ISTANBUL TECHNICAL UNIVERSITY ★INSTITUTE OF SCIENCE AND TECHNOLOGY

USE OF RECYLED POLY(ETHYLENE TEREPHTALATE) FIBERS IN NEEDLEPUNCHED AUTOMOTIVE CARPETS

M.Sc. THESIS

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İĞNELEME YÖNTEMİ İLE ÜRETİLMİŞ OTOMOTİV HALILARINDA GERİ DÖNÜŞTÜRÜLMÜŞ POLYESTER LİFİ KULLANIMI

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Raziye ATAKAN, a **M.Sc.** student of ITU **Institute of Science and Technology** student ID 503021705, successfully defended the **thesis** entitled "**USE OF RECYLED POLY(ETHYLENE TEREPHTALATE) FIBERS IN NEEDLEPUNCHED AUTOMOTIVE CARPETS**", which she prepared after fulfilling the requirements specified in the associated legislations, before the jury whose signatures are below.

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To my family and friends,

FOREWORD

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December 2013   Raziye Atakan
                                                                                          Textile Engineer

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ABBREVIATIONS

RPET   **:** Recycled Poly(ethylene terephthalate)
VPET   **:** Virgin Poly(ethylene terephthalate)
DSC   **:** Differantial Scanning Calorimetry
SEM   **:** Scanning Electron Microscope
IV   **:** Intrinsic Viscosity
DMA   **:** Dynamic Mechanical Analysis
BCF   **:** Bulk Continuous Filament
OEM   **:** Original Equipment Manufacturer
GSM   **:** Grams per Square Meter (g/m²)
PTA   **:** Purified Terephthalic Acid
DMT   **:** Dimethyl Terephthalate
PVC   **:** Poly Vinyl Chloride
PVA   **:** Poly Vinyl Acetate
TGA   **:** Thermo Gravemetric Analysis
FTIR   **:** Fourier Transform Infrared Spectroscopy

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USE OF RECYCLED POLY(ETHYLENE TEREPTHALATE) IN NEEDLEPUNCHED AUTOMOTIVE CARPETS

SUMMARY

The investments in the automotive industry in Turkey have a significant potential for automotive technical textiles. Turkey's aim is to take place among leading automotive textile manufacturers and suppliers in the world. In this case, car carpets are among the most essential parts in the automotive interiors.

Car carpet was once considered a luxury item, but it is now an essential part of interior trim. This is due to its contribution as an aesthetics and sensual comfort component, and also due to the part it plays in noise and vibration control.

Each car contains about 3.5 to 4.5 m² of carpet, made by either tufting or needlepunching. In recent years, needlepunched carpets have appeared in the world and demand is expected to grow. An increasing trend worldwide is to produce lighter carpets using finer fibers; this has more covering power towards the lower weights-approximately 405 g/m² (12 oz/yd²) for tufted and 450 g/m² for needlepunched or even lower.

Needlepunching of car carpets is an even younger process than tufting and is an even more rapid and economical process. Needlepunching has been much refined in recent years and is now producing very attractive materials, comparable in quality with tufted carpets.

Needlepunched carpets are normally composed of synthetic textile fibers such as polyester and polypropylene. Although, both are versatile fibers, preferably, polyester is also recyclable in higher tenacity grades.

Developing technologies for processing recycled material would bring for the automotive industry not only cost savings but also sustainability and efficiency in terms of material utilization. In this case, nonwoven carpets offer various possibilities to include recycled fiber material in the production. Recycled fibers have been successfully used for the development of lightweight car carpet structures.

The objective of this study was the development in the processing of recycled fibers into lightweight carpets for automotive.

For that purpose, in this study, virgin PET fiber and Recycled PET fibers from different suppliers were tested and different blends of RPET and Virgin PET were prepared for production.

Moulded carpets were developed from these blends at HP Pelzer Pimsa A.Ş. The process involved opening of fiber bales, followed by web formation in a card, and web building on a crosslapper. This was followed by the nonwoven formation by needlepunching, which forms the face layer of the moulded carpet. Subsequently, back coating was done with a binder in the form of foam, cured in a stenter and laminated by LDPE powder. This intermediate product was then blank cut and hot

pressed in moulding machines of specific size and shape, and trimmed to give the final moulded carpet.

Finally, these carpets were evaluated due to required spesifications. According to the results obtained, the best carpet blend and the best production parameters were identified. The study helped to obtain high quality, low cost needlepunched carpet composite using recycled PET fibers.

İĞNELEME YÖNTEMİ İLE ÜRETİLMİŞ OTOMOTİV HALILARINDA GERİ DÖNÜŞTÜRÜLMÜŞ POLYESTER LİFİ KULLANIMI

ÖZET

Otomotiv endüstrisi, dokusuz yüzeylerin en büyük kullanıcılarından biridir. Yapılan bir araştırmaya göre her bir araç için otomotiv iç mekânında 14,6 ve 28 m^2 arasında dokusuz yüzey kullanımı gerekmektedir. Bu rakam, dokusuz yüzey malzemeler için talebi açıkça ortaya koymaktadır.

Yeni malzemeler, kullanım teknikleri ve uygulamalar maliyet ve performansa bağlı olarak hızlı şekilde büyümektedir. Ancak, dokusuz yüzeylerin kullanımı maliyet ve gramajı azaltmanın yanısıra diğer ürünlerden daha fazla avantaj getirmektedir. Bu avantajların arasında çok geniş yoğunluk aralığında üretilebilmeleri, akustik ve ses kontrolü, üretim esnasında daha kolay şekil alabilmeleri ve spesifik özelliklere sahip olacak şekilde tasarlanabilmeleri de yer almaktadır.

Otomobillerde zemin kaplamalarında ses geçirmezlik önem taşımaktadır, çünkü bu halılar araba gövdesinin en fazla ses emisyonu olan kısımlarını kaplamaktadır. Bu spesifik özelliklere paralel olarak günümüzde, otomotiv iç mekanlarında dokusuz yüzeylerin kullanımına yönelik çok sayıda önemli ve heyecan verici gelişmeler gerçekleşmektedir.

Otomobilde kullanılan kalıplanmış zemin halıları, koltuk kılıfları, tavan astarları ve döşemelikler gibi ürünler önemli özelliklere sahip olmaları ve maliyet avantajı sunmalarına bağlı olarak dokusuz yüzey olarak üretilebilmekte ve bu da dokusuz yüzey ürünler için önemli bir pazar oluşmasını sağlamaktadır.

Türkiye'deki otomotiv endüstrisi yatırımları otomotiv tekstilleri için önemli bir potansiyel oluşturmaktadır. Türkiye'nin amacı, dünyadaki lider otomotiv tekstil tedarikçileri arasında yer almaktır. Bu bağlamda, otomobil halıları otomobil içindeki en gerekli parçalardır.

Bir zamanlar lüks olduğu düşünülen otomobil halıları, hem estetik açıdan, hem konfora katkısı bakımından hem de ses ve titreşim kontrol parçaları olarak artık iç döşemelerin gerekli birer parçası haline gelmiştir.

Her araba, tafting ya da iğneleme yöntemiyle üretilmiş 3,5-4,5 m² halı içermektedir. İğneleme yöntemi ile üretilmiş halılar son yıllarda ortaya çıkmış olup, talep gittikçe artmaktadır. Dünyadaki eğilim, daha ince elyaf kullanarak yüzey kaplama faktörü daha yüksek, daha düşük gramajlı halılar üretmek yönündedir ki bu da yaklaşık olarak tafting metoduyla üretilmiş halılar için 405 g/m² ve iğneleme yöntemiyle üretilmiş halılar için ise 450 g/m² ya da daha düşük değerlere karşılık gelmektedir.

İğneleme yöntemi ile keçeleştirme işlemi bu uygulamada daha çok tercih edilen bir yöntem olmaya başlamıştır. Çünkü iğneleme yöntemi, tafting metoduyla üretimden daha yeni bir işlem olup aynı zamanda daha hızlı ve de ekonomiktir. İğneleme yöntemi ile keçeleştirme son yıllarda oldukça geliştirilmiştir ve bugün bu yöntemle

tafting halılarla kıyaslanabilecek kalitelerde, dikkat çekici malzemeler üretilebilmektedir.

İğneleme yöntemi ile üretilmiş halılar normalde polyester, polipropilen gibi sentetik liflerden oluşmaktadır. Bazı firmalar ise polyester ve polipropilen karışımı kullanmaktadır. Her iki lif de çok yönlü olmasına rağmen, polyesterin aynı zamanda daha yüksek mukavemet değerlerinde geri dönüştürebilir bir malzeme olması daha çok tercih edilmesine neden olmaktadır.

Geri dönüştürülmüş malzeme işleme teknolojilerinin geliştirmesi ile otomotiv endüstrisi için sadece maliyet tasarrufu değil aynı zamanda malzeme kullanımı açısından sürdürülebilirlik ve verimlilik sağlamaktadır. Bu bağlamda da dokusuz halılar, üretimlerinde geri dönüştürülmüş elyaf kullanımı içerebilmesi açısından oldukça çeşitli imkânlar sunmaktadır.

Geri dönüştürülmüş elyaflar, hafif gramajlı araç halılarının geliştirilmesinde başarılı bir şekilde kullanılmaktadır. Bütün otomotiv tekstilleri üreticileri için araç halısı üretimlerinde geri dönüştürülmüş elyaf kullanımını arttırmak, fakat üretimde bu materyaller kullanılırken hem görsel hem de fiziksel performans olarak kaliteden taviz vermemek büyük önem taşımaktadır.

İğneleme yöntemi ile üretilmiş ve daha sonra kalıplanmış halıların kalitesini etkileyen kritik faktörler arasında hammadde seçiminden başlayıp üretimin çeşitli aşamalarında proses parametrelerinin seçimine kadar uzanan süreç yer almaktadır. Bir otomobil üreticisinden diğerine spesifikasyonlar ve kalite talepleri değişiklik göstermektedir.

Tüm otomobil üreticileri için ortak olan başlıca kalite talepleri; mukavamet, aşınma dayanımı, boncuklaşma dayanımı, uzama kabiliyeti, yüksek sıcaklıklarda ışık haslığı, kolay temizlenebilmesi, boyusal değişim özellikleri, yanmazlık, renk solma dayanımı, tüylenme dayanımı, eskime dayanımı, sertlik gibi özelliklerden oluşmaktadır.

Proses gereklilikleri olarak ise kolay kalıplanabilme, ısı stabilitesi, dikilebilme, ıslanabilme, iyi yapışma, sıkıştırmaya karşı duyarlılık ve araba kullanımı süresince dayanım gösterme gibi özellikler öne çıkmaktadır.

Ayrıca, kalıplanmış halılarda ürünün reddedilmesine neden olabilecek bazı kalite problemleri ortaya çıkabilmektedir. Bunlar; renk uyumsuzluğu, kirlilik, gramaj varyasyonları, aşınma dayanımında problemler ve halılarda gözlenen çizgiler olarak sıralanabilir.

Otomotiv üreticileri kalite konusunda yüksek bilinç derecesinde sahiptir. Ürünü ekonomik olarak üretebilme, bu amaç doğrultusunda üretim maliyetlerini düşürme, ürün kalitesini arttırma ve bunun sonucunda da sektöre rekabet edebilirliğini arttırmak en büyük hedefleridir. Sonuç olarak, gittikçe daha fazla rekabete sahne olan pazarda söz sahibi olmak, daha yüksek pazar payına sahip olmak ve malların reddedilmesini önlemek için iğneleme yöntemiyle üretilmiş ve kalıplanmış halı üreten firmalar ürün kalitesini arttırmaya yönelik çalışmalar yapmaktadır. Bu doğrultuda, prosesten çıkan atıkların geri dönüşümü, çevre ve insan sağlığı için tehdit oluşturmayan geri dönüşebilir malzeme kullanımı, enerji tüketiminin azaltılması, üretim fire oranlarının düşürülmesi konularındaki arge çalışmalarına eğilim oldukça fazladır.

Bu çalışmanın amacı; hafif gramajlı, en katı spesifikasyonu karşılayabilecek derecede yüksek kaliteli otomobil halıları üretimlerinin, hem çevresel faktörler gereği hem de maliyet avantajı sunması nedeniyle geri dönüştürülmüş PET elyafı kullanarak geliştirilmesidir. Bu amaçla farklı üreticilerden tedarik edilen işlenmemiş ve geri dönüştürülmüş PET elyafları, hammadde kalite tayini için test edilmiş ve halı üretimi için farklı oranlarda elyaf karışımları hazırlanmıştır.

Bu karışımlardan HP Pelzer Pimsa A.Ş'de kalıplanmış otomobil halıları geliştirilmiştir. Üretim aşamaları, elyaf balyalarının açılması, tarak makinesinde tülbent oluşumu, çapraz serim tekniğiyle tülbent serme işlemlerini içermektedir. Bu işlemlerden sonra kalıplanmış halının üst tabakasını oluşturan dokusuz yüzeyin iğneleme yöntemi üretilmesi gerçekleştirilmiştir. Akabinde de, köpük formundaki bir binder ile arka kısmın bağlanması, ramözde kurutulması ve düşük yoğunluklu polietilen toz kullanımı ile laminasyon işlemi yapılmıştır. Bu ara ürün, daha sonra kesme makinasında kesilmiş ve sıcak olarak kalıplama makinasında belirli boyutta preslenmiştir ve son olarak halıya son şeklini vermek üzere tekrar kesme işlemi uygulanmıştır.

Son olarak, bu halılar istenen spesifikasyonlara göre değerlendirmeye tabi tutulmuştur. Bulunan sonuçlara göre en iyi kaliteyi veren elyaf karışımı ve en iyi üretim parametreleri tespit edilmiştir. Bu çalışma, geri dönüştürülmüş elyaf kullanılarak yüksek kalitede, düşük maliyetli iğneleme yöntemiyle üretilmiş otomobil halısı geliştirilmesine yardımcı olmuştur.

1. INTRODUCTION

Textile materials are conquering the automobile and will even be deployed in future applications. Even today, most cars contain more than 20 kilograms of manmade and natural fibers: in seat upholstery and belts, airbags and cladding, filter and insulating materials and in numerous further applications (Figure 1.1). Textile researchers anticipate that this will increase to 30 kg and even more by 2015. The reason for this: textiles are increasingly assuming functions relating to comfort, acoustics, safety and fuel economy.
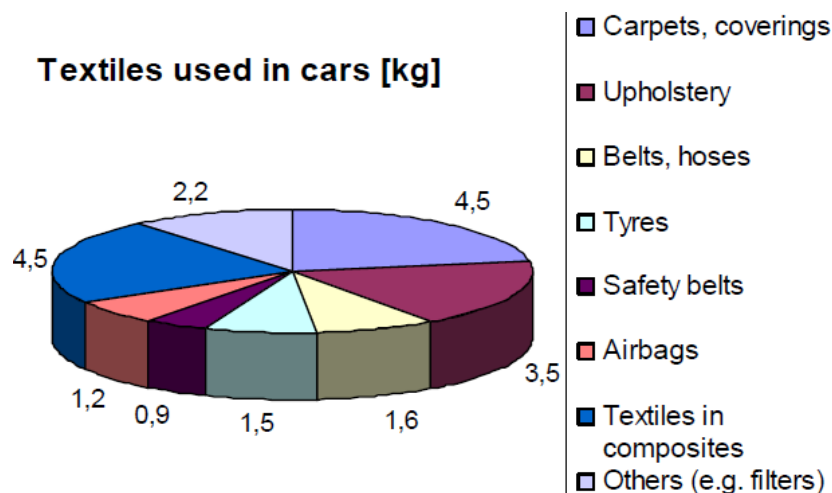
Figure 1.1 : Textile used in cars (kg) (Edana, 2013).

The average weight of textile materials in a mid-size car has increased from 20 kg in 2000 to 26 kg today, and by 2020, it is expected to reach 35 kg (Figure 1.2). The increase stems from a rise in demand for greater comfort and safety, and efforts aimed at reducing the weight of a vehicle in order to lower fuel consumption and CO_2 emissions (Edana Newsletter, 2013).

In terms of fabric type, it has been predicted that woven fabrics and knitted fabrics will continue to account for a predominant share of the global market for automotive textiles. This prediction holds true despite a rise in the use of nonwovens and an increase in the penetration of composites.

Figure 1.2 : Textiles in cars-development in use.

Nonwovens are being increasingly employed in vehicles because of their low weight and low cost, according to a new report, which predicts that the use of textiles in the automotive industry is set for significant growth (Figure 1.3).

Figure 1.3 : Nonwovens in car (Edana, 2013).

As far as nonwovens are concerned, there are now more than 40 applications for nonwovens inside cars, equating to over 35 m² of flat surfaces, according to the report, Automotive Fabrics: Expanding Opportunities in the Vehicles of Tomorrow, published in the latest issue of Technical Textile Markets from the business information company Textiles Intelligence (Edana Newsletter, 2013).
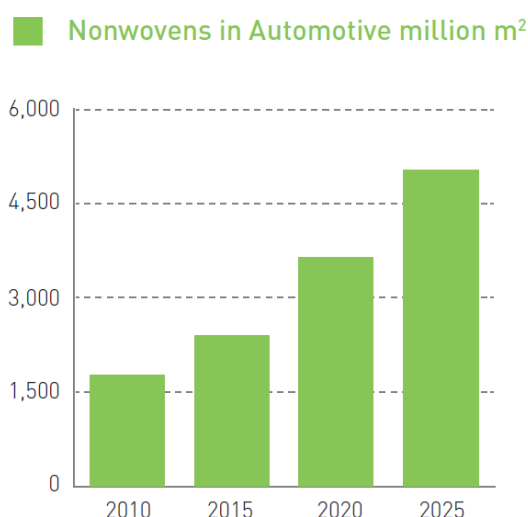
Figure 1.4 : Utilization expectations of nonwovens in automotive (Edana, 2013).

The consumption of nonwovens is currently around 28m² per vehicle and this is expected to rise to over 40 m² per vehicle in the future (Figure 1.4). Visible nonwovens, however, account for only 10 percent of the entire amount, i.e. roughly 3.5 m² (Edana Newsletter, 2013).

Furthermore, the use of recycled materials in nonwovens for automotive applications is increasing. A growing proportion of needlepunched nonwovens for automotive applications, for example, is being manufactured from recycled polyester derived from plastic bottles.

It is desirable to have good quality nonwovens produced from recycled fibers by all automotive textile producers for economic and ecologic reasons. In this respect, a car carpet is a significant part of nonwoven automotive textiles as it allows the utilization of recycled PET. A producers list is shown in Figure 1.5.

Fibertex	Denmark
J.H. Ziegler	Germany
Freudenberg Nonwovens	Germany
Hassan Group	Turkey
IMS Nonwoven	Sweden
Bonar	Netherlands
Rubi Industrial	Spain
Sandler	Germany
TENOWO	Germany
Hollingsworth & Vose	Germany
IPETEX	Portugal
JX Nippon ANCI	France
TWE Group	Germany
Exten	Switzerland
CHA Technologies Group – Cosmotec	UK
Komitex	Russia
Tessiture Pietro Radici	Italy

Figure 1.5 : List of members of the automotive interest group (Edana, 2013).

Each car contains about 3.5 to 4.5 m² of carpet, made by either tufting or needle punching. In a common method, solution-dyed extruded polyester fiber is "needle-punched." In other common method, a colored extruded bulk continous filament (BCF) nylon fiber is more thickly "tufted" on a fabric backing material. Tufted automobile carpets typically are more expensive than needle-punched ones (United States Patent, No: 0159064, 2005).

Both tufted and needlepunched carpets require about 70-100 g/m² of a binder coating on the back, usually SBR or acrylic latex, to stabilize them and to lock in the fibres. Another layer of suitable material is then applied to both types of carpets to confer good thermomouldable properties. This is important for process efficiency, and also to produce a good fit, which will reduce vibration and maximize noise insulation. PE powder is used on both tufted and needlepunched carpets for this purpose, and about 250-600 g/m² is applied by powder scattering and IR heating (Fung, Hardcastle, 2001). An automotive carpet structure is given in Table 1.1.

Table 1.1 : Automotive carpet structure (Fung, Hardcastle, 2001).

Layer	Materials used
Top decorative	Tufted BCF nylon or needlepunched polyester or polypropylene back, latex coated with SBR or acrylic latex
Thermoforming	Polyethylene powder, melteable fibres, EVA or a further thick layer of compounded SBR latex
Acoustic	Heavy layer' of EPDM, shoddy fibres or polyurethane foam

Needled textile fabrics are normally composed of synthetic organic textile fibers, such as polyester or polypropylene fibers, needled together into a consolidated mat. Such fabrics find a variety or applications where relatively high physical properties are required, e.g. high strengths, with substantially uniform physical properties in both the longitudinal and widthwise direction, and particularly in those applications where economics dictate the use of materials less expensive than tufted fabrics or where the applications require more uniform thickness direction properties than tufted fabrics (United States Patent, No: 0159064, 2005).

1.1 Purpose of Thesis

Today, in automotive technical textiles industry, the products have to conform high quality standards in visual and physical performance. In addition to this, they have to be cost-effective. This has led to the use of recyclable materials in the production. Environmental concerns have also made the use of these materials essential.

Because of regulations, one of the most preffered areas in the use of recycling materials is carpets. In parallel to the increase in utilization rates of recycled materials, carpet quality problems that are encountered are also increasing.

The main objective of this study is selection of appropriate recycled PET fibers or fiber blends to be used in automotive carpets, which could be a solution to this problem.

2. NONWOVENS IN AUTOMOTIVES

The expansion of the industrial textile market is good for the textile industry and automotive textiles in particular are frequently in the media (Chapman, 2010). An important factor in this is that the annual capacity of global vehicle production (private cars and freight vehicles) reached about 68 million units by 2013.

From the figure 2.1, it is seen that in 2012, for the first time in the history, over 60 million passenger cars was produced in a single year (or 165,000 new cars produced everyday). Only a 9% decline in 2009 (due to the global financial crisis), global car production immediately jumped back the following year with a 22% increase in 2010, to then consolidate at the current 3% yearly growth rate.

When added to the 885 million units of existing motor vehicles, the worldwide automobile consumption forms a substantial market for automotive textiles. Figure 2.1 lists the automotive consumption among different geographical regions.

International Car Sales Outlook (millions of units)						
	1990-99	2000-09	2010	2011	2012	2013f
TOTAL SALES	39.20	49.93	58.58	60.80	64.94	68.13
North America*	16.36	18.38	13.96	15.22	17.07	18.31
Canada	1.27	1.59	1.56	1.59	1.68	1.75
United States	14.55	15.79	11.55	12.73	14.40	15.50
Mexico	0.54	1.00	0.85	0.90	0.99	1.06
Western Europe	13.11	14.39	12.98	12.80	11.76	11.29
Germany	3.57	3.33	2.92	3.17	3.08	2.93
Eastern Europe	1.18	2.71	3.14	3.90	4.14	3.94
Russia	0.78	1.53	1.91	2.65	2.93	2.75
Asia	6.91	11.93	24.23	24.41	27.25	29.72
China**	0.43	3.85	11.17	12.16	13.18	15.55
India	0.31	0.89	1.87	1.95	2.02	1.87
South America	1.64	2.52	4.27	4.47	4.72	4.87
Brazil	0.94	1.57	2.69	2.64	2.84	2.81

*Includes light trucks. **Includes crossover utility vehicles from 2005.

Figure 2.1 : Annual sales of cars (million unit) (http://www.gbm.scotiabank.com/ English/bns_econ/bns_auto.pdf)

Proportional to the increase of global motor vehicle production, use of automotive textiles keeps growing. Textile materials used for automotive manufacture are very diverse, including woven fabrics, knitted fabrics, nonwoven fabrics, coated fabrics,

and fabric composites (combinations of different fabrics, or combinations of fabrics with plastic materials) (Chapman, 2010). Figure 2.2 presents the global production growth of automotive textiles since 1985.

Worldwide production of automotive textiles					
	Production in 1,000 tonnes				
	1985	1990	1995	2000	2005
Fabrics, Knits	953	1,125	1,170	1,221	1,214
Nonwovens	95	109	107	111	114
Composites	284	443	521	736	976
Others	76	97	119	152	179

Source: Frost & Sullivan

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Figure 2.2 : Worldwide production growth of automobile textiles (1,000 tones).

It is known that consumption of textile products in each car is about 26 kg today. This consumption level of textiles is equivalent to 3% of the total raw materials (in weight) used in a vehicle, compared to the consumption of steel 60%, plastics 20%, aluminum 15%, and other materials 2% (Bottcher, 2002).

The automotive industry is one of the largest users of "engineered" nonwovens (Smith, 2004). While the use of woven and knitted fabrics in automotive applications shows only minimal growth (Table 2.2), nonwoven fabrics are increasingly used in the automotive industry. Major driving factors for growth include:

- Function, cost, weight, advantages, and acoustics improvements are major driving forces for use.
- Severe price/cost pressure by car manufacturers will continue-both opportunity and concern for nonwoven producers.
- Europe, Japan and the NAFTA (US, Canada, Mexico) producers favor different materials, but those differences are likely to merge a global industry.
- Recyclability will be a more pressing concern.
- Interior major emphasis and the major profit potential (Smith, 2004, Chapman, 2010).

    Table 2.1 represents some major producers in automotive textile engineering.

Table 2.1 : Some major producers in automotive textile engineering (Fung and Hardcastle, 2001).

