymer crystalline temperature(Tc). □ □ □ Hf = Hm – Hc J/g Hf = Hm – Hc J/g .Where: Hm is heat given off during melting, Hc is the heat of crystallization

The values of □ □ □ □ □ Hf has been gotten from the DSC diagrams.

ΔHf 100%:is a reference value and represent the heat of melting if the polymer were 100% crystalline. This reference heat of melting has been established for commonly used polymers and for PET is 140.1 J/g.

Table 2: Degree of crystallinity of various samples of PEI-PET blends

Sr. No.	PET % by wt.	PEI %by wt.	Area Of Big Peak m.J	□ Hf J/g	Crystallinity %
1	0	100	_	_	_
2	100	0	193.517	48.2585	34
3	70	30	111.730	26.6658	19
4	50	50	47.753	13.1552	9
5	30	70	34.839	10.1277	7

Differential scanning calorimetry has been extensively used to investigate miscibility of PET/PEI blends. The results shows during the DSC scan a single glass transition. The Tg values determined at the onset of the transitions, as well as Tc values As can be seen, the single Tg and Tc values increase monotonically as the PEI content in the blends increases Table 2. These results indicate the miscibility of the PEI /PET blends. As the PEI content in the blend increases, the width of the exotherm increases, and at the same time, the Tc measured at the minimum of the exotherm, increases. This indicates that PEI hinders the crystallization of PET from the glassy state, as could be expected taking into account the miscibility of the blend figures (3, 4, 5, 6, 7)

The increase of PEI leads to decrease of crystallizability as expected from the dilution effect that spatially prevents the crystallizable polymer chains from coming together. The crystallization of the PET/PEI blends was dramatically slowed by PEI contents higher than 30%. The presence of PEI in the blend reduces the enthalpy of crystallization. It is clear that both the crystallization temperature and enthalpy of crystallization decrease with the increase in PEI content.

For a semi-crystalline/amorphous miscible blend system, the depression of the crystallization temperature is influenced by several factors. First, the reduction of crystallizable polymer in the melt results in concomitant decrease in the thermodynamic driving force favoring crystallization. Second, the PET component is diluted with PEI, the transport process of PET segments to the crystallite– melt interface becomes more protracted and results in slower crystal growth rate. Third, from the thermodynamic consideration, the chemical potential of the crystallizable polymer in a miscible amorphous-crystalline blend is reduced because of the addition of the amorphous component and leads to a decrease in the equilibrium melting temperature of the blend. Finally, the

Tg of the blend rises with the increase of the PEI content, which tends to increase melt viscosity and decreases diffusion rate of the PET segments.

