12-2016

Development of Composites from Waste PET - Cotton Textiles

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DEVELOPMENT OF COMPOSITES FROM WASTE PET - COTTON TEXTILES

by

MADHURI PALAKURTHI

A THESIS

Presented to the Faculty of
The Graduate College at the University of Nebraska
In Partial Fulfillment of Requirements
For the Degree of Master of Science

Major: Textiles, Merchandising and Fashion Design

Under the Supervision of Professor Yiqi Yang

Lincoln, Nebraska
December, 2016
DEVELOPMENT OF COMPOSITE FROM WASTE PET – COTTON TEXTILES

Madhuri Palakurthi, M.S
University of Nebraska, 2016

Advisor: Yiqi Yang

In this research, waste textile materials like polyethylene terephthalate (PET) and cotton were compression molded into composites. The main idea was to use waste textiles to develop composites like PET and cotton. PET acts as matrix and cotton as reinforcement because PET is thermoplastic whereas cotton is non-thermoplastic.

Approximately about 21 million tons of textile solid waste is being disposed into landfills annually, leading to vast environmental properties and waste of valuable raw materials. By using waste PET and cotton textiles there are technical as well as environmental viabilities. However, implementing them into fabricating composites is not widely conducted.

Compression molding is a feasible approach to use waste PET and cotton textiles into fabricating composites that have good potential for industrial applications. In this research, investigating the effects of plasticizers and alkalis on decreasing the processing temperatures of composites was studied so that the cotton is protected during compression molding. In the following paper, it is shown how plasticizers can be effectively used to decrease the melting temperature of PET. The influences of chemicals on the melting temperature of PET and the mechanical properties of the composites are investigated. In my future research, the feasibility of compression molding PET and cotton fabrics into composites with other chemicals will be studied. More molecular
characterizations of individual PET and cotton components plus static and dynamic mechanical characterizations of composites will be conducted.
Dedicated to

My parents - Mr. Surender & Mrs. Sravani

Uncle and Aunt – Mr. Giri & Mrs. Shanthi
ACKNOWLEDGEMENT

First and foremost, I would like to thank my advisor, Professor Yiqi Yang, for accepting me in his team. Without his constant moral support, this research would not have been completed. I would like to express my deepest thanks and sincere appreciation to Professor Helan Xu for her generous advice, kind endless help, and support during the study. I would like to express my gratitude to Professor Rita Kean for her encouragement through my graduate study experience in this department. I’m grateful for my textile science team especially Zhuanzhuan Ma, Yi Zhao and Gangwei Pan for helping me through my research.

I would like to acknowledge National Institute of Food and Agriculture (Multi-State Project S1054 (NEB 37-037)), USDA Hatch Act and Agricultural Research Division at University of Nebraska – Lincoln for giving me this opportunity to work on this research. My graduate study experience at Department of Textiles, Merchandising and Fashion Design benefitted greatly from the courses I took and various opportunities explored.

Finally, I would like to acknowledge my family Mom (Sravani), Dad (Surender), Uncle (Giri), Aunt (Shantha) and friends Lalitha, Suman, Shiva, Aditya, Vikas, Niruktha, Gayathri, Pooja for providing me with unfailing love, support and unlimited encouragement throughout my research.

Author
Madhuri Palakurthi
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<th>Abbreviations</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>PET</td>
<td>Polyethylene Terephthalate</td>
</tr>
<tr>
<td>PP</td>
<td>Polypropylene</td>
</tr>
<tr>
<td>2PP</td>
<td>2 phenyl phenol</td>
</tr>
<tr>
<td>BBP</td>
<td>Benzyl butyl phthalate</td>
</tr>
<tr>
<td>DAP</td>
<td>Diallyl phthalate</td>
</tr>
<tr>
<td>BA</td>
<td>Benzoic acid</td>
</tr>
<tr>
<td>MPa</td>
<td>Mega pascal</td>
</tr>
<tr>
<td>DP</td>
<td>Degree of Polymerization</td>
</tr>
<tr>
<td>TMAH</td>
<td>Tetra methyl ammonium hydroxide</td>
</tr>
<tr>
<td>DMSO</td>
<td>Dimethyl sulfoxide</td>
</tr>
</tbody>
</table>
Chapter 1: INTRODUCTION