Original equipment manufacturers (OEMs) car makers

General Motors	Fiat
Ford	PSA Peugeot/Citroen
Toyota	BMW
DaimlerChrysler	Porsche
Renault/Nissan	Mitsubishi
Volkswagen	Daewoo
Honda	Hyundai

Heavy goods vehicles (over 6 tonnes gross vehicle weight) manufacturers

DaimlerChrysler (Freightliner)	ERF
Navistar (formerly International Harvesters)	MAN
RVI, Renault (Mack)	General Motors (Bedford)
Ford	Isuzu
Volvo/Scania	Dongfeng
Paccer (DAF, Forden, Kenworth, Peterbilt)	Hino
Iveco, Fiat (Margirus Deutz, Unic)	Mitsubishi

Some Tier-1 Suppliers

Company	**Main activity involving textiles**
Delphi	Seating, airbags
Visteon	Seating
Johnson Controls	Seating, headliners, door panels
Lear	Seating, headliners, door panels, floor systems
Magna	Seating, door panels
Textron Automotive	Seating, door panels
Ikeda Bussin	Seating
Collins & Aikman	Floor systems, door panels, interior trim
Trevers	Seating, door panels, floor systems
Sommer Allibert	Door panels, headliners, interior trim
Faurecia	Seating, door panels
Rieter	Floor systems, interior trim
Autoliv	Airbags, seat belts
TRW	Airbags, seating
Toyota Gosei	Airbags
Toyko Seat	Seating, door panels, headliners

Some automotive fabric suppliers

Woven/knitted Fabrics	**Non-woven fabrics**
Collins & Aikman	Freudenberg
Guilford Mills	Cosmopolitan
Milliken	Lohmann
Joan	Fibertex
Viktor Achter	Lantor
Achter and Abels	Fiberduk
De Witte	US Nonwovens
Aunde	National Nonwovens
Borgstena	Texidel
Axelsons	Dexter
Deutsche Bobinet	Libeltex
Eybl	BFF Nonwovens
Thiery	Sandler
Fidivi	Sommer
Kawashima	
Seiren	
Suminoe	
Treves	

New materials, usage, techniques, and applications are growing extensively due to cost and performance. However, their usage is for more than just reducing cost and weight. Nonwovens often do their job better than other products. A good example is acoustics or noise control. Low road noise is often a factor in a sense of "quality". In manufacturing, nonwovens are often more easily conformed to shapes to help in the fabricating process. Frequently, they offer better "value-in-use", and can be engineered to have specific properties, whether balanced or unidirectional, and can often be combined with other materials, most often woven or knit textiles, in a beneficial way. Over 40 automotive parts utilize engineered nonwovens and the list is growing (Smith, 2004).

Table 2.2 lists typical nonwoven applications for auto interior production. The biggest portion of the nonwoven usage in a car is carpet (in both passenger and trunk compartment areas) and insulation, accounting for 73% of the total nonwoven usage.

Table 2.2 : Nonwovens as auto interior materials (Camelio, 2003).

Interior part	Component	Nonwoven area usage (%)	Nonwoven technology
Passenger carpet	Decor fabric, first and second layer of underlayment	43	Needle-punch, spunbond
Insulation	Acoustic and thermal insulators whereever needed	17	Needle-punch, spunbond
Trunk trim	Floor cover, insulating layer, side liners	13	Needle-punch, spunbond
Rear shelf/ package tray	Facing and backing fabrics	3	Needle-punch
Hoodliner	Absorbing layer	10	Needle-punch
Headliner	Decor fabric, insulator, substrate	6	Needle-punch, spunbond
Seat	Decor fabric, back fabric, bolster fabric, construction reinforcement	6	Needle-punch, spunbond, spunlacing
Door trim	Lower facing, panel trim (door insert/bolster)	1	Needle-punch, spunbond, spunlacing
Miscellaneous		1	Needle-punch, spunbond, spunlacing

To meet the increasing demand for automotive nonwovens, nonwoven producers are intensively focusing on enhancement of the production capacity of high quality and versatile nonwoven materials. All new nonwoven technologies can be employed to meet this industrial need. These nonwoven technologies include spunbond, needle punching, and spunlacing (Camelio, 2003).

Spunbond (66%) and needled (27%) materials are the dominant manufacturing types. Fibers are mostly polyester (PET) and polypropylene due to cost and good UV resistance. Nylon is mostly in tufted carpets, headliner and seating in the form of woven, knit or tufted face fabrics. Solution dyed fibers are finding greater use in order to meet increasing demands for high UV resistance.

A breakdown of major nonwoven usage include areas such as carpet (43%), headliner (6%), hoodliner (10%), trunk (13%), insulation (17%), door panels (1%), seating (6%), package trays (3%), and other miscellaneous areas (1%) (Smith, 2004). Table 2.3 lists the parts of an automobile where nonwovens are used.

Table 2.3 : Where nonwovens are used in an automobile (Smith, 2004).

Face Material	Construction	Filtration
Seating	Seating	Engine Air
Headliners	Headliner	Interior Air
Carpets	Carpets	Tranmission
Trunk Liner	Door/Dash Panels	Oil
Package Tray	Acoustic	Brake hose
Door Panel &Trim	Adhesives	
Pillar Cover	Fire Blocker	
	Other	
	Battery Seperators	
	Tire reinforcement	
	Electrical insulation	
	Gaskets/Seals	
	Muffler	
	Catalytic converter	

2.1 Nonwoven Moulded Automobile Carpets

A large volume automotive fabric, and a major target area for nonwovens, involves carpets. While most carpets in automobiles and light trucks (which include minivans and SUVs) in the NAFTA countries (US, Canada, Mexico) are of tufted pile fabric, nonwovens play a crucial role as primary and/or secondary backing materials (Smith, 2004). For a medium-size car, it is known that use of carpet in the driver/passenger area is about 3.5-4.5 m². If including all carpet underlay materials, the weight of a floor carpet interior accounts for about 33% of the weight of total auto interior materials (Nick, 2005). The carpet interior has two functions: floor covering for aesthetical reasons and floor insulation for noise barrier and noise absorption. A

typical structure of the passenger carpet system in a gasoline engine car includes a decor layer and an insulating layer, as shown in Fig. 2.3. For diesel engine vehicles, a secondary insulating layer is often needed to enhance noise insulation performance. The decor fabric is made of a needle-punched nonwoven or a tufted fabric. However, the use of tufted fabrics for automotive carpets is very limited in current auto interior design (Chapman, 2010).
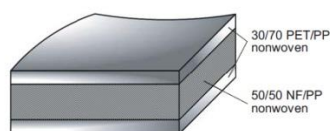
Figure 2.3 : Nonwoven auto carpet construction (Chapman, 2010).

The insulating layer of the carpet system can be either a foam layer or a nonwoven felt layer. Table 2.4 summarizes the major characteristics of nonwovens used in the auto carpet system (Pollock, 2008). Basic physical properties of the auto carpet required by OEMs include excellent resistance to wear and pilling, easy and safe material handling, excellent mechanical resilience, low extensibility and moderate stiffness, and cost effectiveness (Chapman, 2010).

Table 2.4 : Nonwoven characterictics for auto carpet (Chapman, 2010).

Component	Parameter	Value
Decor Layer	Fibre type	Polyproplylene, polyester
	Fibre fineness and length	3-20 dtex, ~40 mm
	Carpet weight	260-550 g/m²
	Carpet thickness	5-7 mm
	Surface structure	Flat felt
		Random Velour[1]
		Patterned Velour[1]
	Back Structure	Latex coating
		Polyethylene coating
		Polyproplylene thermo-bonding
Insulating Layer	Fiber type	Recycled fibers[2]
		Natural fibers[3]
	Fiber fineness and length	Various
	Felt weight	400-1200 g/m²
	Felt thickness	10-20 mm

[1]In some cases, random and patterned velour carpets are also called dilour.
[2]Shoddy fibers are often used to blend with 10–20% biocomponent bonding fibers. [3]Natural fibers are often blended with 30–50% polypropylene bonding fiber.

Needle-punched nonwovens have become dominant in this application because of their special properties and cost advantage. Nonwovens have better adhesion property than woven and so binder application is more uniform. They also possess higher thickness for a given weight per unit length and so are more voluminous and comfortable. In addition they have a more uniform, smooth and random surface. As a result, nonwovens are preferred for moulded carpets used in modern cars (Balasubramanian, 2003).

Automobile manufactories are highly quality concious. They also have some challenges to conform contrary customer requests. These challenges include increased use of recycled fibers, higher homogeneity of nonwovens and higher reproducibility. Therefore, they are required to upgrade their quality monitoring systems and procedures to establish a reliable name in the market, achieve a higher market share and keep down rejections. The critical factors that affect quality of nonwoven moulded carpets include raw material selection, process parameter selection at different stages of manufacture and the precautionary measures.

All carpet materials have to be selected for mouldability, good adhesion to the fibre and other substrates, heat stability to withstand further processing in the car factory, and also durability during the life of the car (Fung, Hardcastle, 2001).

While some manufacturers utilize single shade of fibre in nonwoven manufacturing, some of them could prefer a blend of 2 to 3 shades of fibers to get the shade requirements. When a blend of fibres is used, more precautaions are needed to ensure uniformity in blend proportion and colour of carpet.

PP and PET are the fibres mainly used in making needle punched fleece which forms the top surface of the carpet. Some manufacturers also use the blend of PP and PET. Weight (gsm) of the nonwoven fleece as well as final moulded carpet vary widely from car to car and differ from 640 and 1050 g/m². Plain, Rib and Velour surfaces could be found. For velour type, random and uniform lay of the piles are significant. Velour machines have to be used with a suitable arrangement of fork needles in the board to get velour surface.

2.2 Raw Materials

Polyester and polypropylene are the commonly used fibers for manufacture of needlepunched fleece, which forms the top surface of the carpet. The two fibres are nearly comparable in tensile strength though polyester fibre is available in higher tenacity grades.

Elongation is higher in polypropylene. This gives some advantages in terms of reduced tear during moulding.

Density of polypropylene (0,91 g/m³) is much lower than that of polyester (1,38 g/m³). As a result, thicker, loftier and more comfortable carpets are made with PP for a given area density.

Polypropylene is dope dyed and is available in an extensive range of colours and shades. It is therefore much easier to achieve colour and shade matching by mixing a minimum number of shades of fibres. Dope dyed polyester, on the other hand, is available only in a limited number of colours and shades. The required shade has often to be developed through research and development (R&D) work or fibre has to be dyed.

Melting point of PP (165°C) is much lower than PET (260°C). Heating time, temperature and pressing time are therefore more critical in moulding with PP.

Flame retardancy by burning rate is inferior with PP than with PET. A flame retardant compound has to be added to the binder to meet the flammability requirements in exports with PP. This adds to the cost.

Resistance to UV light is infeiror with PP compared to PET. UV stabiliser has to be added during manufacture of PP to improve its resistance to UV light.

Polypropylene tends to form beads during carding. The beads get deeply loaded on the cylinder wire and on the needles. There has to be a regular programme for removing the beads from the wire and needle. Achievable production rates are therefore lower with PP than PET because of card loading. Card processing problems are more acute with lower deniers and recyled fibres with polypropylene (Balasubramanian, 2003).

2.3 Manufacturing Process

Moulded carpet manufacturing can be broadly divided into 4 sections:

1. Needle punching

2. Back coating of needle-punched fleece with binder

3. Lamination, Blank cutting and Moulding

4. Trimming, checking and packing.

A typical flow chart for manufacturing moulded carpets is given in Fig. 2.4.

Sandwich mixing
↓
Opening Line
↓
Mixing Bin, Spraying Antistat Conditioning
↓
Carding
↓
Crosslaping
↓
Pre needling
↓
Finish Needling
↓
Structure Needling (optional)
↓
Backcoating
↓
Lamination with LDPE Powder/film
↓
Blank Cutting
↓
Moulding
↓
Trimming
↓
Inspection
↓
Heed pad welding
↓
Final Checking
↓
Packing
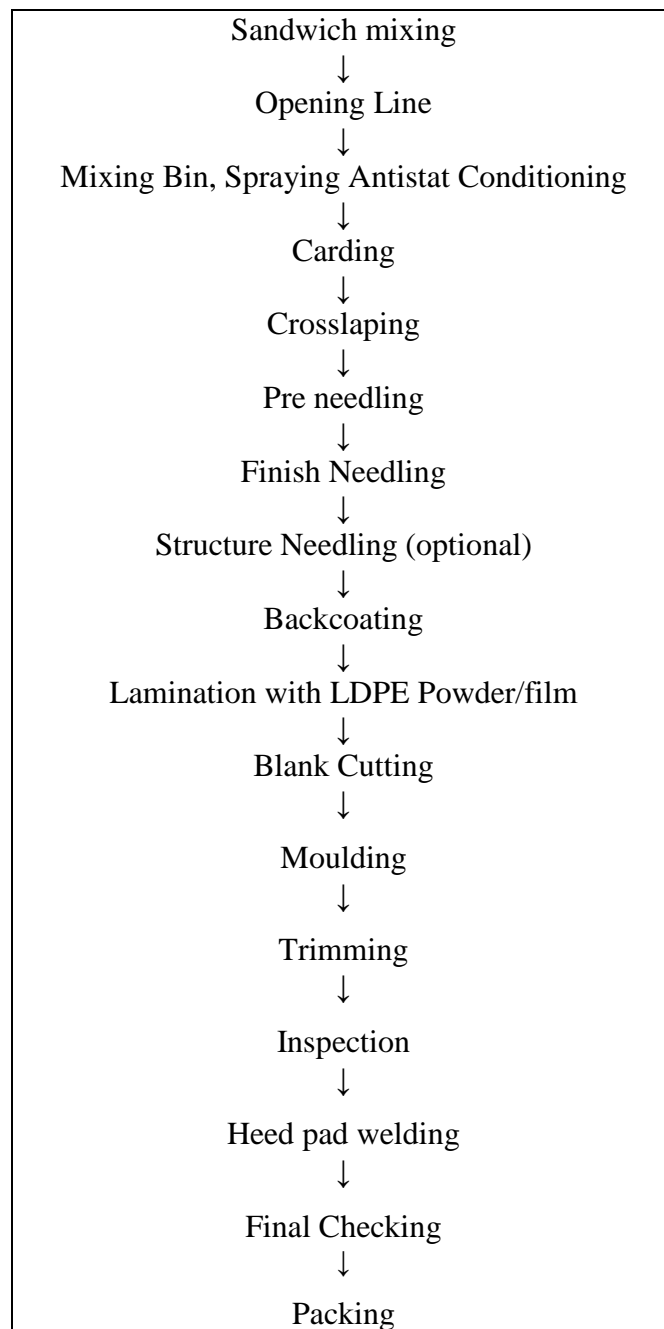
Figure 2.4: Flow chart for manufacturing moulded carpets.

The opening line consists of a bale opener followed by two beating points. Use of high capacity mixing bin with automatic lying of material and simultaneous spraying of antistat is helpful in ensuring intimacy of mixing and inminimising shade variations from place to place. Further performance in carding is improved because of uniform application of antistat. Feeding chute to card should have controls like photocells and vibrator or pressure switch to ensure uniform density of feed sheet. Incorporation of micro weighing system at the top of chute whereby small pre-weighed tufts are dropped into the chute will improve batt weight uniformity further. Card-Cross lap sequence is preferred to air laying because of long length of fibres used for making the felt. Since the fibre denier ranges from 6 to 15 denier, card should be mounted with appropriate wire of coarser gauge. The card room should have a good humidification system with some control over temperature. This will minimise loading of cylinder, doffer and stripping rollers especially with finer denier. Loading of rollers not only causes production loss but also is a major source of variations in linear density (gsm). Needle punching machine of 2 m. width would be adequate, as the length and width of the carpets of automobiles is below 2 m. The needle punching may be done in two stages: Pre needling line and a Finish needling line with a break in between. This has the advantage that if the gsm of the material is lower than the requirements, correction is possible in finish needling by the addition of a thin felt of required gsm. A continuous line with preneedling followed by finish needling does not provide such correction facility (Balasubramanian, 2003).

2.3.1 Pre needling

With die pressed needles with regular barb, spacing should be preferred as it minimises fibre damage and achieves uniform fibre interlocking. Further 3.5-inch needle should be used, as it allows wider gauge between stripper and stitching plate, which in turn minimises distortion of the voluminous batt as it enters the needle loom. 32-34 gauges with a punch density of 90-100 punches/cm² and depth of penetration of 11-13 mm depending on thickness of felt is normally used.

2.3.2 Finish needling

3-inch needle with close barb spacing has the merit that it allows all the barbs to enter the fleece with a relatively low depth of penetration. This improves consolidation and minimizes fuzzy surface at the bottom. The lower needle length is

also less susceptible to breakage. This needle however gives smoother surface. The penetration depth is between 10-12 mm with punch density of 150-250 punches/cm² in case of plain carpets. In the case of ribs and velours, a lower punch density of 90-100 punches/cm² is used to enable easy penetration of fork needles at structure needle machine.

2.3.3 Structure needling

In case of carpets with rib or velour surface, a structure needle loom is used after finish needling. According to the position of the fork in the needle board, rib or velour surface is obtained. For products with rib surface, penetration side of the fork needle will be reverse side of the finish-needled material, as it will give compact ribs. On the other hand, penetration side of fork needle will be the same side of finish needling in case of velour surface as this improves velour effect. Penetration depth is 7-8 mm in ribs and 8-10 mm in velours. The normally used fork needle is of 25 gauges.

2.3.4 Back coating

Back coating with a binder in the form of foam, followed by curing in a stenter gives dimensional stability and strength to the carpet. An auto foaming unit will be helpful as it permits automatic adjustment of foam ratio to get uniform pick-up. Acrylic binder is normally used as it gives the required strength. The binder pick-up is kept low as the material is going to be subsequently laminated with LDPE. Binder pick-up varies from 10-20 % for carpet where subsequent lamination is done. Foam ratio is one of the important characteristics to be monitored and kept under control during the process to minimise variations in binder pick- up, which ultimately leads to variations in linear density of carpet. Curing of binder is carried out in a stenter through heating by electrical heaters kept on both sides of the material. Temperature on the binder face is kept at 140ºC and on the fibre side at 110ºC. Material speed is adjusted as per the material and binder pick up and is usually between 2-3 m/min. To cut down processing time and costs, thermobondable binder is sometimes used. With such binder, there is no need for lamination with LDPE powder/film. Thermo bondable binder is special styrene/butadiene latex. The latex not only binds the fibres but also provides mouldability to the material. The binder film is thermoplastic with little softening in the region of 35-90ºC. On further heating, the softening increases

very rapidly in the region of 120ºC enabling moulding. The ratio of styrene to butadiene in the binder determines the stiffness and thermo plasticity of the binder.

2.3.5 Lamination

Carpets back coated with normal binder (acrylic) require lamination with LDPE powder or film or with both to enable moulding. The properties of LDPE powder have considerable influence on moulding. The average micron size of powder is 70 microns. For satisfactory moulding, the size of the granules should not be uniform throughout but should be variable from 30 to 100 with a peak value of 70. A distribution consisting of coarse and finer particles on either side of the average improves mouldability. The powder is dusted on the moving carpet on the back-coated side by an applicator, which is automatically controlled to achieve the required powder application. The material is then preheated in steps by electrical heaters to softening temperature and calendared by a pair of high-pressure calendars. Lamination of the powder on the carpet takes place as a result of softening and pressure application. With some products, a pre-heated film is also introduced into the nip of calendar rollers along with the carpet, which causes lamination of film on the carpet.

2.3.6 Blank cuttting

Laminated carpets are cut to required length on a blank cutting machine. The length requirements vary with the car and is standardised after initial trials. Lengths are marked on the table of bank cutting machine with different colours (for identification) for the different cars.

2.3.7 Moulding

The laminated and blank cut carpets are gripped on either side by grippers and moved to a pre-heating zone. The LDPE lamination is softened during preheating. After preheating for a specified time, the material is taken forward to a moulding press consisting of a male and female mould. The moulds are fabricated accurately as per the design of car body. Since car body is a patent of the car manufacturer, the moulds have to be prepared after a formal agreement with them to protect secrecy. Either moulds are made of aluminium or resin bonded fibreglass. The former though more expensive has a longer life and has a cooling arrangement, which reduces

moulding time and increases productivity. Time for preheating and pressing in moulding are critical factors affecting quality of moulding. They are determined by type of fibre, gsm of carpet, type of binder and lamination. Carpets made out of high proportion of polyester require in general more preheating than those with high proportion of polypropylene do. Heavier carpets require more time for pressing. Time for preheating and pressing are standardised for each type of car after initial trials. With some cars, which is a bulky material made out of waste fibres, is attached to the underside of the carpet either during moulding itself or subsequently by an adhesive. This improves insulation and helps to absorb vibrations.

2.3.8 Trimming

After moulding, the carpets are trimmed to shape on a trimming unit. The extra material at the sides used for gripping the carpet during moulding is cut out; holes are punched at different place to accommodate car parts like seat legs, break, clutch, and accelerator pedals. Trimming unit is made to match car body perfectly in minutes detail so that trimmed moulded carpets fit perfectly in the car.

2.3.9. Inspection

The moulded and trimmed carpets are checked for fitment on car body or a template provided by car manufacturer. Minor defects in the carpets are mended. Carpets with major defects and improper fitment are rejected. As majority of problems come from improper fitment, a great care has to be exercised in trimming and inspection. To reduce labour costs, some manufacturers get trimming and inspection done on contractual basis by an outside party.

2.3.10. Heel pad fixing

In some cases, heel pad is welded to carpet on a high frequency plastic welder. The object of heel pad is to prevent wear of carpet due to foot pressure from driver of car. Heel pad is usually of PVC material and car manufacturer gives specification.

2.3.11 Final checking

Final checking for fitment is done on car body. As this is time consuming, it will not be possible to have 100% checking. So random checking of 15-25% of production is

usually done. It would be advantageous to carry out checking some carpets on car body with seats in position (Balasubramanian, 2003).

2.4 Major Quality Problems

Some major quality problems in moulded carpets, which lead to rejection of the material and precautionary measures to overcome them, are discussed below.

2.4.1 Colour mismatch

Non-matching of colour of the carpet with the master approved sample and variations in colour and shade from place to place and lot to lot form a major source of rejection.

The following precautionary measures will help to overcome this problem.

1. Raw material (fibre) should be matched in colour against master sample in a colour cabinet. It is advisable to have two master samples representing the extremens in tolerable colour variation.
2. Intimacy of mixing of components can be improved by increasing the number of layers in the sandwich and by having one more toppling before processing.
3. Dope dyed fibre should be preffered to dyed fibre as better consistency in colour and higher fastness to rubbing and light is obtained with the former.

2.4.2 Contamination

Contamination with foreign material and fibres from previous lot arises because of inadequate cleaning of card wired, needle board, stripper, and stitching plate. Molten plastic beads and nep like clusters in needle board also result in contamination. Card wires should be washed with petrol once a shift and during lot change. Needle board should be removed and cleaned with compressed air. Fibre clusters firmly adhered to the barbs should be removed with a wire hook. Needle board cleaning should be done once every 4 hours and at the time of lot change.

2.4.3 Weight variations

High variations in weight (gsm) and thickness from place to place cause rejections. The variations in weight can be at needling stage or during back coating. Stop pages of card due to loading of material on cylinder, workers, strippers and transfer rollers

is one of the causes of variations in gsm. This problem is more often encountered with fine deniers and with polypropylene. Optimisation of antistatic spray applied on the fibre and maintenance of proper humidity and temperature in card room will help minimise loading. The card wire should be washed with petrol and damages in wire rectified. Lower doffer speed with higher web density will also be helpful. Control of foam ratio in the binder during back coating will also minimise variations in binder pick-up.

2.4.4 Lines in the fabric

Two types of defects are common:

1. Longitudinal lines running along machine direction of needle punching line arise from broken or worn out needles. A proper schedule for replacement of needle should be drawn up and adhered. Life of needle depends on type of needles, barb size, type of product, punch density, gsm etc. and it would be difficult to give a schedule for needle replacement that will hold good at all times.

2. Horizontal lines in cross direction of fabric at regular intervals arise because needles are penetrating in the same hole as fabric advances. This arises because net advance of fabric during a stroke matches with the distance between two adjacent rows of needles. The defect can be overcome by altering the slowly the stroke frequency while keeping fabric delivery speed constant.

2.4.5 Shape retention

Rejection due to deformation or non-retention of the shape of moulded carpet is another source of rejection. Moulded carpets laminated with LDPE film in general retain the shape much better than those laminated with LDPE powder do. Nevertheless, lamination with film alone is more difficult and wrinkles are encountered if the speed of film is not properly controlled or if the film is not properly rolled. Therefore, a good compromise is to laminate with both powder and film. Improper packing and transportation of carpets also leads to loss of shape. If a large number of carpets are stacked one over the other and transported, those at the bottom are deformed. Carpets should be laid on specially prepared rack with number of trays conforming to the shape of carpet. The carpets in different layers should be separated from another.

2.4.6 Improper fitment

Improper fitment is one of the major complaints. This is mostly due to improper trimming arising from wrong methods or improper tools. Use of preheated knife helps in getting sharp cuts. The tools used for cutting holes should be checked for wear, tear and sharpness. The car body used for checking carpets in moulding plant must match accurately in shape with the cars in automobile manufacturing unit.

2.4.7 Holes in carpet

Excessive preheating is one of the causes of holes particularly at corners. Excessive tensioning of material during moulding may also contribute to holes. Mould should be free from burrs, scratches or extraneous matter.

2.4.8 Wrinkles in carpet

Wrinkles are caused because of inadequate or excessive preheating or inadequate tensioning of material during moulding. Preheating time has to be optimised for different types of fibres and area density of carpet (Balasubramanian, 2003).

2.5 Product Test Criteria

The common expectancy of the car user is that the auto-interior should have good appearance and aesthetics, good comfort, easy to maintain, retention of good properties even after prolonged usage, good durability, wrinkle resistance, water and stain proof, having antistatic and oil release property, no or minimum emission, which may hinder driving by fogging or contamination of inside atmosphere, flame resistant for safety and low costs.

The main criteria involved in the development of textiles and components in automotive especially in carpets are tensile strength, abrasion resistance, pill resistance, compression resistance, elasticity, light fastness at high temperatures, stiffness, ease of cleaning, separation force, dimensional stability, flame resistance, anti-fogging resistant, resistance to adverse climatic conditions. The others processing requirements are mouldability, susceptibility to compression, sewability, weldability, adhesive properties, vulcanizing properties (Singha, 2012).