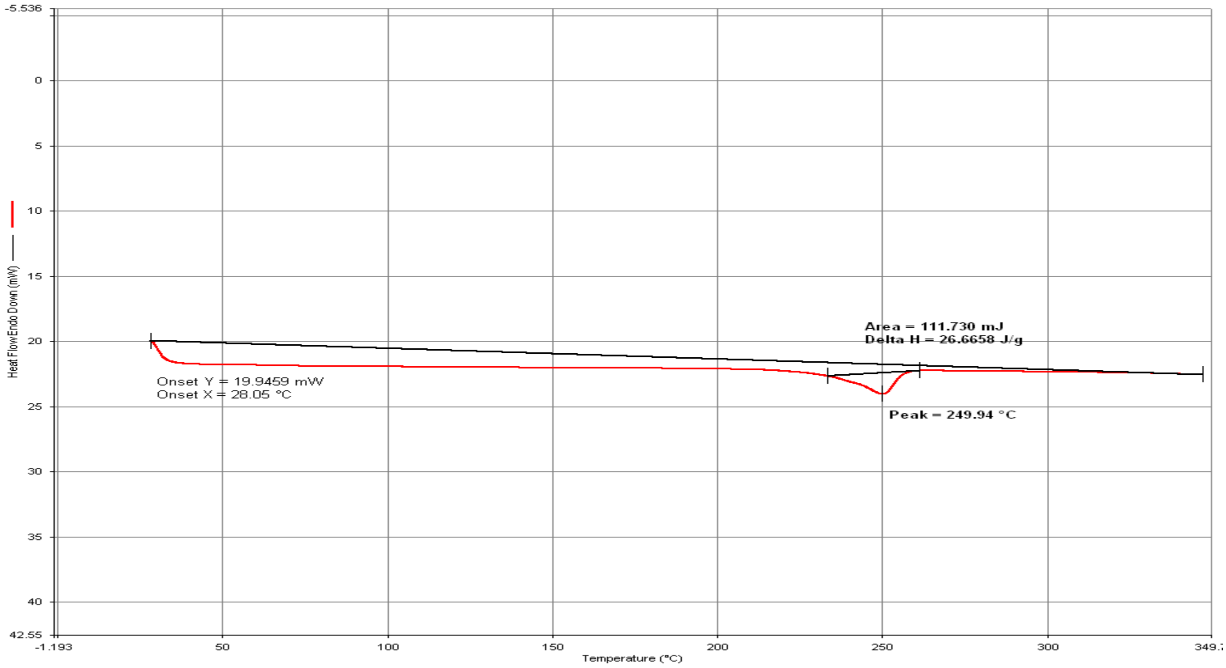

Fig. 3: DSC Scan For 70%PET_30%PEI Blend

Sample ID: 160701_70%PET_30%PEI

Sample Weight: 4.190 mg

Heat from 30.00°C to 300.00°C at 10.00°C/min

Switch the Gas to Nitrogen at 20.0 ml/min

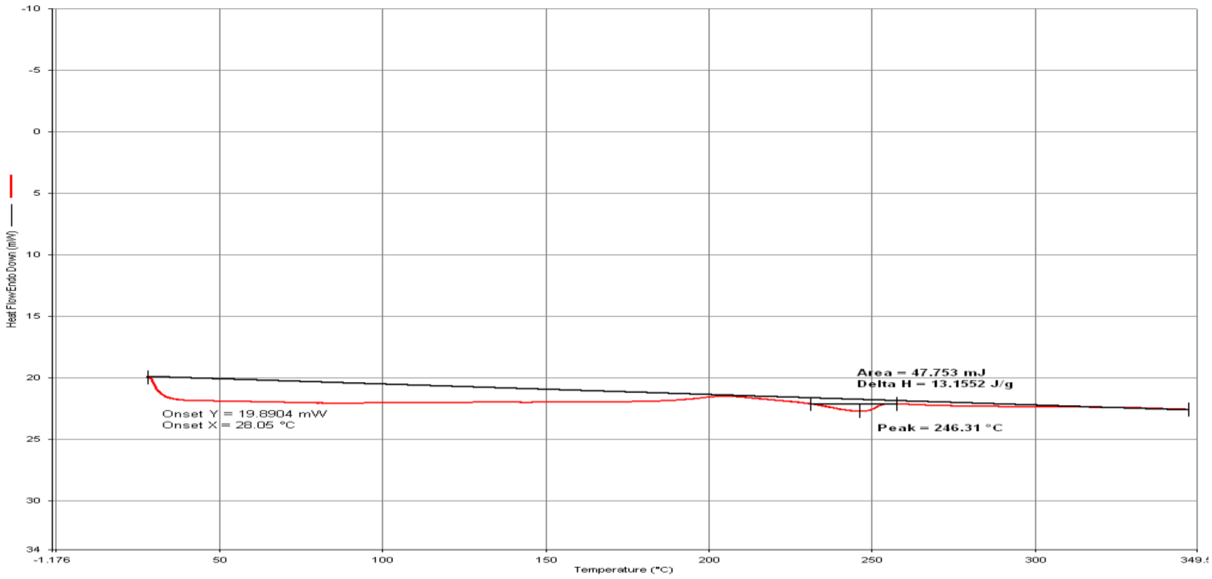

Fig. 4: DSC scan for 50%PET_50%PEI blend

Sample ID: 160701_50%PET_50%PEI

Sample Weight: 3.630 mg

Heat from 30.00°C to 300.00°C at 10.00°C/min

Switch the Gas to Nitrogen at 20.0 ml/min

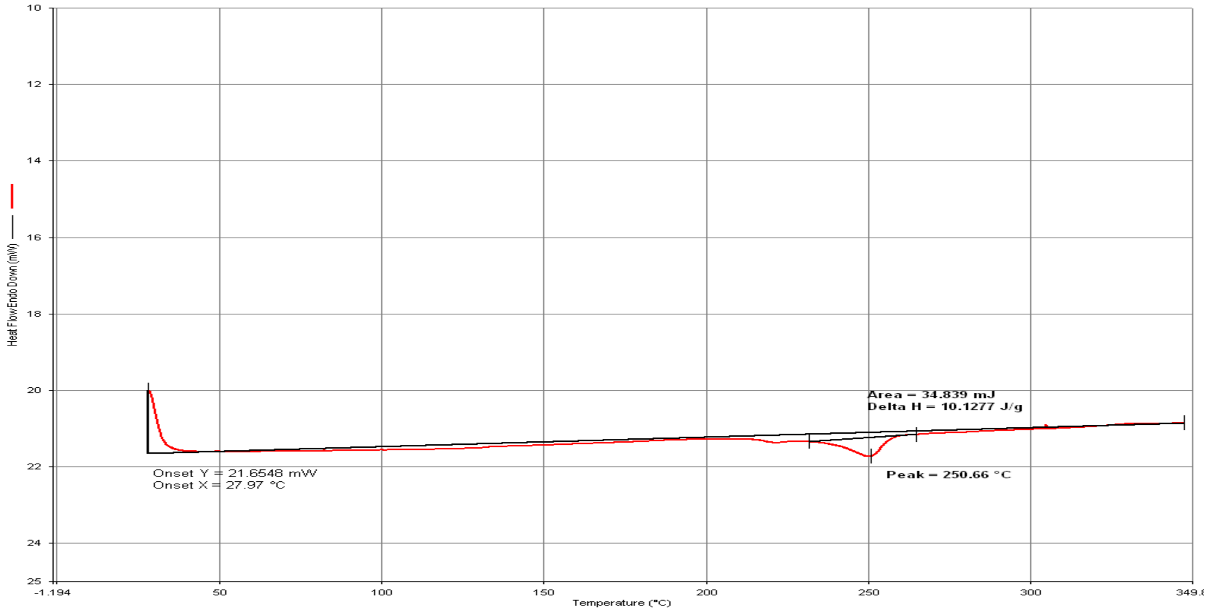

Fig. 5: DSC scan for 70%PEI_30%PET blend

Sample ID: 160630_70%PEI_30%PET

Sample Weight: 3.440 mg

Heat from 30.00°C to 300.00°C at 10.00°C/min

Switch the Gas to Nitrogen at 20.0 ml/min

10.2 Thermo Gravimetric Analysis (TGA)

TGA analysis carried out using Perkin-Elmer thermo gravimetric analysis system TGA-7.all the samples heated from (30°C -900°C) under Nitrogen atmosphere. The measurements of weight loss of the PTT/PEI blends were performed.

Table 3: Thermal stability and weight loss for pure PEI, PET and PEI/PET blends.

Sr. No.	PET % by wt.	PEI % by wt.	5% wt loss Temp	50% wt loss temp	% Remaining wt. at 900 °C
1	0	100	524	890	50
2	100	0	404	434	11

3	70	30	389	434	15
4	50	50	410	510	27
5	30	70	410	600	37

Thermal stability of a polymeric material is one of the most important properties for both processing and application. Thermo gravimetry analysis is the most widely used method to characterize thermal stability of polymers. Table 3. shows the thermal weight loss for pure PET, PEI, and PET/PEI blends. The PET exhibits a well thermal stability below 400°C, begins to decompose at around 384°C. The 5% weight loss temperature is at about 404°C, while The 50% weight loss temperature is at about 434°C and the PET is completely decomposed at 884°C, leaving about 11% residue. From the result of TGA analyses It is noted that the ratio of weight loss is identical to the blend composition Furthermore, the 5%,50% weight loss temperature of the specimen increases with increasing PEI content in the blend. Thus, from this result it is concluded that blending with PEI improve the thermal stability of the PET.fig.8

Pure PEI exhibits high thermal stability along the heating process. The 5% weight loss temperature is at about 524°C, while the 50% weight loss temperature is at about 890°C at the end of the process.fig.9

PEI-PET(30%-70%) blend didn’t show big effect of PEI content on the thermal stability of the blend more than in Pure PET, since The 5% weight loss temperature is at about 389°C, while The 50% weight loss temperature is at about 434°C. and the PET is completely decomposed at 889°C, leaving about 15% residue.fig.10