Nowadays, the scope of development of composites from waste textiles paved a way for increased research activities. The main purposes of using waste textiles are:

1) Textile waste is cheaper and profitable.
2) Most modern textile products are from synthetic materials, recycling this waste to developing new composites can be environment-friendly.

Waste textiles are having a greater impact on the environment as they are being disposed of in the landfill which takes a very long time to decompose. Materials manufactured from non-renewable petroleum extracts tend to release gases while decomposing which is hazardous to nature causing atmospheric pollution. According to the United States environmental protection, and council of textile recycling, it is estimated that 8.7% of the total solid waste in 2012 is from textiles, which is calculated as approximately 21 million tons. The Secondary Materials and Recycled Textiles Association estimated that 10.5 million tons of textiles are sent to landfills every year. Solid textile waste mainly comprises of natural and synthetics. It is estimated that fabric form blended with other types of materials comprises of about 73% of the synthetics. In this case, it is difficult to reuse or separate from other materials like blends, dyes etc.

Figure 1.1: WASTE ANALYSIS
1.1 PET WASTE:

Polyethylene terephthalate (PET) is usually the major contributor to the textile industry which is derived from non-renewable petroleum resources. It is estimated that about 13-14 million tons of PET were being produced as waste annually in 2012. The biggest disadvantage of PET is that it takes a large amount of time for decomposition and degradation when it is disposed. PET waste can be reused, which helps in reducing environmental pollution and saving valuable resources used in producing new PET. There are the different ways of effectively using PET:

**COMBUSTION/ INCINERATION:**

It is a procedure used to generate energy from waste materials. By this process, there is a scope of using about 80 – 90 percent of volume and 70 – 75 percent in weight of waste being utilized.

The main disadvantage with this procedure is that the combustion process releases dangerous gases into the environment, thereby causing environmental pollution.

**DISCARD VIA LANDFILLS:**

It is a major process used for getting rid of textile wastes which are calculated to be about 90% of the total solid waste. In this method, the textile wastes are disposed into landfills and allowed to degrade. Textiles comprising of synthetics or polymers take a relatively large amount of time to degrade or decompose. This process involves many disadvantages which are release of toxic chemicals, takes long time to decompose and waste of valuable polymers.
RECYCLING:

It is estimated that only 7.5 – 10% of waste PET is successfully being recycled. The main idea of this method is to depolymerize PET using the techniques of hydrolysis, methanolysis, and ammonolysis.

**Hydrolysis:** This procedure involves the chemical breakdown of a compound due to reaction with water.

**Ammonolysis:** This procedure is similar to hydrolysis in which ammonia reacts with other compounds usually to form an amine.

**Methanolysis:** This procedure is used to produce biodiesel by the displacement of alcohol from an ester by another alcohol like methanol, so the process also called as called transesterification or alcoholysis. Disadvantages involved in this method are high cost, high energy consumption and complicated procedure.

REUSING:

It is a common process in practice. In this process, PET is melted and then processed into new fibers and nonwovens. It is considered as a good method overall but has some disadvantages i.e., it is hard to remove the color from the PET.
1.2 COTTON WASTE:

Cotton waste is estimated to be 4 – 5 million tons annually. Cotton waste is a multiuse product which can be reused even after being disposed. Cotton waste is biodegradable and can be used in constructions, medical fields, and industrial purposes.

Cotton waste is also sold widely in large quantities. Here is a table which gives us the cost of various types of waste cotton being sold worldwide. Usually, the bulk amount varies between 50,000 kilograms to 100,000 kilograms.