Specifications and quality requirements vary from one automobile manufacturer to another. An example of FORD spesicifation table and FORD test methods are given in Table 2.5 and Table 2.6.

Table 2.5 : Spesifications for Ford Automobile Carpets.

Material Data Sheet

Material Information

Material	PES + SBR +PE + HARDLAYER
Color	Black
Weight	1450 ± %10 gr/m² (650 pes + 150 latex + 150 pe powder + 500 hardlayer)
Dimensions	Fabric Width: 1600 mm

List of Periodic Test

Test Name	Criteria For Admission	Spesification No	Period
Appearance	Very hairy and, folding, wrinkling, discoloration, needle marks, snagging	Final Inspection Report	Each Lot
Weight	1450±%10 gr/m²	Final Inspection Report	Each Lot
Dimensions	Width: 1600 mm	Final Inspection Report	Each Lot
Tensile Strength	Width Min: 400 N	Final Inspection Report	Each Lot
Tensile Strength	Longitudinal Min: 400 N	Final Inspection Report ASTM D 5034	Each Lot
Burn Speed	Max: 100 mm/min.	Final Inspection Report	Each Lot
Taber Abrasion Resistance	Should not be extremely Abraded	Final Inspection Report ASTM D 5034	Each Lot
Pubescence Test	Min: Snagging	Final Inspection Report ASTM D 5034	Each Lot

As seen from the Table 2.5, the most significant specifications for Ford automobile carpets are appreance properties, weight, dimensions, tensile strength, flammability, abrasion resistance and snagging.

Apart from those specified in Table 2.5, specifications for temperature stability, static compressibility, peeling force, fogging resistance, dielectric weldability, and freedom from odour are prescribed by some other manufacturers.

Table 2.6 : Summary of test methods applied to FORD automotive carpets (Ford WWS M8P16B‑Engineering Material Specification).

Specification No	Test Definition	Required Value / Tolerance
WSS M8P16B / ASTM D 5034	Breaking Strength (ASTM D 5034 Grab Method, 305 mm/min machine speed	Widthwise: Minimum 400N Longitudinal: Minimum 400 N
WSS M8P16B / ASTM D 5587	Tear Strength (Dry Only ASTM D 5587, Option 1 Multiple Peak Force Average	Widthwise: Minimum 90N Longitudinal:: Minimum 90 N
WSS M8P16B / FLTM BN 105-01	Shrinkage Water	Maximum 2%
WSS M8P16B / FLTM BN 105-01	Shrinkage Heat 80°C x 7 days	Maximum 2%
WSS M8P16B / FLTM BN 105-03	Dimensional Stability- 2 cycles	Maximum 2%
WSS M8P16B	Heat Ageing	Checking to color shades Minimum rating should be 4
WSS M8P16B	Resistance to Mildrew	Should be no visible mildrew and no rotting
WSS M8P16B / SAE J 1530-A	Resistance to Abrasion (SAE J 1530-A, 1000 g loading, H-18 wheels	Should be better than limiting sample
WSS M8P16B / SAE J 1530-A	Resistance to Snagging	Should be better than limiting sample
WSS M8P16B	Environmental Cycling	There should be no discoloration, splitting of lamination, and rotting.
WSS M8P16B	Cold Resistance (For Heavylayer Coated Carpets)	Should be no cracking and peeling of lamination
WSS M8P16B / FLTM BO 116-01	Resistance to Fade (FLTM BO 116-01, ISO 105 A-02/AATCC Assessment Procedure 1)	Should be no change in color and shade
WSS M8P16B / SAE J 1530-A	Fiber Deterioration After UV Exprosure	Should be equal to referance sample
WSS M8P16B / FLTM BN 107-01	Dry-Resistance to Crocking	Minimum Rating 4
WSS M8P16B / FLTM BN 107-01	Wet-Resistance to Crocking	Minimum Rating 4
WSS M8P16B / FLTM BN 112-08	Soiling and Cleanability	Minimum Rating 3
WSS M8P16B / SAE J 1756	Fogging	Minimum 70%
WSS M8P16B / FLTM BO 131-03	Odor	Maximum Rating 3
WSS M8P16B / SAE J 369	Flammability	Maximum 100 mm/min.

3. USE OF RECYLED POLY(ETHYLENE TEREPHTALATE) FIBERS IN NEEDLEPUNCHED AUTOMOBILE CARPETS

It is estimated that around 4 % of the world's oil and gas production is used for production of plastic and another 3 % - 4 % is used in energy for their production. In addition, most of these materials are being discarded within a year. Thus using plastics has not been a sustainable option and recycling is more important than before especially keeping in mind the need to preserve and protect the environment (Hopewell, Dvorak and Kosior, 2009).

Currently, the efficient use of resources is not just a cost-optimizing alternative but also an urgent necessity in the automotive industry. The framework of waste management set by the EU in 2008, endorses waste-preventation, re-use and recycling as the preferred methods of waste disposal. Under such conditions, the utilization of the recycled material is essential to complete the material cycle (Neuenhofer, 2011).

Furthermore, automotive manufacturers emphasize the use of efficient sound-absorptive materials in vehicles to reduce undesired noise. The existing sound-absorptive materials are generally made of synthetic fibers, which are not biodegradable (Parikh, Chen, Sun, 2006). At the end of vehicle's life, most of auto interior materials usually end up in landfills. As the cost of landfill is escalating, recycling efforts have been increasing. Most of the major OEMs are also making efforts to increase the recycled material content of their cars and certain OEMs are making recycling a key design consideration, specifying that parts supplied must be made from a certain percentage of post-consumer recycled material. From press reports, Fords appear to be especially energetic in this exercise and are putting pressure on their suppliers. Eventually Fords want to attain 90% vehicle recyclability (by weight), whereas others such as Daimler Chrysler want to do even better 95%.

In this case, the use of recycled materials in nonwovens for automotive applications is also increasing. Because, nonwovens offer various possibilities to include recycled fiber material (often blended with virgin fibers) in the production of new webs for

automotive applications (Neuenhofer, 2011). A growing proportion of needlepunched nonwovens for automotive applications, for example, is being manufactured from recycled polyester derived from plastic bottles (Edana Newletter, 2013).

Some polyester fibre manufacturers, Hoechst and EMS have demonstrated the possibility of running recycled polyester face fabric into nonwoven material.

Nonwoven polyester spun from recycled polyester bottles by Wellman, which is Europe's largest PET recycler, is currently being used in some production models. Bottle manufacturers continue to develop and improve polyester bottles for many other end-uses and there could soon be a surplus of polyester bottles available for recycling. However, 'closed loop' recycling is generally recognised as the most satisfactory recycling procedure. This is when the recycled material, in the present context, automotive interior trim fabric is recycled back into the original end-use, i.e. back into an automotive textile.

Because of the recyclability of PET and the relative abundance of post-consumer waste in the form of bottles, PET is rapidly gaining market share as a carpet fiber. Mohawk Industries released ever STRAND in 1999, a 100% post-consumer recycled content PET fiber. Since that time, more than 17 billion bottles have been recycled into carpet fiber (Nikolov, Lebensohn, Raabe, 2006). Pharr Yarns, a supplier to numerous carpet manufacturers including Looptex, Dobbs Mills, and Berkshire Flooring (Papaspyrides, Vouyiouka, 2009), produces a BCF (bulk continuous filament) PET carpet fiber containing a minimum of 25% post-consumer recycled content.

Gurudatt *et al.* (2005), on their study, compared fibers produced by recycling PET bottle wastes with those obtained by recycling polyester fiber wastes and virgin fiber grade polyesters melt spun under identical conditions. It has been demonstrated that bottle wastes yield fibers having properties equivalent to those of virgin polyester but probably cost similar as fiber wastes. Molded automotive carpets developed from recycled fibers from PET bottle wastes have shown excellent mechanical and fastness properties. It is inferred that using dope-dyed fibers based on PET bottle wastes in automotive applications may ensure a high benefit to cost ratio. In that study, the automotive carpets were subjected to various performance tests. Some of

the properties of moulded automotive carpets produced from each of the fibers spun from recycling PET fiber waste (FG waste fiber), virgin PET chips (Virgin FG fiber) and recycling PET bottle waste (BG waste fiber) are presented in Figure 3.1.

Property	FG waste fiber	Virgin FG fiber	BG Waste fiber
Fiber properties			
Denier	11.38	11.81	11.75
Staple length (mm)	64	64	64
Weight (g/m^2)	1048	1060	1026
Thickness (mm)	4.4	4.2	4.8
Breaking strength (kgf)			
MD	41.5	75.8	73.6
CD	56.3	59.1	64.4
Aged tensile test on UV exposure (488.8 kJ/m^2) (% loss)			
MD	5.44	2.21	2.72
CD	4.86	1.44	1.65
Trapezoid tear strength (kgf)			
MD	22.2	18.41	29.3
CD	15.1	21.76	31.8
Taber abrasion resistance (after 1000 rubs with calibrate wheel CS −10)			
Change in shade	3–4	4	4–5
Mass loss %	5.2	1.5	1.7
Color fastness to rubbing			
Dry rating	4–5	5	5
Wet rating	4–5	5	5
Color fastness to light	6	8	8
Flammability 15-s ignition (cm/min)	26.4	29.2	21.4

Figure 3.1 : Comparasion of properties of moulded automotive carpet incorporating fibers spun from different kinds of polyesters (Gurudatt *et al*. 2005).

From the results obtained by this study, it is seen that recycling PET bottle waste fibers have properties comparable to those of virgin fibers and can find ready usage in applications like automobile carpets. Their use in automotive applications ensures high benefit to cost ratio because of lower raw material costs.

On another study, Parikh *et al* (2006) developed auto floor coverings as carded needlepunched nonwovens using naural fibers (kenaf, jute, waste cotton, and flax) in blends with polypropylene (PP) and polyester (PET) to reduce automotive interior noise. The accoustical absorption coeffients of these floor coverings and of floor coverings in combination with an underpad (either rebonded polyurethane foam, or a soft cotton nonwoven) were evaulated in the frequency range of 100 to 3200 Hz. The measurements demonstrated that each of the natural fiber-based nonwoven floor coverings contributed to noise control.

On another study by Abdelfattah *et al*. (2011), produced nonwoven fabric that can be used in car interior components (headliners, door panels and trunk liners) to prevent noise from reaching the passenger compartment and so to achieve comfort in the car interior by using 6 denier polyester and hollow polyester fibers. Three different fabrics of 100 % polyester fibers, 75 % polyester/ 25 % hollow polyester fibers and 55% polyester / 45% hollow polyester fibers were produced with different weights as 300, 400, 500 and 600 g/m². Samples produced with high percentage of hollow fibers recorded the highest rates of sound absorption; whereas samples produced with 100 % polyester fibers recorded the lowest rates. Sample produced with 55 % polyester / 45 % hollow polyester fibers and 600 g/m² achieved the best result.

3.1 Virgin PET and Recycled PET

Polymers are among the largest used materials today in the world. PET has a significant market share among all the other polymers. More than 90% of plastic bottles made in the world are from PET (NAPCOR, 2012 Report on Post Consumer PET Container Recycling Activity). With this huge amount of material being used, the impact on the environment in the form of increasing landfills and carbon dioxide emissions has also been high. Hence, the need to recycle PET and reuse it has been a topic of interest over the last few years. However, loss in properties of recycled PET has been a concern and it is still considered secondary to virgin PET.

3.1.1 Poly (ethylene Terephthalate)

With good thermal, physical, mechanical properties, and comparatively low cost, Poly(ethylene terephthalate) (PET) has been among the most widely used polymer materials. It is estimated that PET has an 8% share in the world market and more than 90% of plastic bottles are made from PET (CMAI, 2012- SBA CCI, 2011).

PET finds major applications as material for fibers; injection molded parts, and most importantly for blow molded bottles. PET is made from Terephthalatic Acid and Ethylene Glycol through the poly-condensation reaction of the monomers. PET production depends upon the chemical reaction between the monomers, which are produced by esterification reactions.
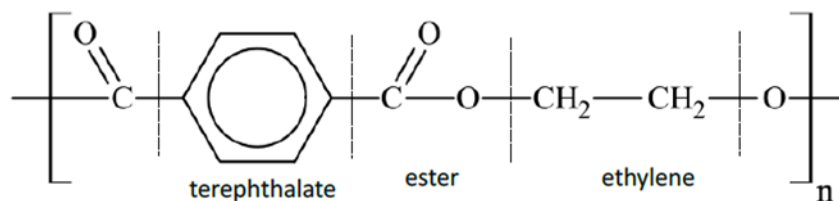
Figure 3.2 : Poly(ethylene Terephthalate) molecular structure.

The PET monomer contains two ester groups, one ethylene group and one terephthalate group as shown in Figure 3.2. PET is a linear thermoplastic that can be melted and molded on heating, which makes it easier to reuse PET. Thermoset plastics on the other hand do not soften; they will break down at high temperatures. Thermoplastics have weak intermolecular forces whereas the thermosets have strong crosslinking between its bonds. Thermoset plastics are usually harder and more brittle (Bolton, 2012).

The production of PET involves the esterification stage or trans-esterification stage where the raw materials Terephthalic Acid and Ethylene Glycol (esterification), and Dimethyl Terephthalate and Ethylene Glycol (trans-esterification) combine for the pre-polymerization reaction. This is followed by the melt condensation, which will provide a lower Intrinsic Viscosity (I.V.) and low molecular weight resin. This process is followed by Solid State Polymerization (SSP), which is a very important technique of increasing the molecular weight and the intrinsic viscosity of the PET pellets before they are used for injection molding (Ravindranath, Mashelkar, 1986 and Bandla, 2010). It is very important to know the processing conditions for the polymers since they are highly sensitive to the molecular structure and macroscopic mechanical properties.

The chemical composition and molecular structure plays an important role in determining the properties of PET and its copolymers. PET is generally considered as a strong, tough, flexible thermoplastic, which will crystallize and can be oriented into different molecular arrangements. Some common properties which favor usage of PET are its strength, temperature tolerance, wear resistance, toughness, low water absorbance, light weight, transparency i.e. clear amorphous state on cooling, wear resistance, corrosion resistance, design flexibility, chemical resistance, recyclability and long shelf life (Brooks, Giles, 2002 and Kenplas, 2012). PET after stretching still has properties of having a proper orientation, high stiffness, creep resistance, clarity, water vapor barrier, chemical and impact resistance. Several additives are mixed with

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PET to improve various properties before they are processed in manufacturing. While reducing the overall cost, fillers and modifiers are additives, which improve the mechanical properties of polymers (Tarr, 2011).

Among these properties, the molecular weight and the Intrinsic Viscosity (I.V.) are the two most common indicators, which will determine its end use. PET is made available in semi-crystalline pellet form to avoid sticking together during the drying process. In general, PET is classified based on its Intrinsic Viscosity (I.V.). The empirical expression, which relates intrinsic viscosity to the molecular weight, is given by Equation **3.1**, which is known as the Mark Houwink equation.

$$[\eta]=K*M\alpha \tag{3.1}$$

Where η is the intrinsic viscosity (I.V.), M is the molecular weight, and K and 'α' are constants. The values of K and 'α' are determined based on different relationships published in literature. The intrinsic viscosity (I.V.) for PET usually ranges between 0.75 dL/g to 1.00 dL/g and the molecular weight is between 24000 g/mol to 36000 g/mol (Brooks, Giles 2002, Bandla 2010). The end group is another important parameter, which affects PET properties.

To use PET for industrial applications, it is necessary for them to have good properties even after the processing stages. Usually for bottling applications; Intrinsic Viscosity (I.V.) levels in the 0.72 dL/g -0.76 dL/g range are a required minimum. Similarly, strapping requires around 0.74 dL/g I.V. and sheet applications require 0.7 dL/g I.V (Markarian, 2009). Intrinsic viscosity range is given on Table 3.1

Table 3.1 : The intrinsic viscosity range of PET.

Intrinsic Viscosity Range of PET
Fiber grade
0.40 – 0.70 Textile
0.72 – 0.98 Technical, tire cord
Bottle grade
0.70 – 0.78 Water bottles (flat)
0.78 – 0.85 Carbonated soft drink grade
Monofilament, engineering plastic
1.00 – 2.00

Determining thermal properties such as glass transition temperature, crystallization and melting temperatures of PET will help tailor the processing methods as per the end use. Thermal properties are usually determined by Differential Scanning Calorimetery (DSC) experiments. A typical semi crystalline PET pellet is expected to have a glass transition temperature of 80˚C, a crystallization temperature of around 160 ˚C and melting temperature around 250˚C (Brooks, Giles 2002).

The crystallinity of PET is an important property to measure, as it influences the mechanical properties (Sichina, 2000). The initial crystallinity refers to the crystallinity level before the processing and the final crystallinity is the crystallinity level in the final product. Orientation of the molecules plays a role in affecting the final properties along with crystallinity. Tensile modulus and yield strength are directly related to the orientation and crystallinity in PET. (Viana *et al*, 2004, Oultache *et al*, 2001). Typical PET has 50% crystallinity. As the degree of fiber stretch is increased (yielding higher crystallinity and molecular orientation), so are properties such as tensile strength and initial Young's modulus. At the same time, ultimate extensibility, i.e., elongation is usually reduced. An increase of molecular weight further increases the tensile properties, modulus, and elongation. Typical physical and mechanical properties of PET fibers are given in Table 3.2.

Table 3.2 : Physical Properties of Polyester Fibers (Hearle *et al*, 1995).

Property	Filament yarn		Staple and tow	
	Regular tenacity[a]	High tenacity[b]	Regular tenacity[c]	High tenacity[d]
breaking tenacity,[e] N/tex	0.35-0.5	0.62-0.85	0.35-0.47	0.48-0.61
breaking elongation	24-50	10-20	35-60	17-40
elastic recovery at 5% elongation, %	88-93	90	75-85	75-85
initial modulus, N/tex[f]	6.6-8.8	10.2-10.6	2.2-3.5	4.0-4.9
specific gravity	1.38	1.39	1.38	1.38
Moisture regain, %	0.4	0.4	0.4	0.4
Melting temperature, ^{o}C	258-263	258-263	258-263	258-263

[a]Textile-filament yarns for woven and knit fabric. [b]Tire cord and high strength, high modulus industrial yarns.
[c]Regular staple for 100% polyester fabrics, carpet yarn, fiberfill, and blends with cellulosic blends or wool.
[d]High strength, high modulus staple for industrial applications, sewing thread, and cellulosic blends.
[e]Standard measurements are conducted in air at 65% rh and 22°C.[f]To convert N/text to ge/den, multiply by 11.33.[g]The equilibrim moisture content of the fibers at 21°C and 65% rh.
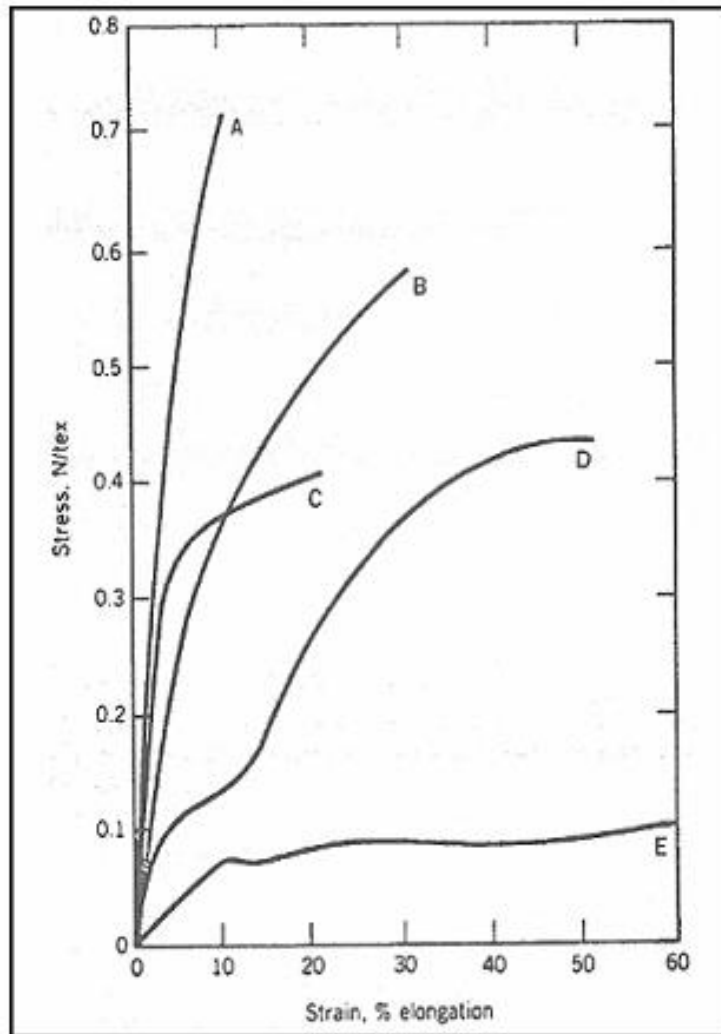
Figure: 3.3 : Typical stress strain curve for PET fibers.
A-High tenacity filament, B-High tenacity staple,
C-Regular tenacity filament, D-Regular tenacity staple,
E- POY filament.

In addition, stress-strain curves are given in Fig. 3.3. It can be seen that the filament represented by curve C has a much higher initial modulus than the regular tenacity staple shown in curve D. On the other hand, the latter exhibits a greater tenacity and elongation. High tenacity filament and staple (curve A and B) have very high breaking strengths and moduli, but relatively low elongations. Partially oriented yarn (POY) and spun filament yarns, exhibit low strength but very high elongation (curve E). When exposing PET fiber to repeated compression (for example, repeated bending), so-called kink bands start to form, finally resulting in breakage of the kink band into a crack. It has been shown in (Hearle *et al*, 1995) that the compressibility stability of PET is superior to that of nylons.

3.1.2 Recycled PET and recycling process

The semicrystalline nature of PET allows one to generate a wide variety of both physical and mechanical properties well suited for the fabrication of fibers, films, bottles and different molded parts. These parts are converted into finished products such as garments, carpets, packages and industrial goods.

The most widely used application for PET is the manufacture of bottles. The need to reduce landfill consumption rates and drives to cut carbon emissions has brought about increased demand to recycle and reuse PET. Together with the increase of virgin (V) PET, the use of recycled post consumer PET (RPET) flakes and resin for production of RPET products has continually increased.

PET is a thermoplastic, meaning that it softens when heated and hardens again when it is cooled and hence it can be reused easily. PET is also among the most recycled polymers in the world. The first ever attempt at recycling PET was done in 1977. Over the years, there has been an increase in PET recycling rates. In 2010, more than 1.5 billion pounds of PET bottles and containers were recycled.

Recycled PET (RPET) is used for applications in several areas like fiber for carpets, jackets, fillers, bottles, containers, films, sheets and strappings. Of the 1002 millions of pounds (MMlbs) RPET used in 2010, 70 % was used for fiber, sheet and film applications while 21% was used for food and beverage bottle applications, which is shown Figure 3.4 (NAPCOR, 2012 Rate Report).

Product Category	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011	2012
Fiber	344	296	479	463	422	383	391	344	381	398	512
Sheet & Film	18	32	58	71	74	128	153	159	195	202	307
Strapping	83	77	116	131	132	144	137	114	127	120	136
Engineered Resin	10	10	12	8	9	11	7	10	9	See Other	See Other
Food & Beverage Bottles	86	106	126	115	139	136	141	203	216	242	276
Non-Food Bottles	43	24	63	63	49	60	55	65	58	57	50
Other	4	7	24	13	30	38	31	42	16	21	31
TOTAL CONVERTER CONSUMPTION	588	552	878	864	855	900	915	937	1,002	1,040	1,312

Figure 3.4 : RPET product categories (RPET used *MMlbbs*)
(http://www.napcor.com/pdf/NAPCOR_2012RateReport.pdf)

Worldwide, approximately 7.5 million tons of PET was collected in 2011. This gave 5.9 million tons of flakes. In 2009, 3.4 million tons were used to produce fibre, 500,000 tons to produce bottles, 500,000 tons to produce APET sheet for

thermoforming, 200,000 tons to produce strapping tape and 100,000 tons for miscellaneous applications (www.pcipetpackaging.co.uk)

Petcore, the European trade association that fosters the collection and recycling of PET, reported that in Europe alone, 1.6 million tonnes of PET bottles were collected in 2011 - more than 51% of all bottles. After exported bales were taken into account, 1.12 million tons of PET flakes were produced. 440,000 tons were used to produce fibres, 283,000 tons to produce more bottles, 278,000 tons to produce APET sheets, 102,000 tons for strapping tape and 18,000 tons for miscellaneous applications. (Source: PCI for Petcore and EuPR)

In 2012, 81% of the PET bottles sold in Switzerland were recycled.

Figure 3.5 shows the PET bottle recollection rates in the United States, the European Union, Brazil and Japan. The European Union has the highest recycling efficiency. There are several factors, which affect the PET recycling rate; these include the market demand, cheaper technologies, value addition and overall cost. However, the process of bottle-to-bottle PET recycling may not be as easy as it seems since there is a need to ensure that the rPET has properties that are on par with virgin PET and reduce contamination in PET (Welle, 2011).
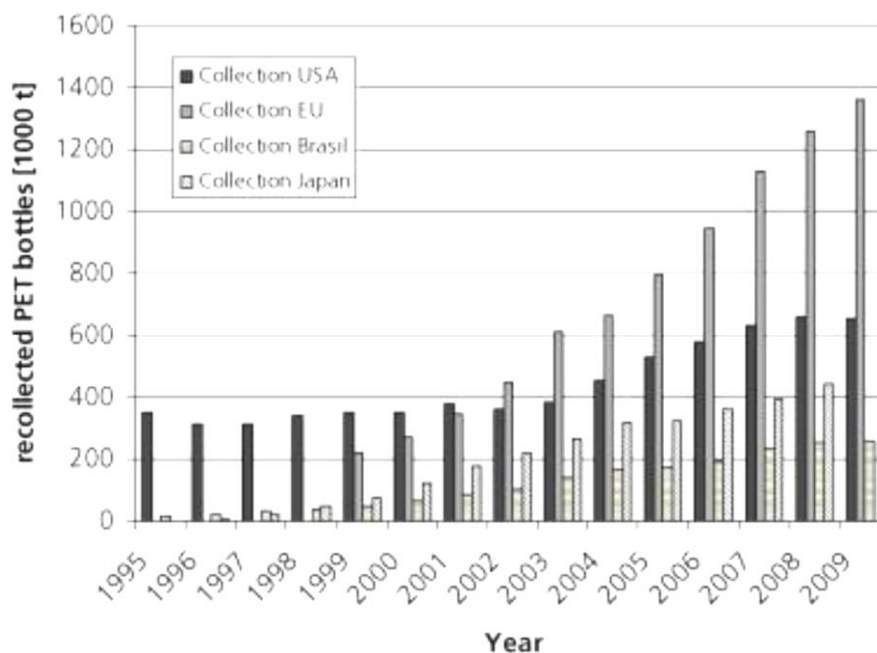
Figure 3.5 : PET bottles recollection statistics for United State of America (USA), European Union (EU), Brazil and Japan (Welle, 2011).