PEI-PET(50%-50%) blend shows increasing in thermal stability with increasing of PEI content, The 5% weight loss temperature is at about 410°C, while The 50% weight loss temperature is at about 510°C.and at the end of the process at 890°C there was 27% weight remaining.fig.11

PEI-PET(70%-30%) blend shows maximum degree of thermal stability with increasing of PEI content, The 5% weight loss temperature is at about 410°C, while The 50% weight loss temperature is at about 600°C.and at the end of the process at 890°C there was 37% weight remaining.fig.12

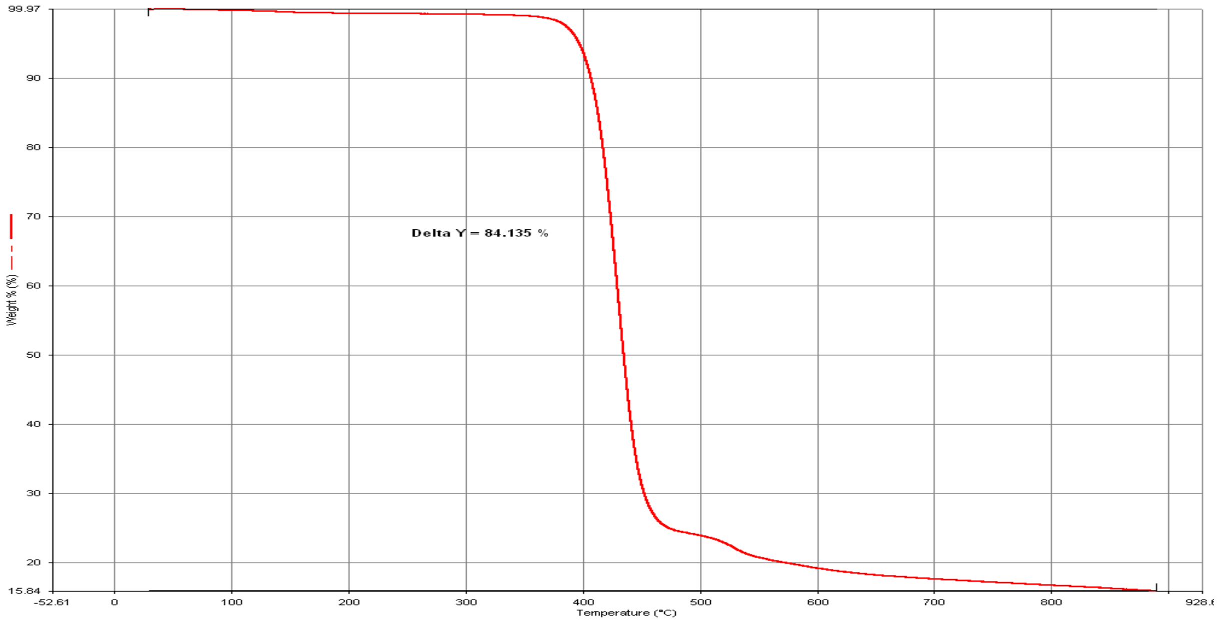

Fig. 6: TGA Scan For 70%PET_30%PEI

Sample ID: 160704_

Sample Weight: 8.326 mg

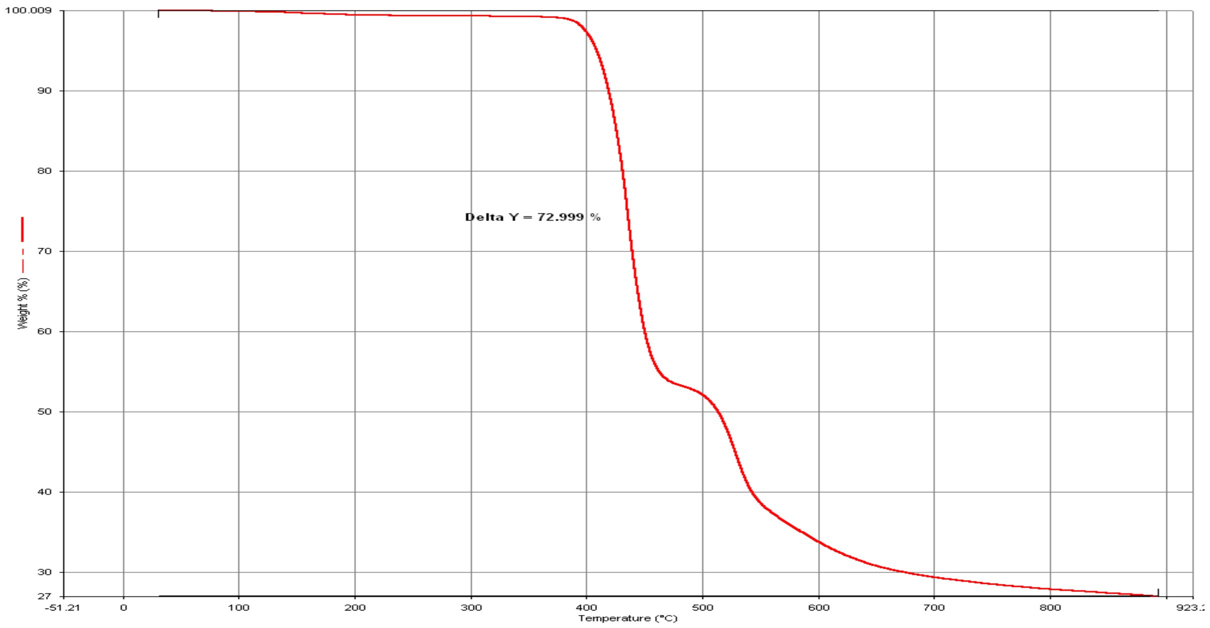

Fig. 7: TGA Scan For 50%PET_50%PEI Blend

Sample ID: 160702_50%PET_50%PEI
Sample Weight: 12.418 mg

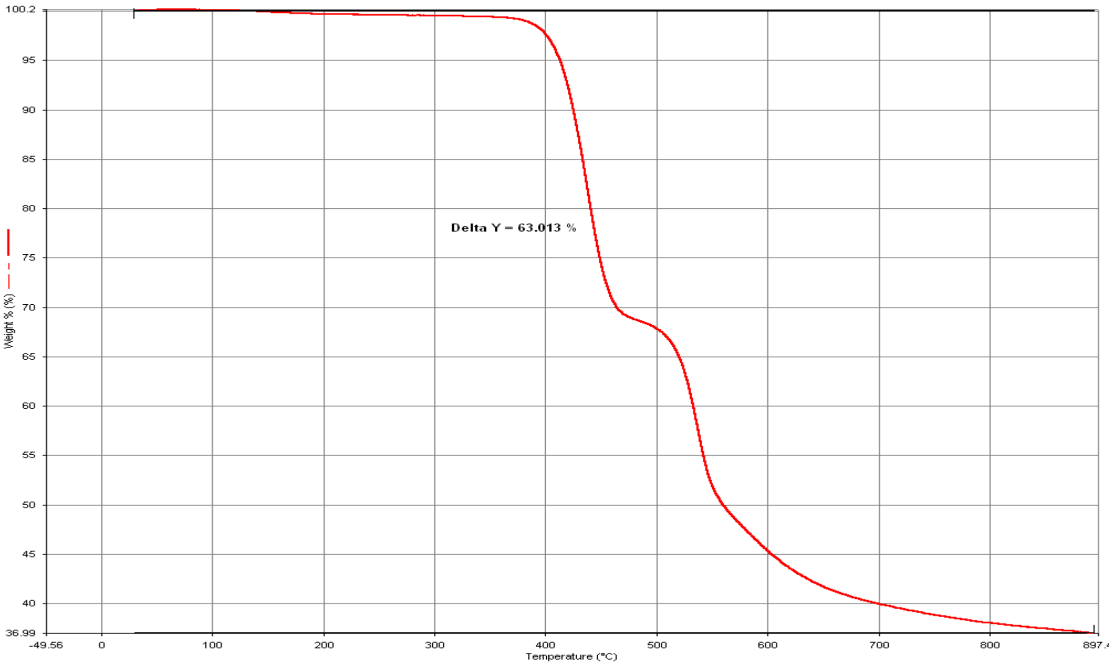

Fig. 8: TGA Scan For 70%PEI_30%PET Blend

Sample ID: 160702_70%PEI_30%PET

Sample Weight: 7.614 mg

XI CONCLUSIONS

In this study, mechanical and thermal properties of PEI/PET blends have been investigated, five samples have been studied ,Pure PEI and PET,PEI-PET(30%-70%), PEI-PET(50%-50%) and PEI-PET(70%-30%) . the results reported in this work show the strong influence that the PEI presence has on the PET in the blends. the increasing of PEI content led to increasing in tensile strength and impact strength of the blends, since PEI is an amorphous thermoplastic has high Tg and Amorphous thermoplastics are generally used at temperatures below their Tg, and requires a large amount of energy to fracture below Tg.