With tones of PET being produced and used in the world every day, it is very important to recycle PET. However, some factors influence the use of recycled PET (PPET) in the market the same manner virgin PET has been used. PET is a very valuable raw material (Market price of up to $1 /lb in 2011) and finding newer applications most importantly replacing traditional raw materials, metals and resins. RPET has a positive impact on the environment and reduces the impact on landfills, consumer waste, reduces emissions and helps in energy conservation. From a study by NgPlastics Corp. (Chilton et al, 2010) it was found that, recycling PET reduced energy consumptions by 52.6 % and the recycling methods reduced carbon dioxide emissions by almost 54 %.

The environmental benefits of Recycled PET are phenomenal. According to the Environmental Protection Agency (EPA), 2/3 less energy is required to manufacture products made out of recyclable plastic. Other studies show that the production of recycled plastic requires 2/3 less sulphur dioxide, 50% less nitrous oxide, and almost 90% less water usage (http://www.ecozuri.com/green-knowledge/recycled-pet.html).

PET, as with many plastics, is also an excellent candidate for thermal disposal (incineration), as it is composed of carbon, hydrogen, and oxygen, with only trace amounts of catalyst elements (but no sulfur). PET has the energy content of soft coal.

When recycling polyethylene terephthalate, PET, or polyester, in general two ways have to be differentiated:

1. The chemical recycling back to the initial raw materials purified terephthalic acid (PTA) or dimethyl terephthalate (DMT) and ethylene glycol (EG) where the polymer structure is destroyed completely, or in process intermediates like bis-ß-hydroxyterephthalate (Figure 3.6). This process of recycling PET is very costly, hence it is not considered as a viable option.

2. The mechanical recycling where the original polymer properties are being maintained or reconstituted. Mechanical recycling is the most widely used method since its easier, faster and much more economical compared to chemical recycling (http://en.wikipedia.org/wiki/Polyethylene_terephthalate)

Figure 3.6 : PET recycling in the factory.

The mechanical recycling process has several stages. Figure 3.4 shows recycling process in the factory and Figure 3.7 gives an outline of the PET recycling process.
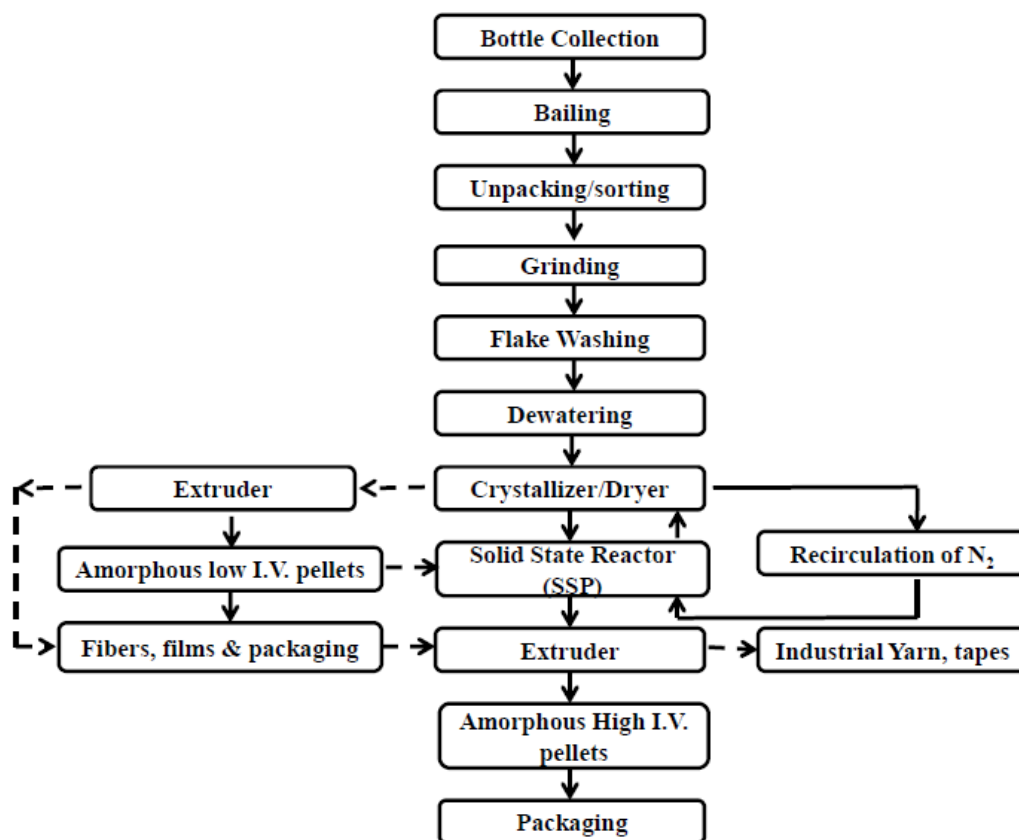
Figure 3.7 : Mechanical PET Recycling Process (BePET Recycling Technology for rPET Report, 2011).

Mechanical process is most common way of PET recycling. In this method, bottles and other PET base material must be washed and grinded. The resulting flakes blend with virgin PET in various percent according to future applications (Khoramnejadian, 2011).

According to the Association of Postconsumer Plastic Recyclers (APR), approximately 95% of RPET is mechanically recycled in the U.S. In general, mechanical recycling of RPET is performed by collecting scraps from homogeneous deposits as carbonated and non-carbonated drink bottles, and from heterogeneous deposits contaminated with polyvinyl chloride (PVC), nylon and various additives (Kang *et al*, 2011).

During recycling processing steps, the PET flakes or pellets are subjected to different temperatures and mechanical processing, which causes contamination, degradation and loss of properties. The most common acid contaminants found in RPET are acetic acid form poly vinyl acetate (PVA), rosin acid, abietic acid from adhesives, and hydrochloric acid from poly vinyl chloride (PVC). These acids usually end up and hydrochloric acid from poly vinyl chloride (PVC) (Awaja *et al*, 2005). These acids usually end up causing chain scission reactions during the melt processing stage (Scheirs, 1998, Cardi *et al*, 1993 and Paci *et al*, 1998). Water is another important contaminant and this causes reduction in the molecular weight via hydrolysis reactions. Coloring contaminations include those fragments from colored bottles or from labels (Awaja *et al*, 2005). Acetaldehyde is another contaminant, which is a by-product of PET degradation reactions (Villian *et al*, 1995). There may also be traces of pesticides, air borne contaminations, sand, fuel and detergents owing to use of PET in various environments.

2.4 Comparasion of VPET/RPET Properties and Literature Review

There are some concerns about using reycyled PET along the same lines as virgin PET during the production of a needlepunched automobile carpet. The point is whether RPET has properties on par with virgin PET. It is very important to study the properties of RPET that would be relevant to its processing. Moreover, the mechanical behavior of RPET would need to be studied in order to come to a strong conclusion that RPET can indeed be considered for automotive carpet applications and also assert that its properties are similar to virgin PET or maybe in some cases even better.

There have been several studies on literature depending on the reusing of PET in the different products and comparison of virgin PET to Recycled PET according to their mechanical and dynamic properties.

Galeski *et al* (2000) listed out characterization techniques that could be used for scrap poly(ethylene terephthalate) and recycled polymers in general. They characterized samples using several methods and suggested that using Thermogravimetric analysis (TGA), Differential Scanning Calorimetry (DSC), Fourier Transform Infrared Spectroscopy (FTIR), tensile testing, and rheometry to determine intrinsic viscosity and molecular weight were efficient methods to characterize samples. They have also concluded that if more than 50 ppm of contaminants like PVC were found in PET, it would make it worthless for advanced applications like film forming since it catalyzes the hydrolysis reactions and and reduces the strength of the material.

Pattabiraman *et al*, on their study (2005) discussed the thermal and mechanical properties of virgin PET, recycled PET and their blends. They used Differential Scanning Calorimetry (DSC) and thermogravimetric analysis (TGA) to study the thermal properties. The tensile tests at ambient and elevated temperature were used to study the mechanical properties. The test results obtained are shown in Figures 3.8, 3.9, 3.10 and 3.11.

Recycled content of PET in %	Crystallinity in %	Melting temperature, °C
100% virgin PET (BK3180)	33.01	250.0
90% virgin PET/10% RPET	30.84	250.0
80% virgin PET/20% RPET	27.21	249.8
70% virgin PET/30% RPET	32.03	249.9
50% virgin PET/50% RPET	32.04	249.7
100% RPET (food grade)	34.03	251.2
100% RPET (fibre grade)	36.39	251.6

Figure 3.8 : Degree of crystallinity of PET blends (Pattabiraman, 2005).

According to results obtained, it was determined that there were significant differences in the recrystallization behaviour as far as the thermal properties were concerned.
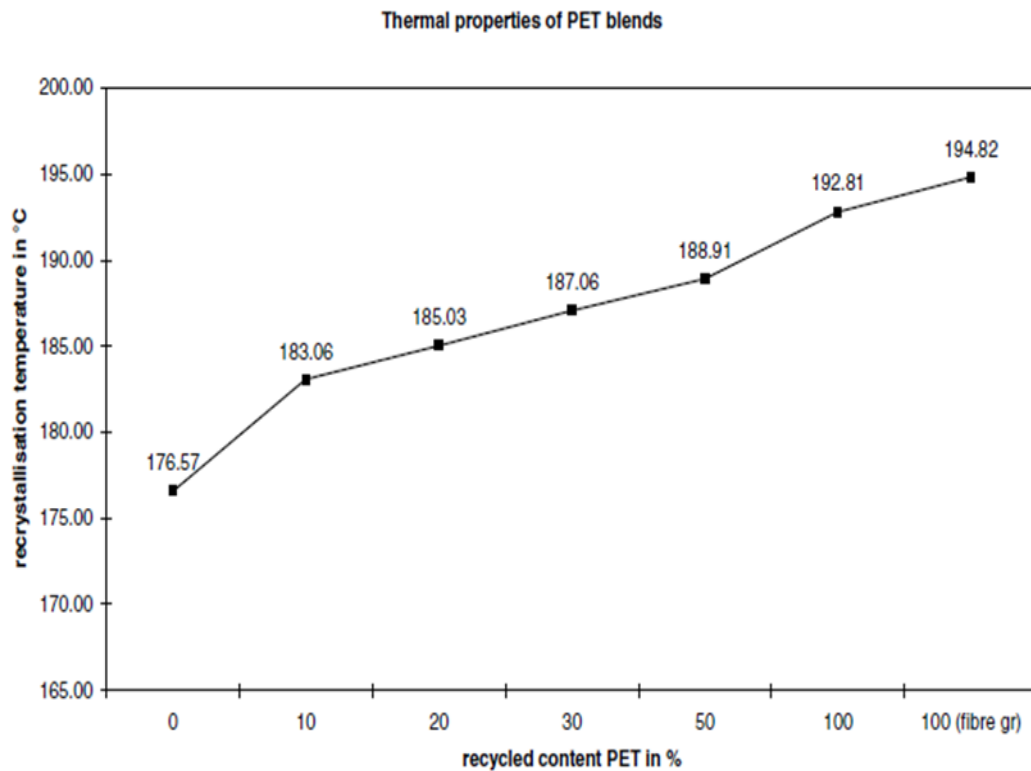
Figure 3.9 : Recrystallisation temperature of PET blends (Pattabiraman, 2005).
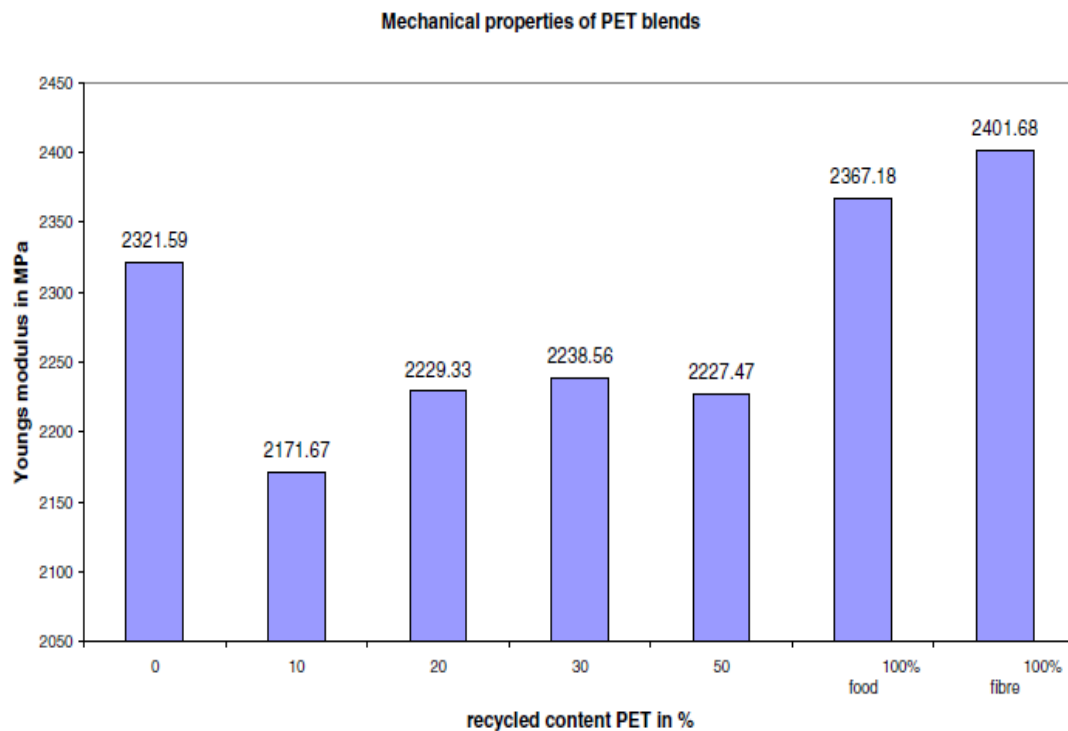
Figure 3.10 : Young modulus of PET blends at ambient temperature
(Pattabiraman, 2005).

Figure 3.11 : Mechanical properties of PET blends at 110°C (Pattabiraman, 2005).

The tensile tests elevated temperature showed that the strength of the blends of recycled PET/virgin PET were lower than those ones of virgin PET.

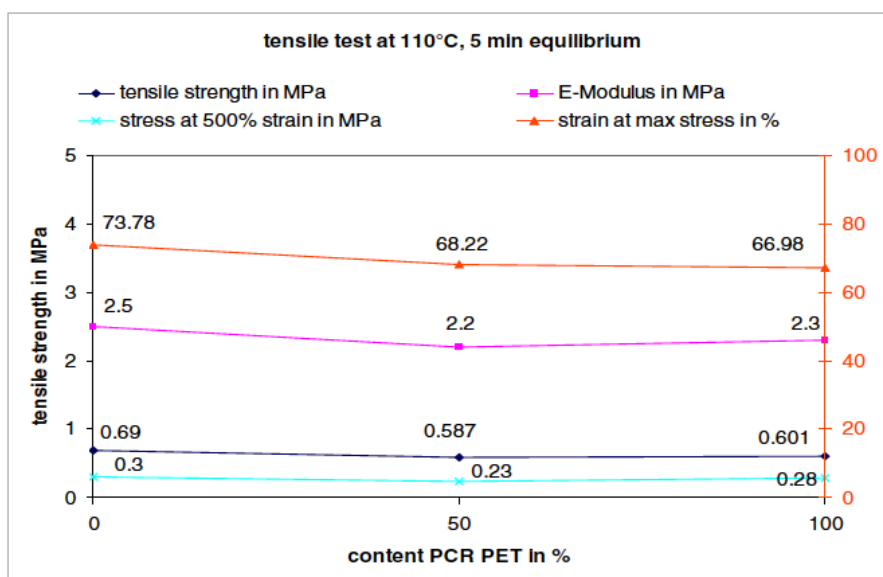At elevated temperature, the tensile strength and modulus of elasticity of virgin PET are higher than the recycled PET and their blends. This showed that the mechanical properties of the blends of virgin/recycled PET are worse than those of the virgin ones.

In another study by Torres *et al* (1999), it was compared the thermal properties (glass transition, melting point and crystallinity) and mechanical properties (Young's Modulus, elongation at break and impact strength) of post consumer poly(ethylene terephthalate) (PET) bottles with those of virgin ones. They studied two types of scraps of recycled PET: one arising from homogeneous deposits of bottles and the other of heterogeneous deposits soiled by contaminants such as PVC and adhesives. The presence of contaminants and residual moisture coming in the shape of scraps facilitate the crystallization of recycled PET compared to virgin PET and induces cleavages of chains during the melt processing. This leads to a reduction in intrinsic viscosity and consequently in molecular weight, and these decreases are more significant when the recycled resin is soiled. The study showed that virgin PET exhibited a ductile behaviour (>200 % of elongation at break), whereas post-consumer PET bottles exhibited a brittle one (<10 % of elongation at break). This is a consequence of the difference in crystallinity, the presence of impurities in the

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recycled PET and the different thermal and mechanical history of the virgin and recycled materials. The obtained t est results are given on Figure 3.12, 3.13 and 3.14.

	T_g (°C)	$T_{c,onset}$ (°C)	$T_{c,min}$ (°C)	$|\Delta H_c|$ (J g^{-1})	$T_{m,onset}$ (°C)	$T_{m,max}$ (°C)	ΔH_m (J g^{-1})	χ_c (wt.%)
PETV	80	131	137	23	233	247	36	10
PETRb	81	128	133	23	234	249	41	13
PETRc	80	126	134	20	235	251	42	16

Figure 3.12 : Data given DSC thermograms during a first of injection molded VPET and RPET (Torres *et al,* 1999).

This study shows that semi-crystalline samples possessing a different thermal ad mechanical history keep the rates of crystallization close (less than 10%) after heating run following quenching.

	$[\eta]$ (dl g^{-1})	$\overline{M}_w$ (g mol^{-1})
Pellets of PETV	0.76	44 000
Injection molded PETV	0.74	42 200
Scraps of PETRb	0.77	44 900
Injection molded PETRb	0.69	37 900
Scraps of PETRc	0.80	47 600
Injection molded PETRc	0.61	31 300

Figure 3.13 : Intrinsic viscosity and average molecular weight of virgin and recycled PET before and after injection moulding (Torres *et al*, 1999).

	$\overline{M}_w$ (g mol^{-1})	Young's modulus (N mm^{-2})	Elongation at break (%)	Charpy impact strength (notched, 20°C, kJ m^{-2})	Aspect of test bars
PETV	42 200	2140 (±206)	270 (±57)	3.0 (±0.2)	Transparent
PETRb	37 900	2170 (±184)	5.4 (±0.6)	2.4 (±0.5)	Opaque
PETRc	31 300	1996 (±210)	3.0 (±0.4)	1.8 (±0.3)	Opaque

Figure 3.14 : Mechanical Properties of injection molded virgin and recycled PET. (Torres *et al*, 1999).

From the results obtained, the crystallization of recycled PET can be favored by:

-the presence of impurities and the increased content of cyclic or linear oligomers that act as nucleating agents

-the decrease in instrinsic viscosity and average molecular weight,

-the different thermal and mechanical histories (the scraps coming from the bottles were crystallized by mechanical strectching, whereas the pellets were crystallized by heating),

-the presence of residual moisture coming in the shape of scraps

-the molecular orientation of material during the injection molding

These results also showed that degree of purity of recycled resin is also an important parameter on recycled PET properties (Torres *et al*, 1999).

In a another different study by Kang *et al* (2011), six kinds of PET sheets with varying percent of virgin(V) and recycled (R) PET contents (i.e., 100V, 80V20R, 60V40R, 20V80R and 100R PET) were commercially extruded and the optical, thermal, physicomechanical and barrier properties of the PET sheets were evaulated as function of RPET content. Differences were found between the sheets for UV and visible light absorption in the regions 200-350 nm and 670-700 nm, respectively (Figure 3.15). Intrinsic viscosities of 100V and 100R PET sheets were different.

PET sheet type	T_g (°C)	T_{cc} (°C)	T_c (°C)	T_m (°C)	χ_c (wt. %)
100V	79.2 ± 0.2^a	137.0 ± 0.5^a	168.9 ± 1.8^a	245.2 ± 0.1^a	6.9 ± 0.9^a
80V20R	77.1 ± 0.4^b	134.4 ± 0.2^b	184.6 ± 0.7^b	245.6 ± 0.4^a	8.6 ± 1.5^{ab}
60V40R	77.7 ± 0.4^b	133.3 ± 0.4^{bd}	185.1 ± 0.6^{bc}	245.8 ± 0.4^{ab}	9.3 ± 0.5^{ab}
40V60R	77.2 ± 0.4^b	130.6 ± 0.8^c	189.8 ± 2.4^c	246.7 ± 0.7^{bc}	10.1 ± 0.9^b
20V80R	77.9 ± 0.5^b	132.1 ± 0.6^d	188.6 ± 0.3^c	247.0 ± 0.2^c	9.3 ± 1.6^{ab}
100R	77.5 ± 0.5^b	132.5 ± 0.2^d	190.9 ± 0.9^c	247.5 ± 0.1^c	8.1 ± 0.7^{ab}

Values are means $\pm$ SD; within the same column, means with different superscripts are significantly different at $\alpha = 0.05$.

Figure 3.15 : Thermal properties of selected PET sheets (Kang *et al*, 2011).

PET sheet type	η (dl/g)	$\overline{M}_V$ (g/mol)
100V	0.722 ± 0.029^a	40742 ± 2052^a
80V20R	0.696 ± 0.022^a	38449 ± 1839^a
60V40R	0.630 ± 0.006^b	32989 ± 449^b
40V60R	0.631 ± 0.006^b	33038 ± 478^b
20V80R	0.607 ± 0.009^b	31141 ± 695^b
100R	0.533 ± 0.017^c	25479 ± 1275^c

Values are means $\pm$ SD; within the same column, means with different superscripts are significantly different at $\alpha = 0.05$.

Figure 3.16 : Intrinsic viscosity, η, and viscosity molecular weight of the solutions selected PET sheets at 24 ±0.5°C (Kang *et al*, 2011).

From that study, it obtained that all samples had intrinsic viscosity values between 0.53 and 0.72 ld/g; viscosity molecular weights ranged from 25,000 to 41,000 g/mol (Figure 3.16). Statistically significant differences in η was found between 100V and 100R PET. In general, the higher percent of RPET in the PET sheet solution, the lower the corresponding η and molecular weight.

One of the main drawbacks of RPET is the decrease in quality compared to virgin PET due to degradation caused by the simultaneous presence of retained moisture and contaminants during mechanical recycling (Karayannidis *et al,* 2000). The main degradation mechanisms of PET that occur during mechanical recycling are hydrolysis and thermal degradation. PET hydrolysis generally introduces carboxylic acid and hydroxyl-ester end groups. Thermal degradation generally produces carboxylic acid and vinyl ester end groups. These two degradation mechanisms reduce the molecular weight and decrease the intrinsic viscosity of the RPET

(Gionnotta *et al,* 1993). Thus, RPET products generally present reduced optical, physical, mechanical and barrier properties.

S. Khoramnejadian (2011) also studied about mechanical and thermal properties of recycled poly(ethylene terephetalat) (PET) from used bottle in 2011. He used polycarbonate for improve the properties of recycled PET, which decreased during recycling process. In this case, different level of polycarbonate is used mixing from 20% to 50%. This study showed that increasing the amount of polycarbonate decreased the brittleness of RPET, and improved the thermal resistance. Alloying the recycled PET with polycarbonate improved properties of PET matrix and, usability of recycled PET increased for various applications.

In addition, there is an interesting study called "The role of crystalline, mobile amorphous and rigid amorphous fractions in the performance of recycled poly(ethylene terephthalate) (PET)" (Badia *et al,* 2012). In that study, the action of thermo-mechanical degradation induced by mechanical recycling of poly(ethylene terephthalate) was simulated by successive injection moulding cycles. The role of crystalline, mobile amorphous and rigid amorphous fractions were searched leading to a loss of thermal, viscoelastic and mechanical features of recycled PET. This comprehensive study included to assess the surface characterization changes while reprocessing to PET (Figure 3.17).
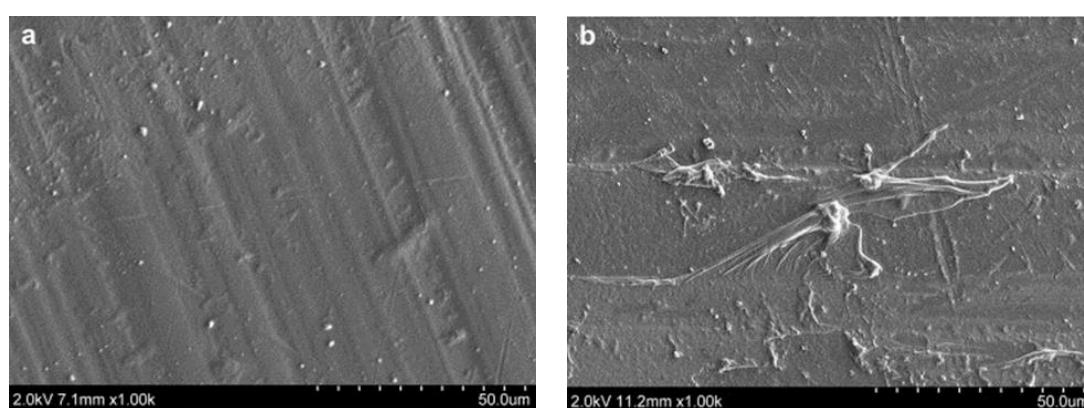
Figure 3.17 : Surface analysis by SEM (a: VPET, b: RPET) (Badia *et al,* 2012).