The miscibility, melting, and crystallization behaviors of solution-blended PEI/PET blends have been investigated. from TGA analyses these PEI/PET blends show improved thermal stability with increasing PEI content than the pure PET. From DSC measurements PEI/PET blends are miscible over the whole composition range based on single Tg of these blends. This result further demonstrates that PET and PEI are miscible, and the presence of PEI in the blends retards or even inhibits the crystallization of PET depending on PEI content. The presence of PEI depresses the crystallization of PET during the DSC heating scan. Both crystallization temperature and enthalpy of crystallization decrease with increasing the content of PEI in the blend.

The retardation or inhibition of PET component crystallization can be attributed to: (1) higher Tg of the blend with increasing PEI content results in lower PET chain mobility; (2) high melt viscosity of the amorphous PEI component in the blend tends to decrease the rate of PET segments diffusing to the crystallite–melting interface and leads to lower crystallization growth rate. From above studies it can be concluded that blending of the semi crystalline PET with the amorphous PEI results in a miscible blend and the PET crystallization behavior is altered significantly.

XII ACKNOWLEDGEMENT

I take this opportunity to express my sincere gratitude and appreciation to my guide Prof. (Dr.) Hemant V. Joshi, who was a constant source of my motivation and inspiration .without his guidance and suggestions, this work would have never reached completion.

I would also like to thank Mrs. Prof. (Dr.) Deepti S.Marathe for her advices and comments which helped me a lot.

I would like to thank Mr.A.S.Patil, Lab.Asst., Mr.P.R.Marathe and Mr.B.S.Shaikh Lab.Attendant for helping me through blending and molding processes .

Last but not the least, I am grateful to my family for their unflinching support, encouragement and motivation without which this endeavor was not possible.

Mr.Qayssar M.Alobaidi
Department of Polymer Engineering, M.I.T, Pune

REFERENCES

[1] C. Joly, D. Le Cerf, C. Chappey, D. Langevin & G. Muller, Solvent effect on the conformation in solution of two polyimides, Polymer International 44 (1997) (497-502)

[2] Kathy C. Chuang, Daniel A. Scheiman, Joyce F U, Marcus Crawford,(Synthesis and Characterization of Polyimides with Ether) Linkages, (1999)

[3] Jason E. Lincoln, Roger J. Morgan, E. Eugene Shin, (Effect of Thermal History on the Deformation and Failure of Polyimides), (2001)

[4] Sabrina Carroccio, Concetto Puglisi, Giorgio Montaudo, (Thermal degradation mechanisms of polyetherimide investigated by direct pyrolysis mass spectrometry), Macromol. Chem. Phys. 200, 2345–2355 (1999)

[5] Jonghan Choi and Miko Cakmak,(Morphological evolution during thermal and strain induced Crystallization in poly(ethylene terephthalate)/poly(ether IMIDE) blend films) , journal ISSN :0032-3861DOI10.1016/j.polymer.2015.12.038,(2016)

[6] Robert R. Gallucci and Mark A. Sanner. (PET-Polyetherimde Blends), Proceedings of ANTEC-2011 (Manuscript ID PENG)-11-2010-0077

[7] J. Ramiro, J. I. Eguiazábal, J. Nazábal, (Compatibilized poly(ether imide)/amorphous polyamide blends by means of poly(ethylene terephthalate) addition), DOI: 10.1002/pen.20575,(2006)

[8] Lee JK, Choi WS, Kwon YK, Lee KH,(Liquid-liquid phase separation and crystallization behavior of poly(ethylene terephthalate)/poly(ether imide) blend), Polymer, Vol.43, No.9, 2827-2833, 2002

9] Adhemar Ruvolo-Filho, Adriana de Fatima Barros; Correlation between thermal properties and conformational changes in poly(ethylene terephthalate)/poly(ether imide) blends; Polymer Degradation and Stability 73 pg.467–470, Elsevier , (2001)