Zanin and Mancini (2000) studied the effects of consecutive recycling steps on PET and its impact on mechanical properties and the changes in glass transition temperatures and crystallinity. They observed increases in the number of carboxyl end groups and the crystallinity. They also reported an increase in modulus but a

decrease in ductility and impact resistance. Exposure to the atmosphere was also found to affect the properties.

Kegal *et al*. (2002) studied the effects of additives on processing and properties of RPET and found that additives like Titanium Dioxide (TiO2) and Linear low-density polyethylene (LLDPE) wax in master batch compositions can improve processing capability and other properties of RPET.

Koester *et al.* (2003) studied about thermoforming &die cutting recycled/virgin PET sheets and represented numerous aspects, considerations, and suggestions for thermoforming and die cutting PET sheets. In that context, they also determined propeties of RPET and PVC, which is shown in Figure 3.18.

Material	RPET	PVC
Gauge, Mils	10	10
Density, g/cm^3	1.33	1.35
Haze, %	0.5	1.2
Gloss at 45 deg. (Gardner)	110	93
Transparency, %	85	36
Tensile strength, psi	7,100	7,100
Tensile Modulus of Elasticity, psi	280,000	325,000
Oxygen Transmission Rate cc/sq.meter/24 hr/mil	109	174
HVTR.g/100 sq.in. /24 hr	0.40	0.19
Vicat Softening Point (°C)	80	82
Blushing	No	Yes
Dart, Impact, ½ in. Dart, g@ 26 in. drop		
At 73 °F (23 °C)	425	415
At -20 °F (-29 °C)	300	345
Heat Deflection (°F at 264 psi)	145	167
Sealing Temperature (°F)	275-400	315-360
Sheet Temperature (°F)	250-300	275-350

Figure 3.18 : Property comparison: RPET and PVC Koester *et al,* 2003).

Cornier-Rios et al (2007) also studied on effect of recycling on material properties of glass-filled PET. The objective of that study to characterize %15 wt.% glass-filled PET (RPET-15GF) using six recycle generations and four recycle ratios. Mechanical properties such as tensile strength, elastic modulus, and percent elongation to failure of PET composite were determined for varios recycle generations and recycle ratios. Results showed that the mechanical properties of RPET-15GF decrease slightly per cycle generation. In contrast, thermal properties of RPET-15GF were not all affected

by the recycling process. This data demonstrates that recycled glass-filled PET can be used effectively to fabricate components without significantly affecting their mechanical performance.

A study carried out by Oromiehie and Mamizadeh (2004) on a variety of PET and RPET samples was aimed at recycling PET bottles and looking at means to improve their properties. It was found that the molecular weight and intrinsic viscosity decreased with increase in the content of RPET. Their inference was that this was due to shear degradation of the RPET. They also stressed the importance of the number of thermal cycles for recycling PET and its blends, since it would impact the crystallinity and mechanical properties.

There is also a comphensive study (Rajakutty, 2010) called "Static and Dynamic Mechanical Properties of Amorphpus Recycled Poly(ethylene terepthalate" by A. Rajakutty in 2010. That work was aimed at studying the mechanical properties of RPET and comparing these properties with those from virgin PET. The dynamic behavior of PET was part of that study. Apart from studying the mechanical properties of RPET, several other tests were performed to study thermal properties, crystallinity, color measurements (yellowing), friction behavior and also to determine structural performance of blow molded bottles. Material properties obtained from experimental results were used as input for Finite Element simulations. The results obtained from the study are;

- The DSC results from the dynamically stretched samples confirmed the effect of strain induced crystallization at room temperature from higher strain rates (>50 mm/min). This was associated with whitening or "crazin" in the polymer and was the same in RPET or virgin PET.
-Both virgin PET and RPET had similar crystallinity.
- A 5% decrease in total energy consumption was observed when rPET was blow molded compared to other virgin PET resins. This is a very encouraging result, which would support using RPET on a larger scale and reducing the impact on the environment. This is supported by the higher IR absorption of RPET.
-RPET resin was found to have lower values of friction coefficient than some other virgin PET resins.
-The hoop strength results for the bottle stiffness showed RPET bottles having stiffness values close to virgin PET bottles.

- Finite element lean test simulations showed similar deformation pattern for the bottles of different resins. The structural performance of RPET bottle was superior compared to some of the virgin PET resins used in industry.

The percent crystallinity, the size of spherulites and the molecular weight of semi-crystalline polymers, such as PET, affect the mechanical properties of materials. Usually, the crystallization produces a drastic mobility restriction that renders the material brittle. Giannotta *et al* (1993) has shown that PVC contamination can also increase the level of undesirable cyclic and linear oligomers formed in PET during melt processing. These oligomers can affect mechanical properties of the material (Torres, Robin 1999).

In fact, the difference between virgin PET and Recycled PET is extremely small. A study by the Waste & Resources Action Programme (WRAP) showed that consumers could not tell the difference between products made of recycled material.

4. MATERIALS AND METHOD

4.1. Materials

The aim of this study was to use recycled PET fibers instead of virgin PET fibers in needlepunched automobile carpets in order to achieve cost reduction. The main goal was to reach the same or comparable quality levels in the carpets with recycled PET fibers as would be achieved by using virgin PET fibers.

Seven different PET fibers supplied from different producers were used in this study. The suppliers were;
-Advansa from Adana-Turkey,
-Green Fiber International S.A from Romania,
-DS Fiber from Belgium,
-Yılmaz Textile from Adana-Turkey and
-Spin Global.

These fibers are given at Table 4.1.

Table 4.1 : Fiber samples supplied from different producers.

Fiber Samples	Fiber Fineness and Supplier
Sample A	VPET 12 denier (Advansa)
Sample B	RPET 11 Denier (Green Fiber)
Sample C	RPET 6 Denier (Green Fiber)
Sample D	RPET 11 Denier (DS Fiber)
Sample E	RPET 12 denier (Yılmaz Textile)
Sample F	RPET 6 denier (Yılmaz Textile)
Sample G	Bicomponent PET 5 dtex (Spin Global)

4.2 Preparation of Fiber Blends to be used in Needlepunched Carpet Production

Blends of virgin, recycled and bicomponent (bico) PET fibers were prepared in the following weight ratios (Table 4.2). Eight needlepunched nonwoven carpets were produced from these blends using the same process parameters. These carpets were moulded at laborotary conditions and tested according to specification requirements.

Table 4.2 : Fiber blend combinations for production.

Production Samples	
Sample No	**Fiber Blend Combination**
Sample 1	VPET 12 denier (Advansa) + 5 dtex Bicomponent PET (Spin Global)
Sample 2	VPET 12 denier (Advansa)
Sample 3	RPET 12 denier (Yılmaz Textile)
Sample 4	RPET 12 denier (Yılmaz Textile) + 5 dtex Bicomponent PET (Spin Global)
Sample 5	RPET 12 denier (Yılmaz Textile) + RPET 6 denier (Green Fiber) + 5 dtex Bicomponent PET (Spin Global)
Sample 6	RPET 6 denier (Green Fiber) + RPET 11 denier (Green Fiber) + 5 dtex Bicomponent PET (Spin Global)
Sample 7	RPET 6 denier (Yılmaz Textile)
Sample 8	RPET 11 denier (Green Fiber) + 5 dtex Bicomponent PET (Spin Global)

4.3 Characterization of Fiber Samples

The following tests were carried out on the fiber samples in order to characterize their physical, mechanical and morphological structure. This was intended to be able to make a comparison between recycled and virgin PET fiber properties and also to make analysis about their effects on the end product quality.

- Intrinsic Viscosity (IV)
- Differential Scanning Calorimetry (DSC)
- Fiber Physical Tests (Staple Length, Fineness and Crimp)
- Breaking Strength and Elongation
- Dynamic Mechanical Analysis (DMA)
- Scanning Electron Microscopy (SEM)

4.3.1 Determination of intrinsic viscosity (IV)

Intrinsic viscosity is a measure of a solute's contribution to the viscosity of a solution. Intrinsic viscosity (Equation **4.1**) is defined as,

$$[\eta] = \lim_{c \to 0} \frac{\eta_{sp}}{c} \equiv \lim_{c \to 0} c^{-1} \ln \eta_{rel} \qquad (4.1)$$

It is one of the most precise measurements in polymer science, also the simplest and cheapest. Intrinsic viscosity, which is measured from the flow time of a solution through a simple glass capillary, has considerable historical importance for establishing the very existence of polymer molecules.

In this test, the intrinsic viscosity was measured by means of LAUDA DL30 KP capillary viscometer type at a temperature of 100-110°C, using a mixture of phenol and 1,2 dichlorobenzene as the solvents in the ratio of 3:2 (w/w) at Kortex Laboratory.

Capillary viscometry (Figure 4.1) is conceptually simple: the time it takes a volume of polymer solution to flow through a thin capillary is compared to the time for a solvent flow. It turns out that the flow time for either is proportional to the viscosity, and inversely proportional to the density.
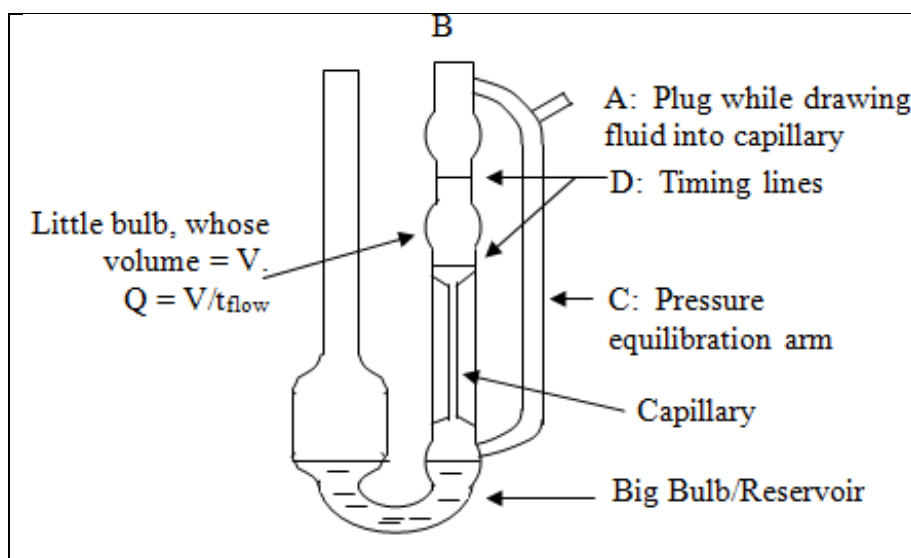
Figure 4.1 :A sketch of capillary viscometer (macro.lsu.edu/howto/intrinsicvisc.doc)

Five main measurements were performed and the average viscosity value of the measurements was reported automatically by software.

4.3.2 Differential scanning calorimetry (DSC)

DSC analyses of the samples were carried out by DSC Q1000 instrument (Figure 4.2) at Istanbul Technical University Laboratory. Melting temperatures of the fiber samples were determined by this analysis.

In order to determine crystallinity ratios of the fiber samples, DSC analysis was also carried out in Pelzer Pimsa Laboratory in Germany.

Thermal analysis using Differential Scanning Calorimetry (DSC) is considered a very effective method of determining the crystallinity content in polymers. Thermal analysis helps to determine properties of the material as a function of temperature. DSC helps us to determine the percentage of crystallinity content as well as the amorphous content in a polymer.

Crystallinity is considered as one of the most important properties for a semi crystalline thermoplastic. It helps in determining the mechanical properties of a polymer such as brittleness, toughness, modulus, clarity, creep and barrier properties (Sichina, 2000).

A differential scanning calorimeter measures the heat in and out of a sample and also measures heat of the sample with respect to a reference. It can heat or cool the sample based on the input parameters given and is ideally used to measure the transition temperature such as the glass transition temperature, melting temperature, crystallization temperature and also the heat capacity. One can observe endothermic and exothermic peaks in the DSC thermogram.

For all the DCS measurement, samples weighing approximately 10 mg were cut from the test specimens, placed in a small chamber, and closed with lids. That chamber was heated at a constant rate of 10˚C per minute up to 300˚C and then cooled. The energy in the chamber was measured as the samples were heated. The glass transition was characterized according to the temperature in its inflection point (T_g) and the jump in heat capacity (ΔC_p) were recorded. Crystallization and melting phenomena were assessed in terms of their onset and peak temperatures, along with the enthalpies related to their areas to common baselines (Rajakutty, 2010).
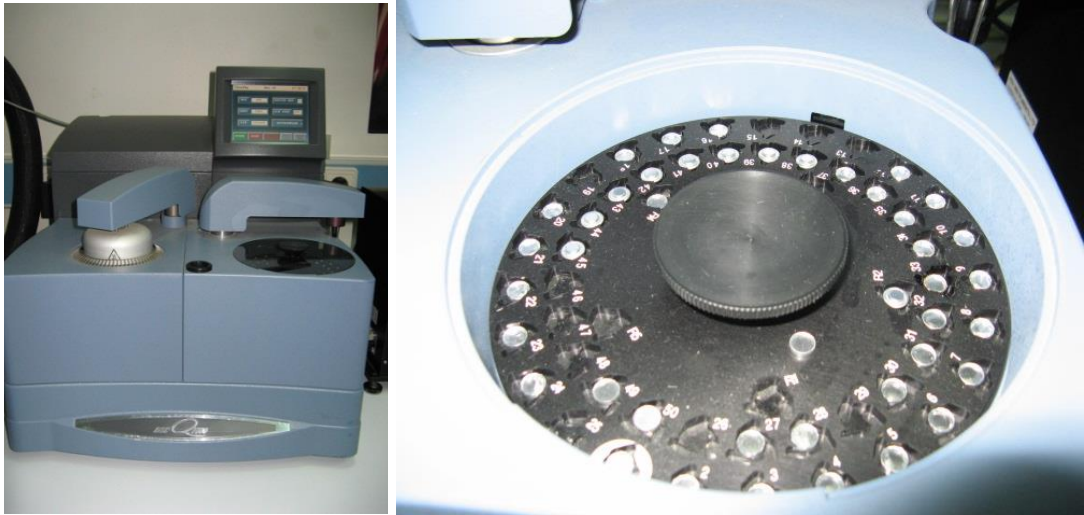
Figure 4.2 : DSC Q 1000 and the chamber.

A Typical thermogram for quench cooled PET is shown in Figure 4.3.
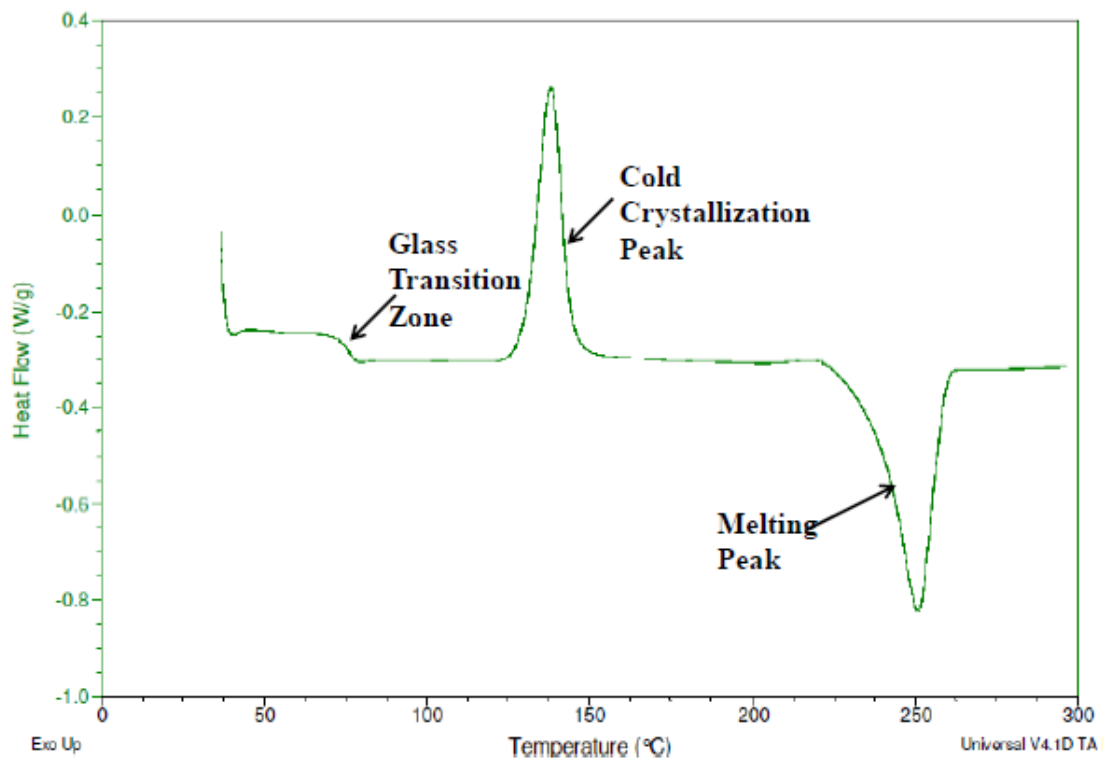
Figure 4.3 : A Typical thermogram for quench cooled PET.

4.3.3 Fiber physical tests (fiber staple length, fineness and crimp)

For physical characterization of the fibers tests, fiber fineness, fibre staple length and crimp number tests were made in Cerkezkoy TAV Laboratory founded by Turkish Textile Foundation.

4.3.3.1 Fiber fineness

Fineness is one of the most important properties of the fibres that are made into textile products. The fibre fineness has a number of effects on the properties of the yarn and hence the fabric that is made from it.

The most important effect of fibre fineness is on the fibre stiffness. This is because the rigidity of a fibre increases with the fourth power of the fibre diameter so that a coarser fibre is a great deal stiffer. The stiffness of the fibres affects the stiffness of the fabric made from it.

When considering ways of measuring fibre fineness there are a number of factors that need to be taken into account which make it difficult to define a measure of fineness that is applicable to all fibres:

1. The cross-section of many types of fibres is not circular. Wool has an approximately circular cross-section but silk has a triangular cross-section and cotton is like a flattened tube. Manmade fibres are often made with trilobal, star or hollow cross-sections for particular purposes. This makes it impossible to have a universal system of fibre fineness based on fibre diameter.

2. The cross-sections of the fibres may not be uniform along the fibre length. This is often the case with natural fibres.

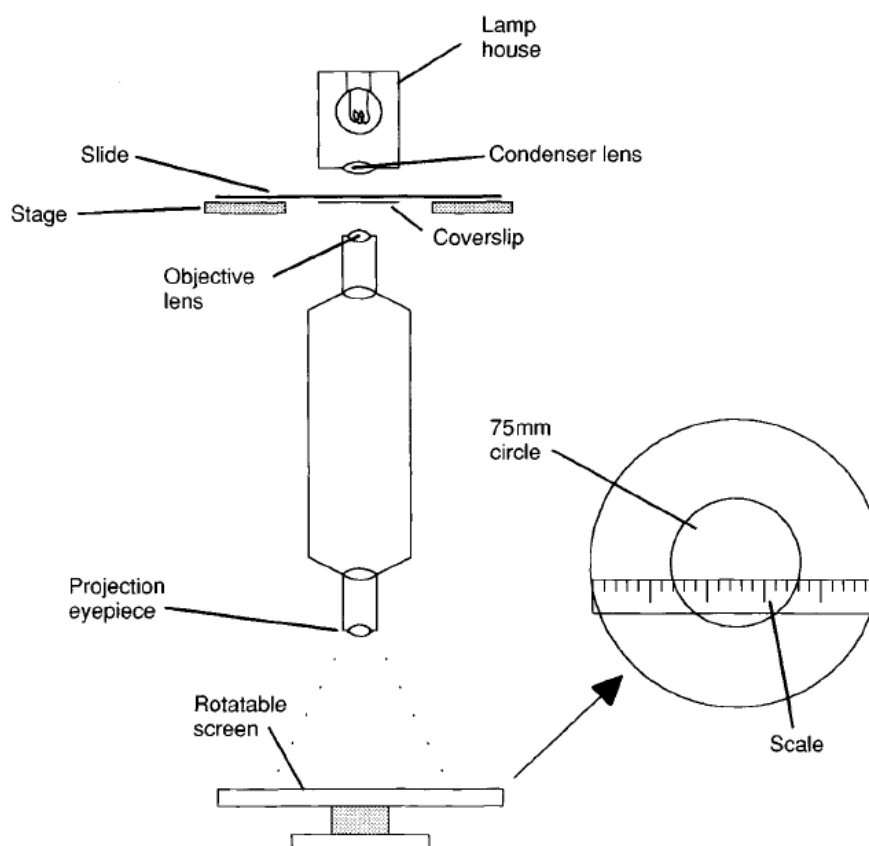3. The cross-sectional shape of the fibres may not be uniform from fibre to fibre.

Because of these problems a definition of fibre fineness is needed that can allow for all the variations but that leads to a method of measurement which is relatively simple to carry out. The great degree of variability found in natural fibres means that a large number of measurements have to be carried out in such cases. There are a number of different ways of measuring fibre fineness/ diameter, which differ fundamentally in their definitions of fineness so that the measurements are not easily interconvertible**:**

- Gravimetric Method

- Fibre fineness by projection microscope

- Fibre fineness by the airflow method (Saville, 1999).

Projection microscope was used to measure fiber fineness of samples according to TS 1186 standards.
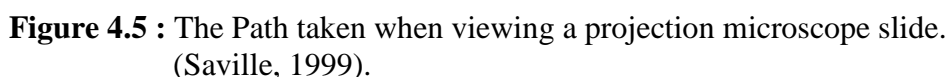
The projection microscope is the standard method for measuring diameters of fibers with a circular cross-section. The method involves preparing a microscope slide of short lengths of fibre, which is then viewed using a microscope that projects an image of the fibres onto a horizontal screen for ease of measurement. The apparatus is shown diagrammatically in Fig. 4.4. Techniques are followed that avoid bias and ensure a truly random sample.

A suitable random and representative sample is conditioned for 24 h in a standard testing atmosphere. Using a modified Hardy microtome the fibres are cut to a suitable length (0.4mm for fibres below 27 μm) and a slide is prepared by carefully mixing the fibres into the mountant. The use of short fibres gives a length-biased sample so that proportionally more of the longer fibres will have their diameter measured. The mounting agent should be non-swelling and have a suitable refractive index (for example liquid paraffin).

Figure 4.4 : The projection microscope (Saville, 1999).

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The slide is placed on the stage, coverglass down (microscope inverted) and fibres are selected for measurement in the following way. The slide is traversed in a zigzag fashion as shown in Fig 4.5, measuring every fibre that complies with the following requirements:

1. has more than half its length visible in the 7.5cm circle which is drawn in the centre of the field of view;

2. is not in contact with any other fibre at the point of measurement.

Figure 4.5 : The Path taken when viewing a projection microscope slide. (Saville, 1999).

The traverse of the slide is continued until the required number of fibres has been measured. The magnification of the microscope is adjusted to be 500x so that on the scale used to measure the fibres each millimetre represents 2 μm.

For accurate tests, three slides should be measured from randomly selected areas of the material and not less than 150 fibres per slide should be measured.

According to TS 1186 standard, the magnification of the microscope was adjusted to be 250x and the number of 400 single fiber samples was tested as a micron based, then the results were converted to denier.

The coefficient of variation of diameter for unblended fibers lies between 20% and 28%. From this value, the number of tests to give certain confidence limits has been calculated and is shown in Table 4.3.

Table 4.3 : Number of measuremets required for a given accuracy (Saville, 1999).

Confidence Limit	Number of measurements
1%	2500
2%	600
3%	300
5%	100

4.3.3.2 Fiber length

In this test, for each sample 10 individual fibers were measured directly by steel ruler and calculated the average fiber lenght in accordance to the TS 1162 standard.

After fineness, length is the most important property of a fibre. Synthetic fibres like PET can be cut during production to whatever length is required either with all the fibres having the same length or with a distribution of lengths. Nevertheless, it is still needed to measure the fiber lenght.

The methods used to measure fibre length fall into two main types: the direct measurement of single fibres mainly for research purposes and methods that involve preparing a tuft or bundle of fibres arranged parallel to one another. In this case, the fibres can be grouped for measurement or ultimately the measurement can be completely automated.

The simplest direct way of measuring single fibres is by hand. Each end of the fibre is grasped by a pair of tweezers and the fibre stretched alongside a rule. The tension applied when holding the fibres must be just sufficient to remove any crimp but not enough to stretch the fibre. In order to have a more even tension during the measurement a weight may be hung on the end of the fibre but the method then becomes slower still (Saville, 1999).

4.3.3.3 Fibre crimp

Like fiber length determination, fiber crimp was tested by hand. 10 single fibers with 1 cm length were prepared and measured by a steel ruler. The number of crimp was calculated for each individual fiber. Then the average of crimp number was determined.

Crimp in a textile strand is defined as the undulations or succession of waves or curls in the strand, induced either naturally during fiber growth, mechanically, or chemically. Crimp in a fiber is thus considered as the degree of deviation from linearity of a non-straight fiber. Fiber crimp characteristics have a big influence on the processing performance of the fibers. Crimp also contributes essentially to the properties of intermediate fiber assemblies, yarn and finished fabrics. Fiber crimp imparted to synthetic fibers, which are initially straight, makes it possible to process these fibers with existing machinery designed for natural fibers. Straight, slick

synthetic fibers would not have sufficient cohesion for carding, combing, drawing, roving, and spinning. In nonwoven processes, crimp and crimp retention during processing are major contributors to processing efficiency, cohesion, fabric bulk and bulk stability (Saville, 1999).

The crimp of the textile fibers is also very critical to determine its properties and voluminisity. The microscopic characteristic of the nonwoven fabric is totally depends on the linearity and length of the crimp and the amplitude of the crimp. (Singha et al, 2013)

4.3.4 Determination of breaking strength and elongation

Instron 4411 Universal Testing Instrument was used to measure breaking strength and elongation of the fiber samples. The tests were carried out in Marmara University Textile Physical Testing Laboratory. Before the test, all samples were conditioned under standard atmosphere at a temperature 20±2ºC and a relative humidity 65% for 24 hours.

Tensile tests were performed on the Instron 4411 Universal Testing Instrument at a crosshead speed of 10 mm/min., a 1 kN load cell and gauge length of 10 mm. Analysis were repeated at least twenty (20) times per sample, and the average elongation at break and break stress were determined.

The Instron 4411 Universal Testing Instrument (Figure 4.6) is a material testing instrument designed to test the strength of a wide variety of materials. The system is made up of a load frame, in which a specimen of the test material is mounted, that applies a tension or compression load to the specimen, and a control console that provides the calibration, test setup, and test operating controls. The control console is compact enough to mount directly on the load frame, eliminating the need for a separate support table or workbench.

The Instron 4411 control system is made up of two major subsystems: a crosshead drive and control system that applies tensile or compressive loading to a specimen; and a highly sensitive load weighing system, that measures the loading of a specimen.

Figure 4.6 : The Instron 4411 Tensile Tester.

During the test, results occur as tracked (instantaneous) values of load, extension and strain or, after a test, as stored break and peak values of these parameters. Total energy and load and energy values at preset points are also available as stored parameters. A digital output device is viewing and recording test results.

Test results were given as tenacity (breaking force/ denier), elongation % and CV %. CV % is calculated in Equation **4.2.**

$$CV \% = (s/x) *100 \tag{4.2}$$

s: Standard Deviation
x: Average Strengh of Fiber

4.3.5 Dynamic mechanical analysis (DMA)

Dynamic mechanical analysis (DMA) is very useful method for investigation of mechanical properties, relaxation process, and the associated molecular motions and internal changes in polymeric materials as a fuction of temperature, time, frequency, stress, atmosphere or a combination of these parameters.

In this study, we performed DMA measurements using Gabo Eplexor 100 N Dynamic Mechanical Analyzer (Figure 4.7) at Kordsa Global in a temperature sweep test in order to determine how well the fibre will absorb/loose energy as the temperature increased from room temperature up to 240°C with isothermal steps.

This kind of temperature sweep will provide the information of the fibre behaviour under real conditions coupled with certain frequency, which is mostly at 100°C. Storage and loss moduli of PET fibers are obtained. With dynamic mechanical method, glass transitions are observed only for such a unique combination of deformation (1%), temperature and frequency (10Hz) that causes mobility of

sufficiently large numbers of chain segments in a given time period. Thus, as the frequency increases, more energy in the form of heat is required for the motion of a sufficient number of segments. This is mainly the reason why Tg values obtained by dynamic mechanical methods may be as much as 50°C higher than values obtained from DSC, in general.

In this test, only 11-denier RPET, 6-denier RPET and PET produced by Kordsa were tested and compared to each other in order to observe the thermal differences between a virgin PET and Recycled PET.

Figure 4.7 : Gabo Eplexor 100 N Dynamic Mechanical Analyzer.

4.3.6 Scanning electron microscopy (SEM)

Scanning Electron Microscopy (SEM) is a high-resolution imaging of surface method that is used to identify microscopic characteristics of polymers and fillers. The SEM uses electrons for imaging, much as a light microscope uses visible light. The advantages of SEM over light microscopy include greater magnification (up to 100,000X) and much greater depth of field (www.mee-inc.com/sem.html). SEM techniques have been routinely used for characterizing the polymer morphology and the investigation of fractured surfaces (Lee et al, 2003).

The morphology of the specimens was analysed by means of an EVO MA10 Scanning Electron Microscope at Istanbul Technical University Laboratory. The samples from each material were prepared by cutting squared pieces from a randomly chosen part of the processed specimen. The pieces were mounted on metal

studs and sputter-coated with a 2 nm gold layer using a Emitech high resolution sputter coater (Figure 4.8) to improve conductivity properties.

Figure 4.8 : Film coating by Emitech sputter coater and preparation of specimens.

After coating process, specimens for each sample were imaged of 250x, 500x, 1000x, 2500x, 5000x magnifications by using EVO MA10 SEM Instrument (Figure 4.9). Our aim was to observe fiber surface characteristics and determine differences on fiber surfaces among sample fibers.
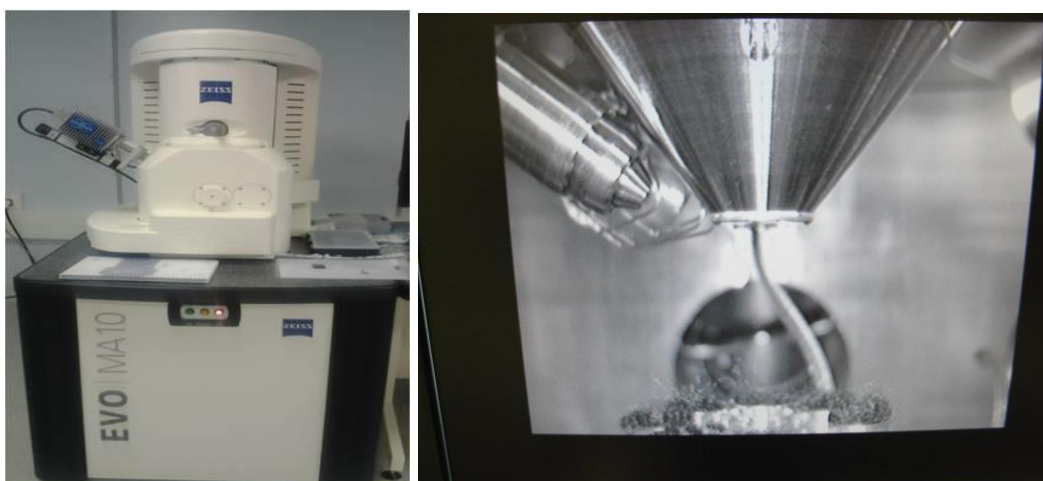
Figure 4.9 : EVO MA10 SEM Instrument and imaging head.

5. RESULTS AND DISCUSSION

5.1. Intrinsic Viscosity (IV) Test Results

Table 5.1 shows the intrinsic viscosity values of the fibers used in the study.

Table 5.1 : Intrinsic viscosity (η) values of sample fibers.

SAMPLE CODE	INT.VISCOSITY
SAMPLE A- VPET (Advansa)	0.635 dl/g
SAMPLE B- RPET 11 Denier (Green Fiber)	0.585 dl/g
SAMPLE C- RPET 6 Denier (Green Fiber)	not solved
SAMPLE D- RPET 11 Denier (DS Fiber)	not solved
SAMPLE E- RPET 12 denier (Yılmaz Textile)	0.523 dl/g
SAMPLE F- RPET 6 denier (Yılmaz Textile)	0.529 dl/g
SAMPLE G -Bicomponent PET (Spin Global)	0.627 dl/g

As seen on the table above, all samples had intrinsic viscosity (η) values between 0.523 and 0.635 dl/g. It is known that PET bottles are normally produced with an intrinsic viscosity value ranging between 0.70-0.78, which is higher than the virgin PET fibers' value.

According to obtained (η) values on the table 5.1, the recycled PETs produced from bottles have a strong decrease in (η) values, consequently in molecular weight. In parallel to literatur (Kang *et al*, 2011 and Torres *et al*, 1999), this is a consequence of the difference in crystallinity, the presence of impurities in the recycled PET.

VPET and Bicomponent PET's intrinsic viscosity (η) values are the highest (above 0.6), however RPET's intrinsic viscosity (η) is below than 0,6. Nevertheless, there is no significant difference in intrinsic viscosity (η) values among RPETs from different suppliers.

5.2. Differential Scanning Calorimetry (DSC) Analysis

The DSC thermograms of the fiber samples are given in Figures 5.1, 5.2, 5.3, 5.4, 5.5, 5.6 and 5.7.
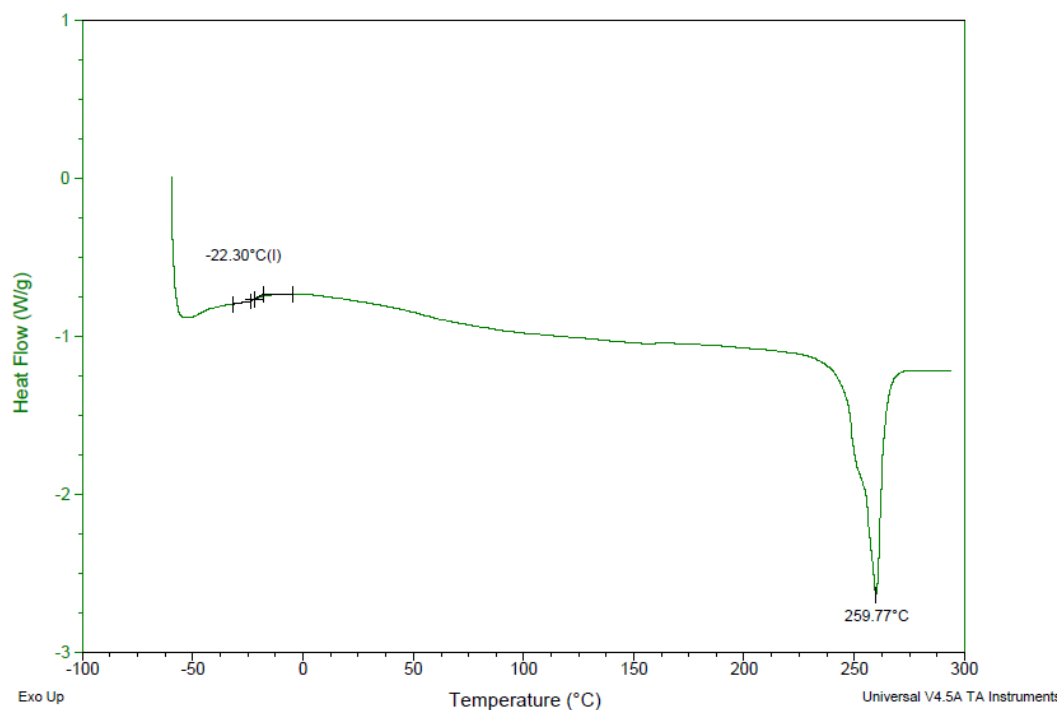
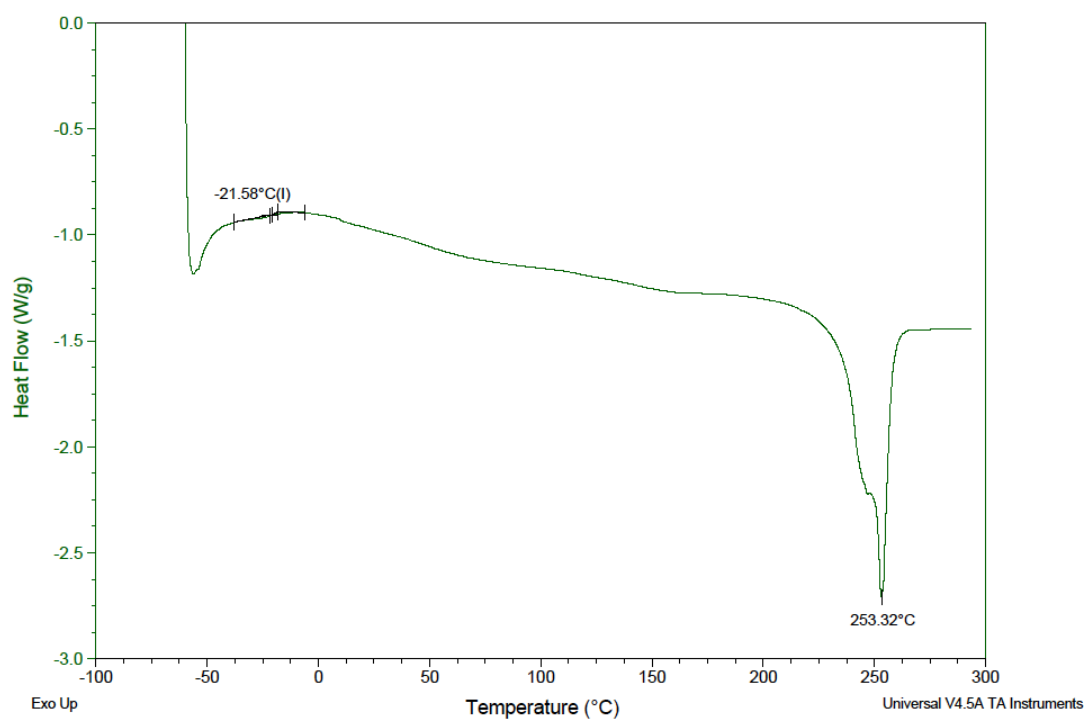**Figure 5.1 :** DCS thermogram of Sample A-VPET.
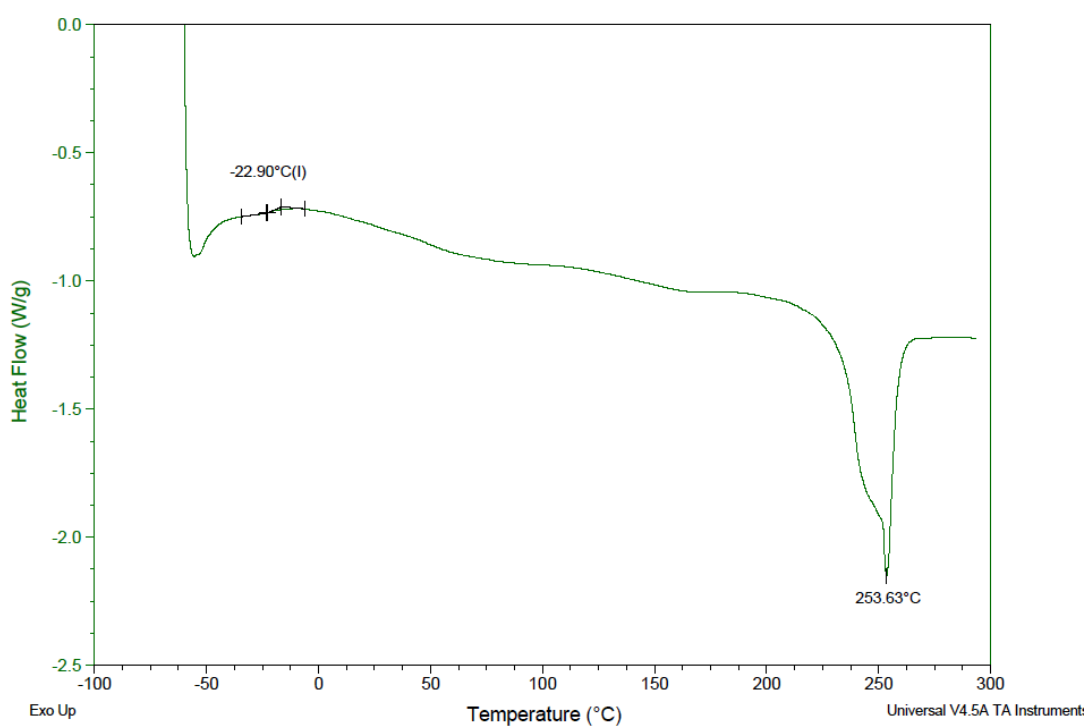
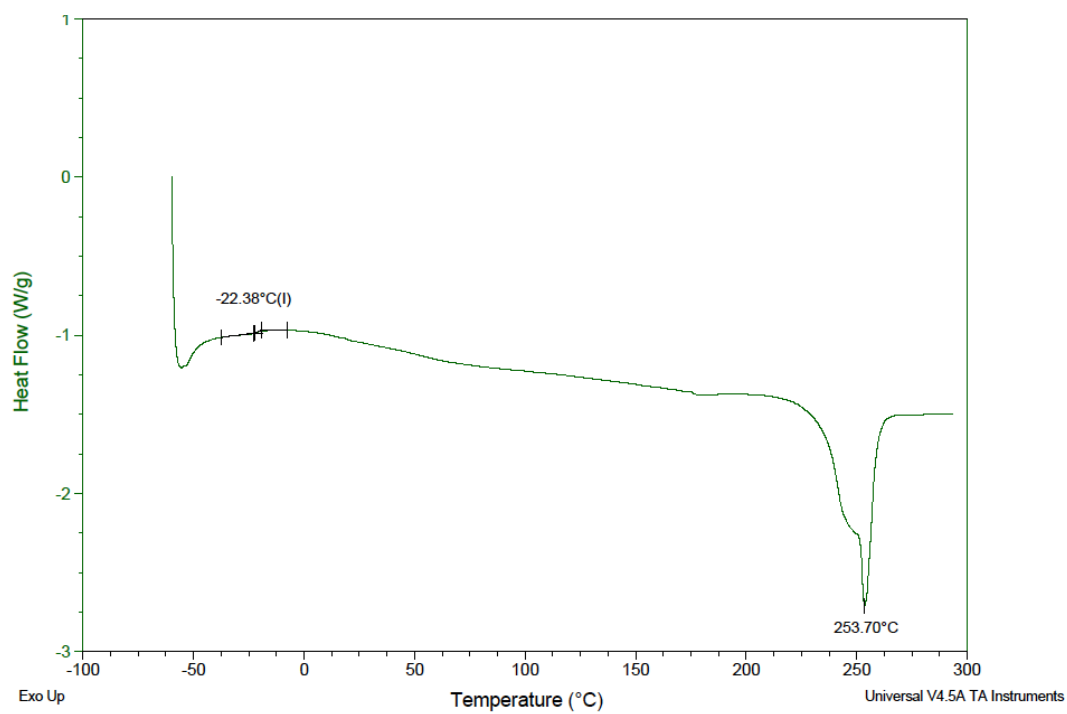**Figure 5.2 :** DCS thermogram of Sample B- RPET 11 Denier (Green Fiber).
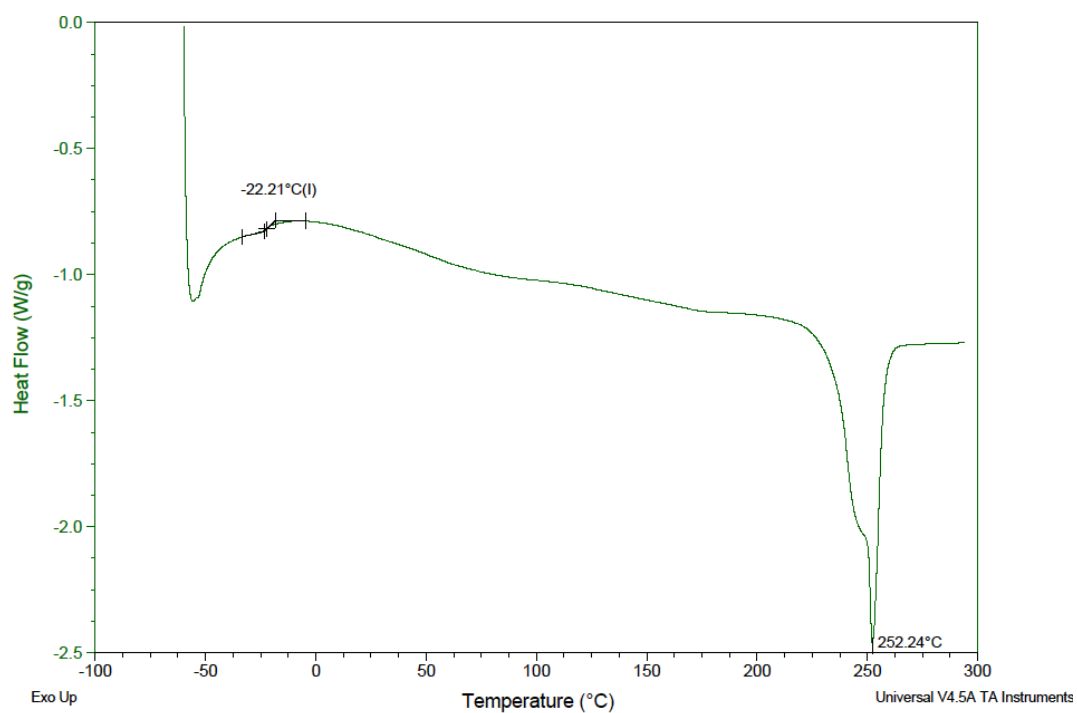
Figure 5.3 : DCS thermogram of Sample C- RPET 6 Denier (Green Fiber).
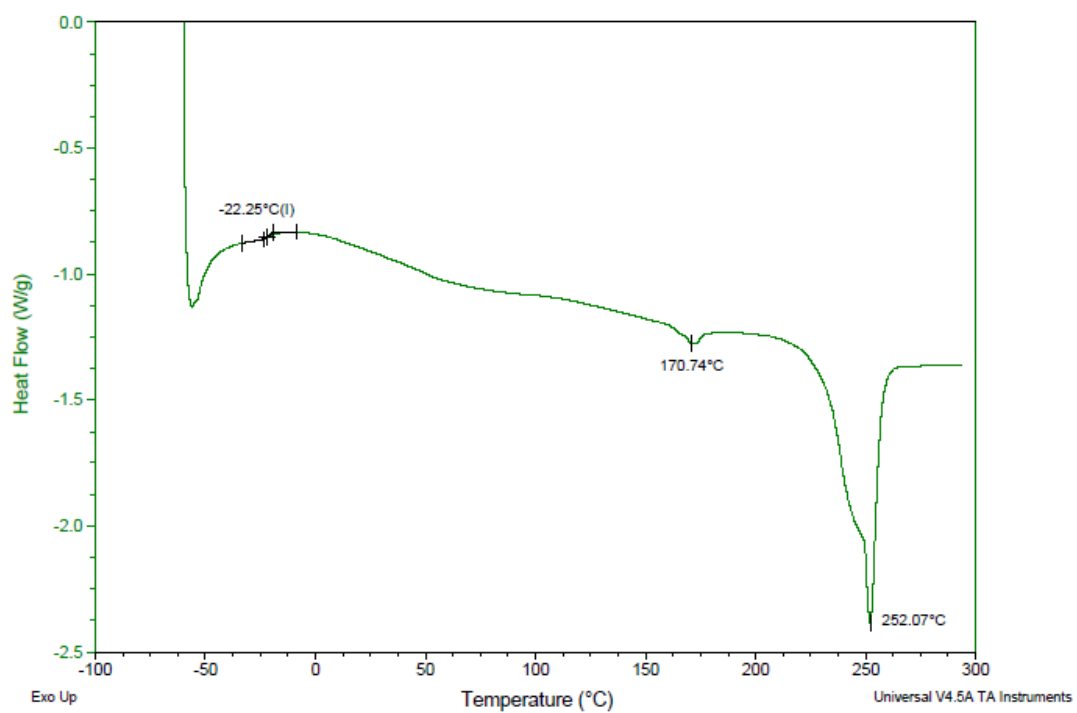
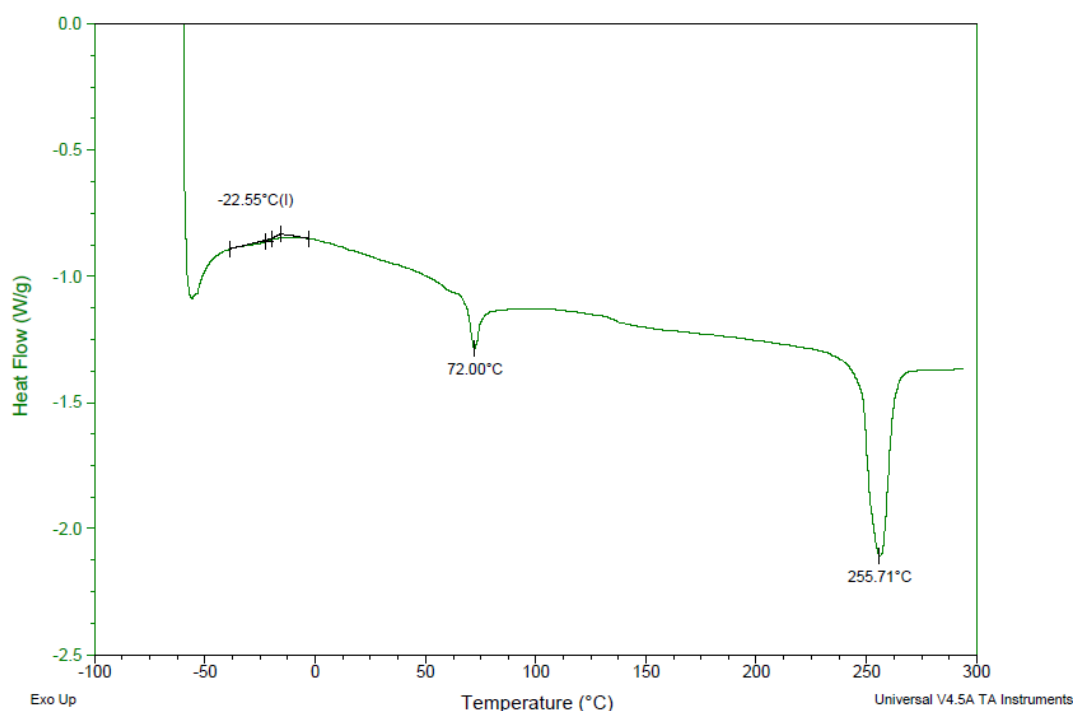**Figure 5.4 :** DCS thermogram of Sample D- RPET 11 Denier (DS Fiber).

Figure 5.5 : DCS thermogram of Sample E- RPET 12 denier (Yılmaz Textile).

Figure 5.6 : DCS thermogram of Sample F- RPET 6 denier (Yılmaz Textile).

Figure 5.7 : DCS thermogram of Sample G -Bicomponent PET (Spin Global).

Melting temperatures of the PET fibers were determined from the thermograms and are given on Table 5.2.

Table 5.2 : Melting Points of Fibre Samples.

SAMPLE CODE	MELTING POINT
SAMPLE A- VPET (Advansa)	259.77 °C
SAMPLE B- RPET 11 Denier (Green Fiber)	253.63 °C
SAMPLE C- RPET 6 Denier (Green Fiber)	252.24 °C
SAMPLE D- RPET 11 Denier (DS Fiber)	252.07 °C
SAMPLE E- RPET 12 denier (Yılmaz Textile)	253.32 °C
SAMPLE F- RPET 6 denier (Yılmaz Textile)	253.70 °C
SAMPLE G -Bicomponent PET (Spin Global)	72 - 255.71 °C

Sample A, which is the virgin PET fiber supplied from Advansa has the highest melting temperature, being 259.77°C. This fiber also has the highest intrinsic viscosity value. This is followed by Sample G, which is the virgin bicomponent PET fiber from Spin Global, as 255.71°C belonging to the high-temperature melting component. Recycled PET fibers have lower melting points in comparison to the virgin PET fibers as it was expected. Their melting temperatures range between 252.07°C to 253.70°C and also the intrinsic viscosities of these fibers range between

0.523-0.585 dl/g. It would be expected that virgin fibers would have higher intrinsic viscosities and also melting temperatures than recycled ones.

From DSC analysis, it was found that the other component of Bicomponent fiber has 72 °C melting temperature value, providing bonding in the production.

Table 5.3 : Crystallinity % of Fibre Samples.

SAMPLE CODE	(ΔCp) (j/g)	Crystallinity in %
SAMPLE A- VPET (Advansa)	58	41
SAMPLE B- RPET 11 Denier (Green Fiber)	Not recorded	Not recorded
SAMPLE C- RPET 6 Denier (Green Fiber)	61	43
SAMPLE D- RPET 11 Denier (DS Fiber)	50,1	36
SAMPLE E- RPET 12 denier (Yılmaz Textile)	56,08	41
SAMPLE F- RPET 6 denier (Yılmaz Textile)	53,3	38
SAMPLE G -Bicomponent PET (Spin Global)	22,8	16

The crystallinity of PET is an important property to measure, as it influences the mechanical properties (Sichina, 2000).

Tensile strength is directly related to the orientation and crystallinity in PET (Viana *et al*, 2004, Oultache *et al*, 2001).

From the Table 5.3 it was achieved that similar degree of crystallization were achieved by sample A- virgin PET from Advansa, sample C-RPET from Green Fiber and sample E- RPET from Yılmaz Textile.

Cyrstallinity % of sample G-bicomponent PET fiber had the lowest value due to low temperature melting component of bicomponent fiber.

In paralell to literature (Pattabiraman, 2005 and Torres, 1999), these results showed that all these semicrystalline samples kept the rates of crystallization close (less than 10%) after a heating run following quenching. That indicated that the degree of cyrstallinity is hardly affected by the RPET content.

5.3. Fiber Physical Tests (Staple Length, Fineness and Crimp) Results

The Physical test results of the fiber samples are given in Tables between 5.4-5.11.

Table 5.4 : Fiber Physical Test Results of Sample A- VPET (Advansa).

Fiber Fineness (denier)	Distribution %
Fiber Length: 57,1 mm	Denier
Crimp no: 3/1 cm	6,3 dn: 1.5 %
	7,3 dn: 4,1 %
	8,4 dn: 3,0 %
	9,5 dn: 17,8 %
	10,8 dn: 12,7 %
	12,1 dn: 31,5 %
	13,4 dn: 7,1 %
	14,9 dn: 14,2 %
	16,4 dn: 1,5 %
	18,0 dn: 5,1 %
	19,7 dn: 0,5 %
	21,5 dn: 1,0 %
Average Fiber Fineness: 11,9 denier	
Number of fiber tested: 400	

Table 5.5 : Fiber Physical Test Results of Sample B- RPET 11 Denier (Green Fiber).

Fiber Fineness (denier)	Distribution %
Fiber Length: 63 mm	Denier
Crimp no: 4/1 cm	2,4 dn: 0,4 %
	3,7 dn: 0,8 %
	4,5 dn: 0,8 %
	5,4 dn: 3,8 %
	6,3 dn: 5,5 %
	7,3 dn: 12,7 %
	8,4 dn: 6,0 %
	9,5 dn: 24,6 %
	10,8 dn: 13,6 %
	12,1 dn: 16,5 %
	13,4 dn: 2,1 %
	14,9 dn: 6,0 %
	16,4 dn: 1,3 %
	18,0 dn: 3,8 %
	19,7 dn: 0,8 %
	21,4 dn: 0,8 %
	23,3 dn: 0,4 %
Average Fiber Fineness: 10,2 denier	
Number of fiber tested: 400	

Table 5.6 : Fiber Physical Test Results of Sample C- RPET 6 Denier (Green Fiber).

Fiber Fineness (denier)	Distribution %
Fiber Length: 57,5 mm	Denier
Crimp no: 5/1 cm	2,4 dn: 1,5 %
	3,7 dn: 7,5 %
	4,5 dn: 13,6 %
	6,3 dn: 23,3 %
	7,3 dn: 16,2 %
	8,4 dn: 28,3 %
	9,5 dn: 4,6 %
	10,8 dn: 3,6 %
	13,4 dn: 0,5 %
	16,4 dn: 0,5 %
	18,0 dn: 0,5 %
Average Fiber Fineness: 6,1 denier	
Number of fiber tested: 400	

Table 5.7 : Fiber Physical Test Results of Sample D- RPET 11 Denier (DS Fiber).

Fiber Fineness (denier)	Distribution %
Fiber Length: 60,22 mm	**Denier**
Crimp no: 3,6/1 cm	5,4 dn: 0,6 %
	6,3 dn: 1,2 %
	7,3 dn: 4,1 %
	8,4 dn: 10,5 %
	9,5 dn: 29,6 %
	10,8 dn: 17,4 %
	12,1 dn: 25,5 %
	13,4 dn: 2,9 %
	14,9 dn: 5,2 %
	16,4 dn: 1,2 %
	19,7 dn: 1,2 %
	21,5 dn: 0,6 %
Average Fiber Fineness: 10,65 denier	
Number of fiber tested: 594	

Table 5.8 : Fiber Physical Test Results of Sample E- RPET 12 denier
(Yılmaz Textile)

Fiber Fineness (denier)	Distribution %
Fiber Length: 63,4 mm	**Denier**
Crimp no: 4/1 cm	5,4 dn: 1,0 %
	6,3 dn: 1,0 %
	7,3 dn: 2,8 %
	8,4 dn: 3,3 %
	9,5 dn: 17,1 %
	10,8 dn: 12,3 %
	12,1 dn: 31,7 %
	13,4 dn: 9,9 %
	14,9 dn: 10,9 %
	16,4 dn: 3,3 %
	18 dn: 2,8 %
	19,7 dn: 2,4 %
	21,5 dn: 1,0 %
	25,2 dn: 0,5 %
Average Fiber Fineness: 12 denier	
Number of fiber tested: 611	

Table 5.9 : Fiber Physical Test Results of Sample F- RPET 6 denier
(Yılmaz Textile)

Fiber Fineness (denier)	Distribution %
Fiber Length: 62,4 mm	**Denier**
Crimp no: 4/1 cm	0,6 dn: 0,5 %
	1,3 dn: 1,3 %
	2,4 dn: 5,1 %
	3,7 dn: 15 %
	4,5 dn: 14 %
	5,4 dn: 25 %
	6,3 dn: 11,4 %
	7,3 dn: 17,0 %
	8,4 dn: 15,4 %
	9,5 dn: 4,8 %
	10,8 dn: 0,8 %
	12,1 dn: 0,5 %
Average Fiber Fineness: 6 denier	
Number of fiber tested: 605	

Table 5.10 : Fiber Physical Test Results of Sample G -Bicomponent PET.

Fiber Fineness (denier)	Distribution %
Fiber Length: 48,02 mm	Denier
Crimp no: 3/1 cm	1,3 dn: 1,3 %
	2,4 dn: 32,2 %
	3,7 dn: 48,6 %
	4,5 dn: 3,2 %
	5,4 dn: 7,5 %
	6,3 dn: 3,4 %
	7,3 dn: 2,8 %
	8,4 dn: 0,6 %
	9,5 dn: 0,4 %
Average Fiber Fineness: 4,6 denier	
Number of fiber tested: 465	

Table 5.11 : Physical Test Results of Samples.

SAMPLE CODE	FIBER LENGTH	FIBER FINENESS	CRIMP
SAMPLE A- VPET 12 denier (Advansa)	57 mm	11.9 denier	3/1 cm.
SAMPLE B- RPET 11 Denier (Green Fiber)	63 mm.	10.2 denier	4/ 1 cm.
SAMPLE C- RPET 6 Denier (Green Fiber)	57.5 mm	6.1 denier	5/ 1 cm.
SAMPLE D- RPET 11 Denier (DS Fiber)	60.22 mm	10.65 denier	3.6/1 cm.
SAMPLE E- RPET 12 denier (Yılmaz Textile)	63.4 mm	12 denier	4/1 cm.
SAMPLE F- RPET 6 denier (Yılmaz Textile)	62.4 mm	6 denier	4/ 1 cm.
SAMPLE G -Bicomponent PET (Spin Global)	48.02 mm	4.6 denier	3/1 cm.

According to physical properties of fiber samples;

Sample B- RPET 11 Denier (Green Fiber) and sample E- RPET 12 denier (Yılmaz Textile) have the highest fiber staple lengths. They have longer fiber staples than the the same denier VPET (sample A-12 denier Advansa) and another RPET supplied from DS Fiber.

The comparasion between two 6-denier RPETs shows that;

Although sample F- RPET 6 denier (Yılmaz Textile) has higher fibre staple length, sample C- RPET 6 Denier (Green Fiber) has more crimp on the fiber.

Sample A-VPET (Advansa) and sample G-Bicomponent PET (Spin Global) has the lowest crimp numbers among others.

5.4. Breaking Strength and Breaking Elongation Test Results

The breaking strength and breaking elongation values of the fiber samples are given in Table 5.12 and 5.13.

Table 5.12 : Strength values of samples.

SAMPLE CODE	Strength (gf)	Standard Deviation (s)	CV %	Tenacity (g/denier)
SAMPLE A- VPET (Advansa)	53,05 gf	5,57	10,49	4,45
SAMPLE B- RPET 11 Denier (Green Fiber)	39,83 gf	4,69	11,77	3,90
SAMPLE C- RPET 6 Denier (Green Fiber)	21,46 gf	3,2	14,91	3,51
SAMPLE D- RPET 11 Denier (DS Fiber)	33,76 gf	4,01	11,87	3,17
SAMPLE E- RPET 12 denier (Yılmaz Textile)	39,99 gf	5,81	14,52	3,33
SAMPLE F- RPET 6 denier (Yılmaz Textile)	22,71 gf	3,5	15,41	3,78
SAMPLE G -Bicomponent PET (Spin Global)	15,38 gf	1,22	7,93	3,34

Table 5.13 : Elongation values of samples.

SAMPLE CODE	Elongation %	Standard Deviation (s)	CV %
SAMPLE A- VPET (Advansa)	51,49	10,87	21,11
SAMPLE B- RPET 11 Denier (Green Fiber)	77,42	20,23	26,13
SAMPLE C- RPET 6 Denier (Green Fiber)	85,33	16,32	19,12
SAMPLE D- RPET 11 Denier (DS Fiber)	80,62	19,76	24,51
SAMPLE E- RPET 12 denier (Yılmaz Textile)	77,91	9,43	12,1
SAMPLE F- RPET 6 denier (Yılmaz Textile)	64,43	16,74	25,98
SAMPLE G -Bicomponent PET (Spin Global)	62,38	21,95	35,18

In accordance to comparasion of tensile properties of fiber samples, all fiber samples have the tenacity over 3 g/denier. Sample A- VPET (Advansa) has the highest tenacity value and lowest breaking elongation value among the fiber samples.

In the literature (Hearle et al, 1995), regular tenacity values of staple PET fibers range between 0,35-0,47 N/tex (3,95- 5,3 g/denier). Tenacity value of Sample A, which is 4,45 is between the normal values as expected, however tenacity values of recycled PET samples are lower than expected values.

As seen from the comparasion, RPETs and virgin PET have different mechanical properties however recycled PET fibers shows similar properties.

According to literature (Hearle *et al*, 1995), an increase in crystallinity and molecular orientation yields an increase in tensile strength, a reduction of extensibility and elongation. Reduction of tensile strength values of recycled PET samples yields higher elongation degrees. Regular breaking elongation values of staple PET fiber range between 35-60 %. As seen from the Table 5.13, all recycled PET samples are higher elongation degrees than expected values. As a result, the virgin fibers have higher tenacity and lower breaking elongation values than recycled PET fibers have.

5.5 Dynamic Mechanical Analysis (DMA)

For DMA measurement tests of samples E-RPET 12 denier (Yılmaz Textile) and sample F-RPET 6 denier (Yılmaz Textile) were carried out at Kordsa Global. The test results of these samples were measured and plotted on Figure 5.9 and 5.10. The test value of a virgin PET fiber produced by Kordsa was also recorded and plotted on the same figure. Figure 5.8, 5.9, 5.10 represent plot of E', E'' and tan δ of these PET fiber samples.
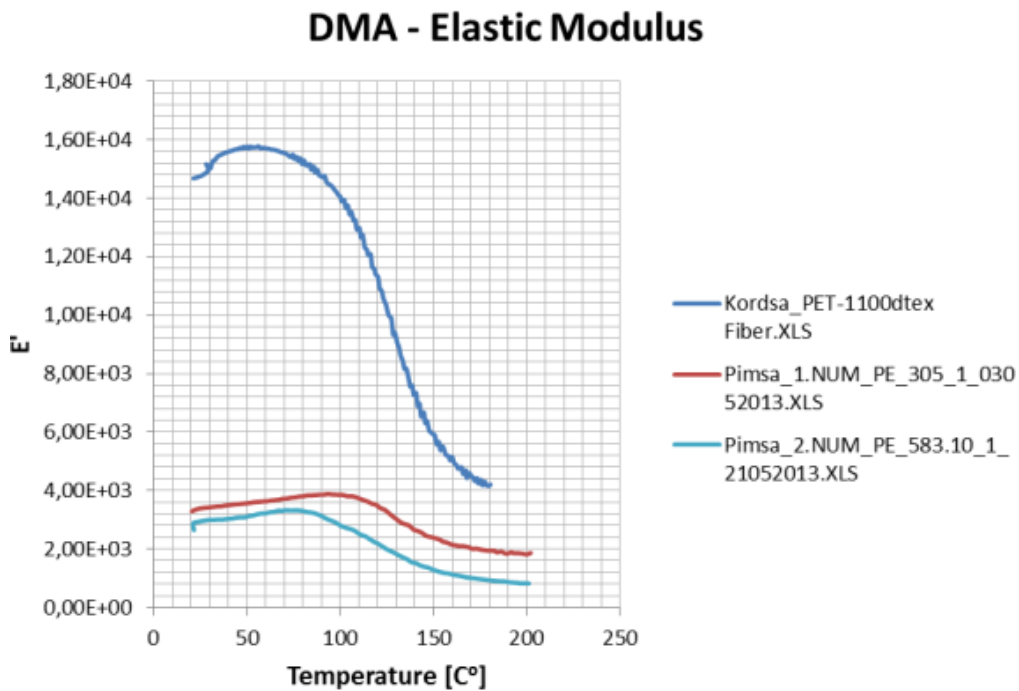
Figure 5.8 : Elastic modulus (E') of Sample E, Sample F and Kordsa 1100 dtex PET.
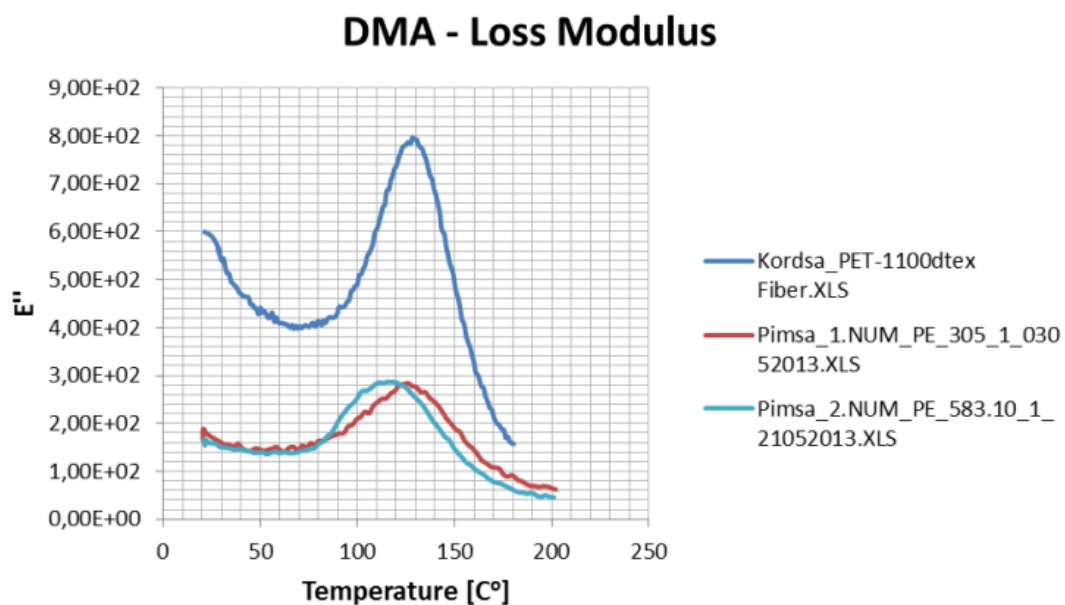
Figure 5.9 : Loss modulus (E'') of Sample E, Sample F and Kordsa 1100 dtex PET.
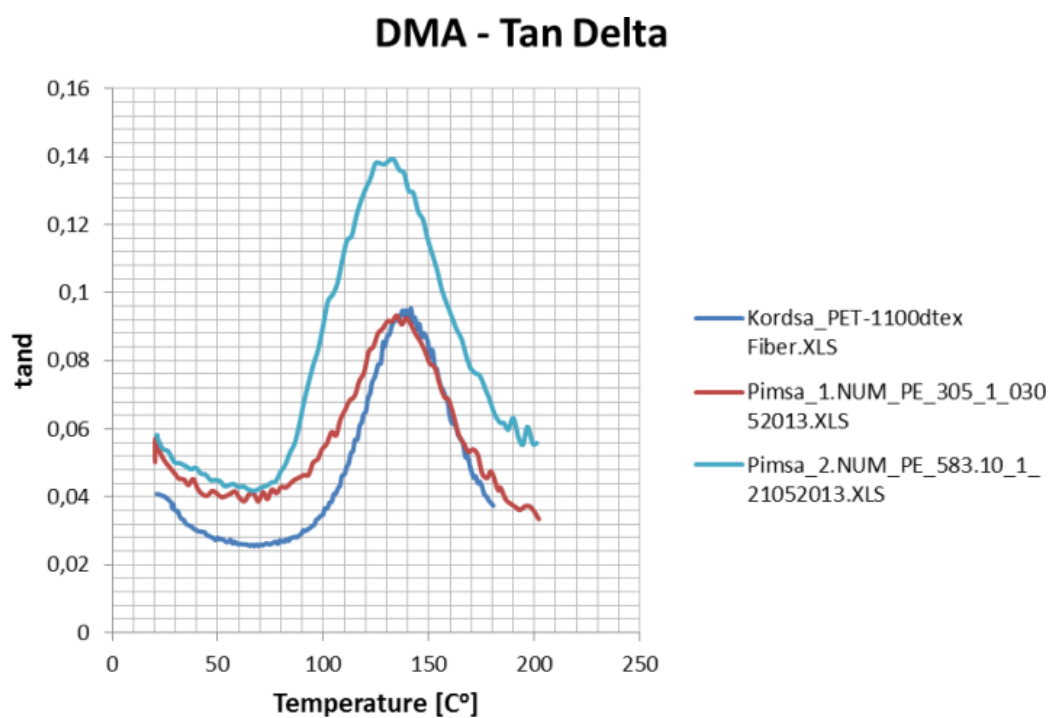
Figure 5.10 : Tan Delta (δ) of Sample E, Sample F and Kordsa 1100 dtex PET.

According to DMA results;

As seen from the Figure 5.8, elastic modulus of 1100 dtex virgin PET gradually decrease with temperature and rapid increase in E' is observed above 50°C. 1100 dtex virgin PET shows the highest value in the whole range of temperature. There is a significant difference between elastic modulus of virgin and RRET. This means that the PET has a rapid reduction on elastic modulus after recycling process.

From the loss modulus data (Figure 5.9), the peaks E'' of all samples appear at around 125°C which is attributed to the relaxation transition peak (α-relaxation). Therefore, α-relaxation is associated with the chain segment mobility in the crystalline phases, which is probably due to reorientation of defect areas in the crystals. Loss modulus (E'') of virgin PET is clearly higher than those RPETs, respectively.

These elastic modulus and loss modulus results are parallel to the differences on crystallinity rates of RPET and PET fibers.

The ratio of loss modulus to storage modulus is measured as the mechanical loss factor or tan δ. The damping properties of material give the balance between the elastic phase and viscous phase in a polymeric structure (Kayaisang et al, 2012). In the present investigation, the variation of tan δ as a function of temperature is presented (Figure 5.10). Tan δ peaks of all samples appear at around 135°C. At the temperatures below 135°, all samples have a gradually increase, at the temperatures above 135°C, samples have a gradually decrease on tan δ. In our case, Virgin PET shows to have slightly lower storage modulus than RPET fibers. This test result shows that virgin PET fiber sample from Kordsa tends to dissipate more energy than recycled PET fibers used in the study.

5.6. Scanning Electron Microscopy (SEM) Analysis

SEM micrographs of the samples are given in Figures between 5.11 and 5.45.
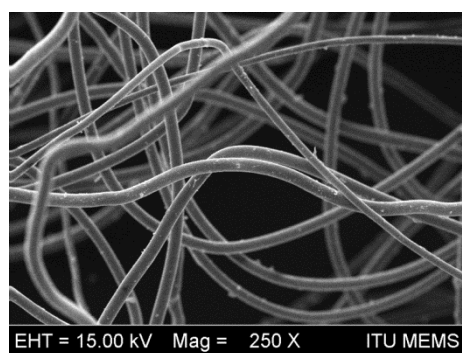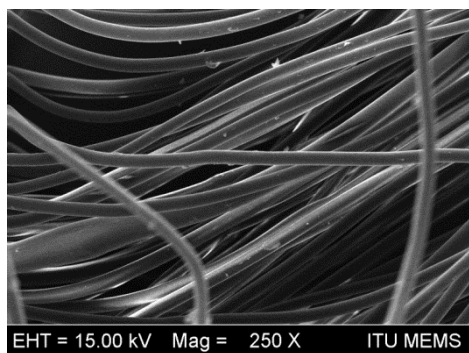
Figure 5.11 : SEM of Sample A (250x).**Figure 5.12 :** SEM of Sample B (250x).
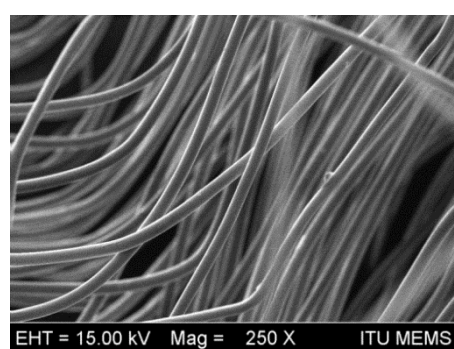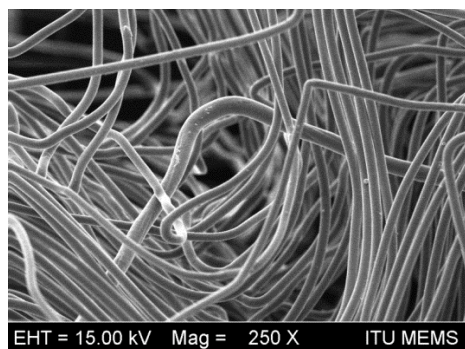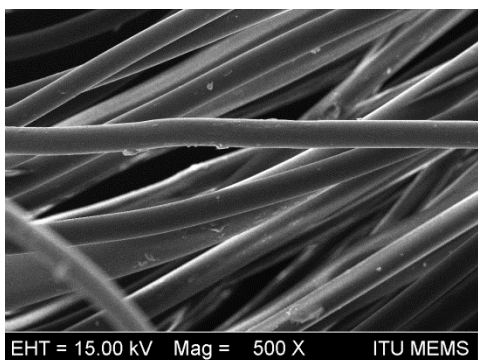

Figure 5.13 : SEM of Sample C (250x). **Figure 5.14 :** SEM of Sample D (250x).
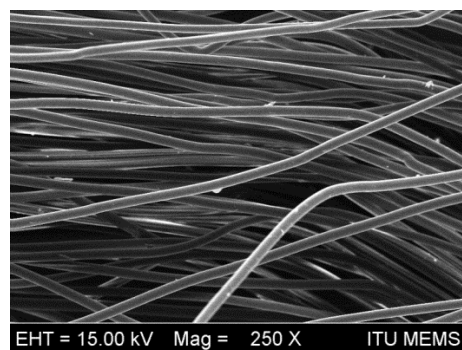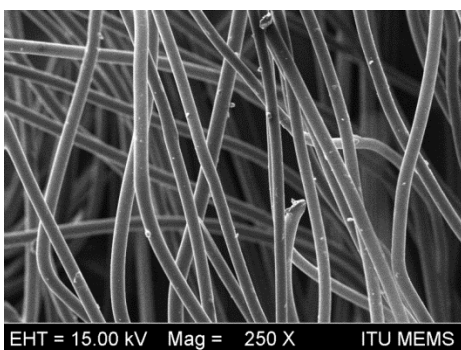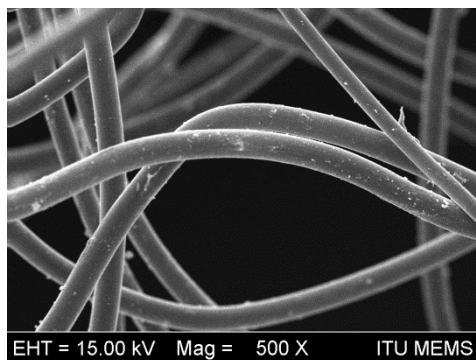

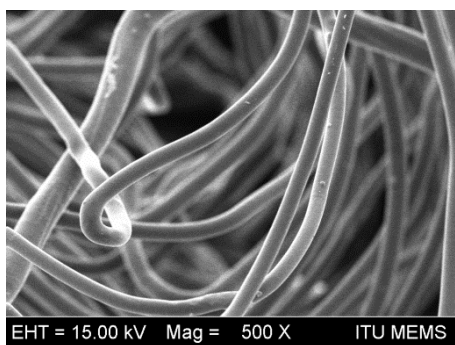**Figure 5.15 :** SEM of Sample E (250x). **Figure 5.16 :** SEM of Sample F (250x).
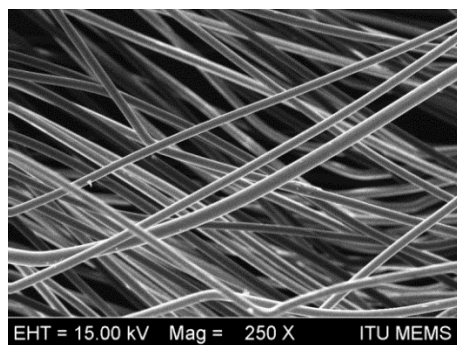
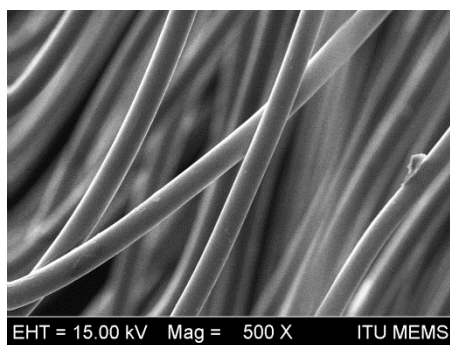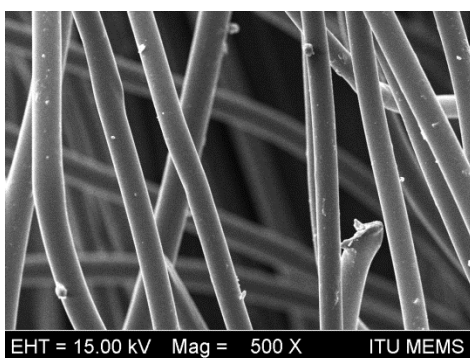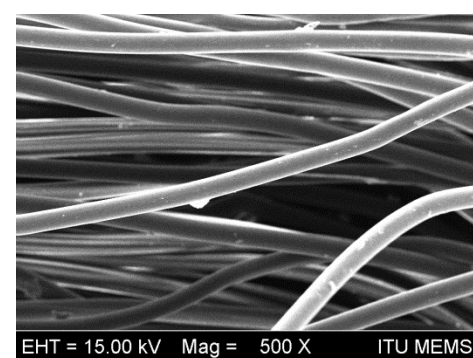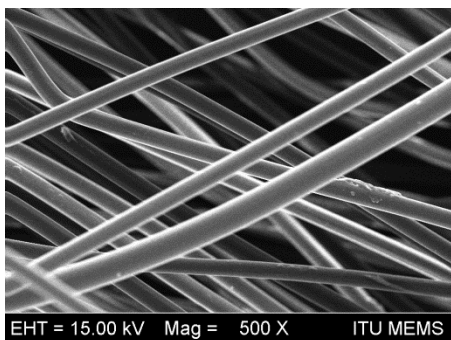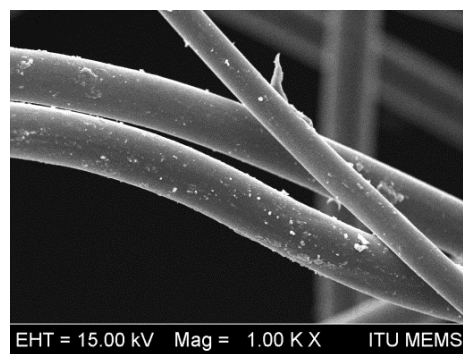**Figure 5.17 :** SEM of Sample G (250x).

Figure 5.18 : SEM of Sample A (500x).   **Figure 5.19 :** SEM of Sample B (500x).

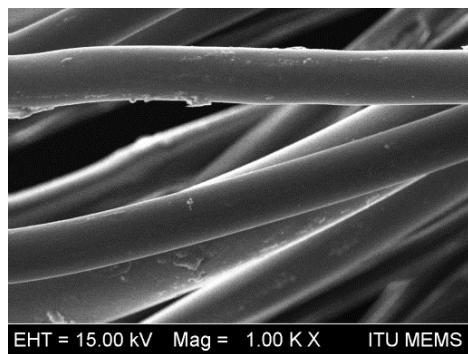**Figure 5.20 :** SEM of Sample C (500x).   **Figure 5.21 :** SEM of Sample D (500x).

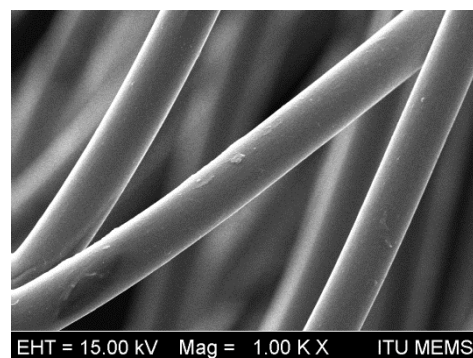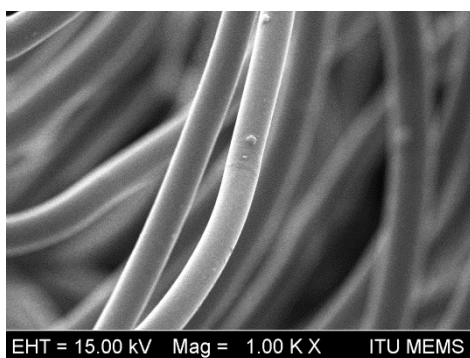**Figure 5.22 :** SEM of Sample E (500x).   **Figure 5.23 :** SEM of Sample F (500x).
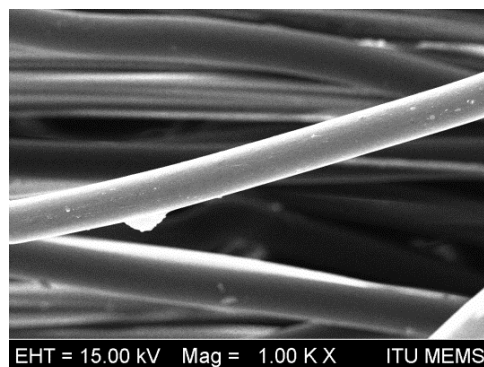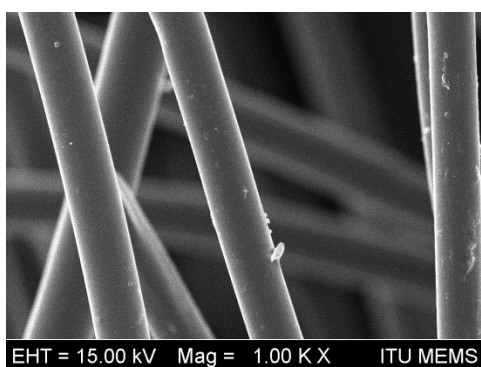

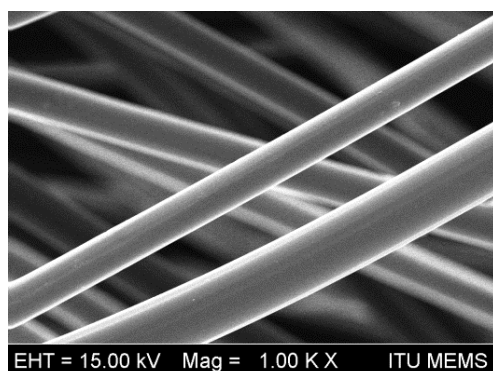**Figure 5.24 :** SEM of Sample G (500x).

Figure 5.25 : SEM of Sample A (1000x). **Figure 5.26 :** SEM of Sample B (1000x).
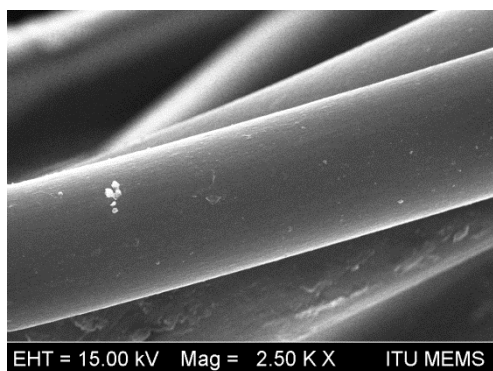

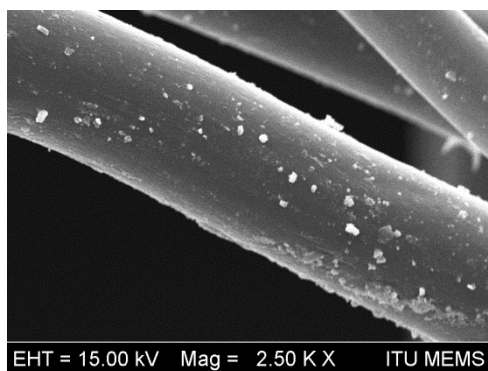**Figure 5.27 :** SEM of Sample C (1000x). **Figure 5.28 :** SEM of Sample D (1000x).

Figure 5.29 : SEM of Sample E (1000x). **Figure 5.30 :** SEM of Sample F (1000x).
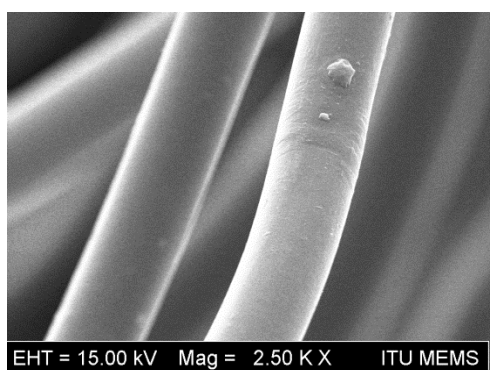

Figure 5.31 : SEM of Sample G (1000x).

Figure 5.32 : SEM of Sample A (2500x). **Figure 5.33 :** SEM of Sample B (2500x).

Figure 5.34 : SEM of Sample C (2500x). **Figure 5.35 :** SEM of Sample D (2500x).

Figure 5.36 : SEM of Sample E (2500x). **Figure 5.37 :** SEM of Sample F (2500x).

Figure 5.38 : SEM of Sample G (2500x).

Figure 5.39 : SEM of Sample A (5000x). **Figure 5.40 :** SEM of Sample B (5000x).

Figure 5.41 : SEM of Sample C (5000x). **Figure 5.42 :** SEM of Sample D (5000x).

Figure 5.43 : SEM of Sample E (5000x). **Figure 5.44 :** SEM of Sample F (5000x).

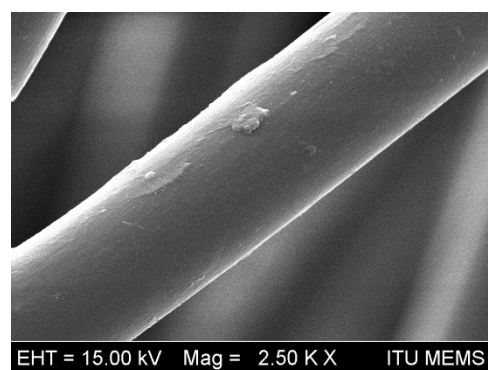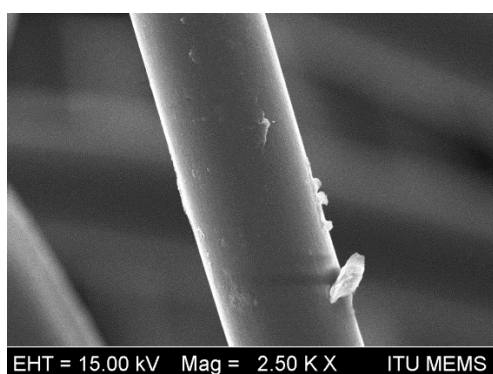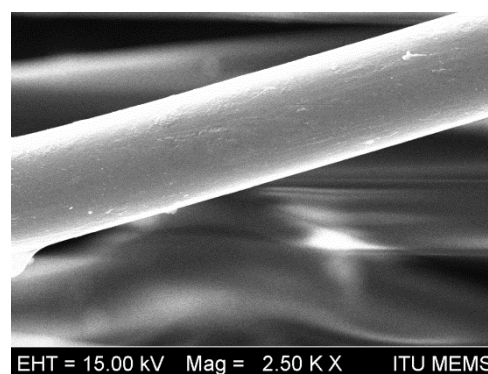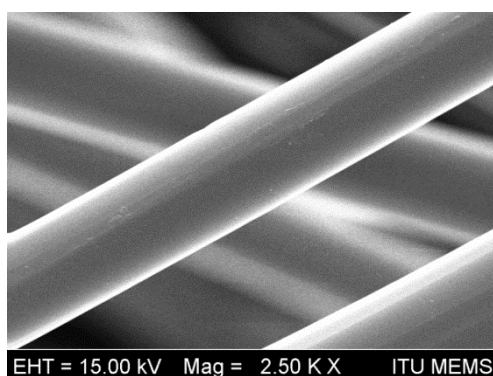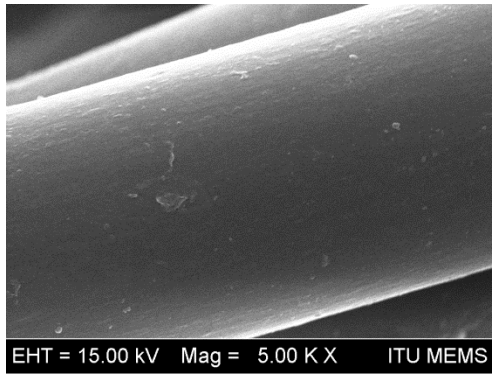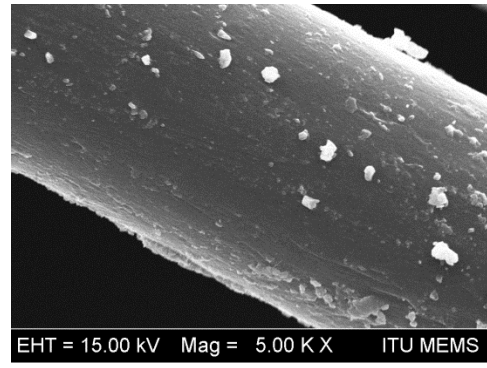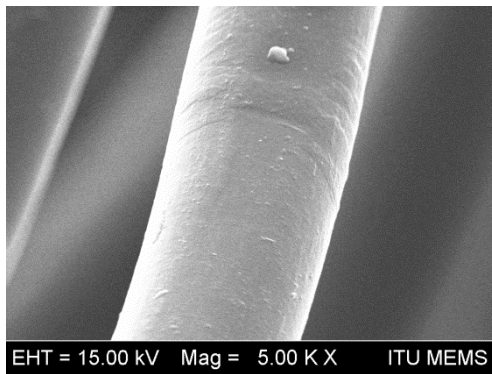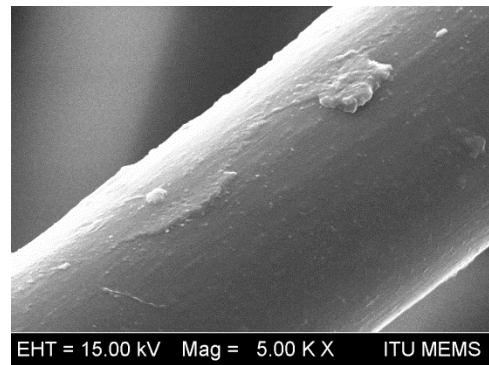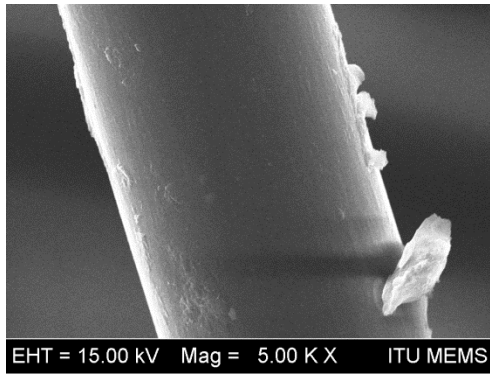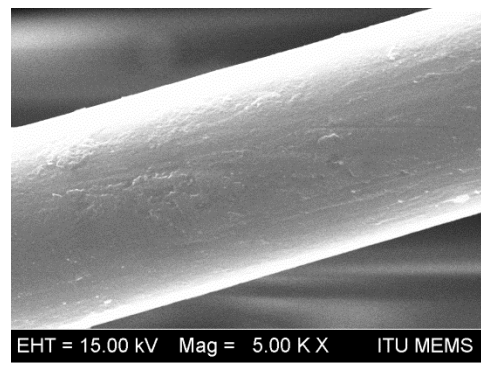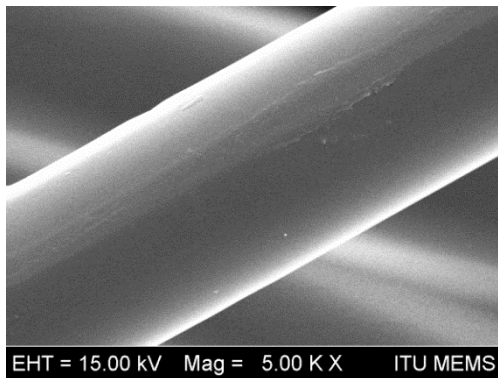**Figure 5.45 :** SEM of Sample F (5000x).

Surface characterization of fibers samples were clearly exhibited by SEM micropgraphs especially with 2500 and 5000 magnifications. According to SEM micrographs, Sample G- Bicomponent fiber has the smoothest surface, which is followed by sample-A which is another virgin fiber among fiber samples.

SEM micrograph of sample F, which is a recycled PET fiber supplied from Yılmaz Textile, can be seen in Figure 5.44 with 5000 magnification, has also comparable surface characterization properties with sample A and sample G that are virgin PET fibers. However, SEM micrograph of sample B, which is a recycled PET fiber supplied from Green Fiber, has a rougher surface.

All fiber samples except sample G-bicomponent virgin fiber, have some little particles on the surfaces which might be arised due to spin finishing.These particles that might be also lubricants or dust, do not cause the morphological defects on the surfaces.

As seen from the SEM micrographs with 5000 magnification, all the surfaces seem smooth. There are almost no significant differences on surface characteristics among fiber samples, which could be an effect on the quality of novel product. Because, only significant morphological defects on fiber surfaces may affect the processability of recycled PET fibers.

5.7 Discussion of Fiber Tests and Production Results

As a part of our research on possibility of using recycled PET fibers instead of virgin PET fibers for the production of needlepunched automobile carpets, various fiber samples supplied from different producers of virgin and recycled PET fibers were tested and characterized in order to select the most appropriate fiber or fiber blend for needlepunched car carpet production. All fiber test results are exhibited in Table 5.14.

For a nonwoven carpet quality, mechanical properties such as tensile strength, young modulus at break are critical. Fiber fineness, stiffness and elongation values are also very significant in terms of influencing entanglement of fibers in the product structure. In order to achieve required quality and to comform specification requirements as a nonwoven product, fibers as raw materials must have at least minimum levels of mechanical properties.

Table 5.14 : Fiber Test Results.

	SAMPLE CODE						
	Sample	Sample	Sample	Sample	Sample	Sample	Sample
FIBER TEST	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Finer Fineness (denier)	11,9	10,2	6,1	10,65	12	6	4,6
Staple Length (mm)	57	63	57,5	60,22	63,4	62,4	48,2
Crimp (no/1 cm)	3	4	5	3,6	4	4	3
Int.Viscosity (dl/g)	0,635	0,585	not solved	not solved	0,523	0,529	0,627
Melting Point (°C)	259,77	253,63	252,24	252,07	253,32	253,7	72-255,71
Crystallinity %	41	not recorded	43	36	41	38	16
Strength (gf)	53,05	39,83	21,46	33,76	39,99	22,71	15,38
Tenacity (g/den)	4,45	3,9	3,51	3,17	3,33	3,78	3,34
Elongation (%)	51,49	77,42	85,83	80,62	77,91	64,43	62,38

As seen from the Table 5.14, virgin PET and recycled PET have differences in their mechanical properties because of the difference in crystallinity, molecular orientation, intrinsic viscosity (consequently molecular weight). Sample A-virgin PET has the highest tenacity, lowest elongation values.

Melting points, intrinsic viscosity (consequently molecular weigh) and degree of crystallinity values are all parallel to each other. Higher melting temperature, higher intrinsic viscosity and higher crystallization rate yield higher mechanical properties. In contrast, higher tenacity values indicate lower elongation, which might be a negatif contribution to the quality of needlepunched carpet structures in terms of entanglement.

After recycling process, recycled PET has a reduction in intrinsic viscosity and molecular weight that leads to a decrease in mechanical properties. Despite this reduction, properties of recycled fibers are still close to virgin PET properties.

At the end of several production trials in the factory, the most efficient fiber or fiber blends were evaluated in accordance to OEMs specification requirements in order to acquire comparable quality levels of car carpets. Table 5.15 exhibits specification conformations of production samples.

Table 5.15 : Spesification conformation of needlepunhed carpets produced from different fiber blends.

Sample No	Fibre Blend Combination	Spesification conformation
Sample 1	VPET 12 denier (Advansa) + 5dtex Bico PET	PASS
Sample 2	VPET 12 denier (Advansa)	FAIL
Sample 3	RPET 12 denier (Yılmaz Textile)	FAIL
Sample 4	RPET 12 denier (Yılmaz Textile) + 5dtex Bico PET	FAIL
Sample 5	RPET 12 denier (Yılmaz Textile) + RPET 6 denier (Green Fiber) + 5dtex Bico PET	FAIL
Sample 6	RPET 12 denier (Yılmaz Textile) + RPET 6 denier (Green Fiber) + 5dtex Bico PET	PASS
Sample 7	RPET 6 denier (Yılmaz Textile)	FAIL
Sample 8	RPET 11 denier (Green Fiber) + 5dtex Bico PET	FAIL

As seen from the Table 5.15, in the production, Sample 1, which has virgin PET and bicomponent PET in the structure, and sample 6, which consists of a recycled PET blend and bicomponent PET have passed the strict spesification requirements set by OEMs. A combination of 6-denier and 11-denier recycled PET with a bico PET achieve the same quality with virgin PET.

6. CONCLUSIONS AND RECOMMENDATIONS

Recycling and reusing RPET has been a topic of interest over the last few years. Loss in properties of recycled PET has been a concern and RPET has been considered secondary to virgin PET, however environmental concerns have also made the use of these materials essential.

In order to investigate appropriate recycled PET fibers or fiber blends as raw materials to be used in the production of needlepunched car carpets, virgin PET and recycled PET fibers supplied from different producers were tested. Several different production trials were made and using a combination of 6-denier RPET, 11-denier RPET and bicomponent PET fiber in the production of needlepunched car carpet found to be an alternative to using a combination of virgin PET and bicomponent PET by conforming high quality standards.

This study shows that;

- 6-denier RPET is extremely fine to achieve required performance of the needlepunched car carpet.
- A combination of 12-denier virgin PET and 5-dtex bicomponent PET is the best combination among the tested fiber blends for achieving the desired quality and specifications of needlepunched car carpet.
- Use of bicomponent fibers in the fiber blends also provides a benefical effect on the needlepunched automobile carpet properties due to provide bonding in the production.
- Use of 6-denier recycled PET and 11-denier recycled PET blend shows to combine the positive effects of both fibers; 6-denier recycled PET provides better entanglement in the carpet structure, while 11-denier recycled PET contributes to physical performance of needlepunched car carpets.
- Although virgin PET fibers have advantages with higher tenacity grade, recycled PET fibers also have benefits due to their high elongation degrees, which lead better entanglement, consequently better quality needlepunched carpets.

- Using recycled PET in the production of needlepunch car carpets is possible and this could be a cost-effective option.

This study also shows that equal or comparable quality levels could be achieved using RPET fibers instead of virgin PET fibers. Use of recycled PET offers considerable energy and cost savings. The results obtained in the study encourage use of recycled PET fibers not only in automobile carpet applications, but also for other industrial applications on a large scale. Increased use of them will have a positive impact on the environment in the long term.

As further study, use of recycled PET fibers in other automotive components can be studied. The effect of fiber properties on the final performance of these products can be investigated. The acoustic performance of these fibers can be compared to that of virgin PET fibers.

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PUBLICATIONS/PRESENTATIONS ON THE THESIS

R. Atakan, H. Karakaş, "Use of Recycled Poly(ethylene terephthalate) Fibers in Needlepunched Automotive Carpet Composite Structures", has been accepted for oral presentation by *Mech. Comp. 2014 Organisation Congress,* Long Island, New York, USA.