**TABLE 1.1: COST OF COTTON WASTE**

<table>
<thead>
<tr>
<th>CATEGORIES OF COTTON</th>
<th>COST</th>
</tr>
</thead>
<tbody>
<tr>
<td>White cotton waste</td>
<td>$ 0.5 - $ 0.7</td>
</tr>
<tr>
<td>Colored cotton waste</td>
<td>$ 0.17 - $ 0.25</td>
</tr>
<tr>
<td>Mixed cotton waste</td>
<td>$0.10 - $0.15</td>
</tr>
<tr>
<td>Cotton threads and fiber waste</td>
<td>$0.10 - $0.13</td>
</tr>
</tbody>
</table>

**TABLE 1.2: PROPERTIES OF PET AND COTTON**

<table>
<thead>
<tr>
<th></th>
<th>PET</th>
<th>COTTON</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>THERMAL BEHAVIOUR (°C)</strong></td>
<td>260</td>
<td>149; 246</td>
</tr>
<tr>
<td><strong>STRENGTH (g/denier)</strong></td>
<td>2.5 to 9.5</td>
<td>Dry - 3.0 to 4.9</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Wet - 3.3 to 6.0</td>
</tr>
<tr>
<td><strong>ELONGATION AT BREAK (%)</strong></td>
<td>15 – 20</td>
<td>8 – 10</td>
</tr>
<tr>
<td><strong>LENGTH (mm)</strong></td>
<td>32 – 102 (38)</td>
<td>22 – 31</td>
</tr>
<tr>
<td><strong>SPECIFIC GRAVITY( g/cm³)</strong></td>
<td>1.38</td>
<td>1.54</td>
</tr>
<tr>
<td><strong>MOISTURE REGAIN (%)</strong></td>
<td>0 – 0.4</td>
<td>7.0 – 8.5</td>
</tr>
</tbody>
</table>
Studies suggest that about one-third of PET earmarked for the textile industry is being used to produce staple fibers, which are mostly blended with cotton fibers to make PET/cotton blend fabrics. PET/cotton blended fabrics are widely used in the manufacturing of garments, home furnishings and other very commonly used household textile products. Disposal of these PET/cotton blended fabrics is a grave environmental concern due to polymer going into debris.

Unfortunately, the physical and chemical techniques of recycling pure PET are not conclusive for recovering PET from PET/cotton blended fabrics. PET in blended fabrics is intricately mixed with cotton fibers and cannot be separated mechanically. Moreover, dissolving PET from the blends is not economically feasible since PET has limited solvents that are very expensive and to make things worse, PET/cotton fabrics contain plastic and metallic accessories like buttons and zippers, which cannot be easily separated. Finally, the process of removing dyes (color) from PET is very complicated and tedious.

There is a sustained and increasing pressure from environmental activists to safeguard and preserve invaluable natural resources. Several nations paved a way for new inventions and development for developing composites. Characteristics of PET and cotton are having potential in developing the composites. PET is thermoplastic, lighter in weight and has high strength. Cotton is non-thermoplastic and easily biodegradable.

Composites are materials made up of two or more dissimilar materials, which when combined are stronger than their respective individual materials, as they usually take up the desired characteristics of the individual materials. Composites can be easily found in our daily lives. Wood is a classic example of a composite.
Composites are gradually replacing the conventional materials in various fields ranging from appliances to space crafts, sporting goods to building industries due to their low cost, eco-friendliness, and easy processing.

Stiffness, thermal expansion, strength and flexural properties of composites can vary over a broad range depending on the fiber, resin and fabrication mechanisms during manufacture. Nowadays, the popular textiles materials in composites are glass fiber, aramid, and carbon fiber because these reinforcement fibers give composites high strength and stiffness.

1.3 PHASES OF COMPOSITES

Composites generally have two phases. They are:

MATRIX PHASE:

It is a structural constituent, which determines the internal structure of composites. The Matrix phase may contain a metal, a ceramic or a polymer.

REINFORCEMENT PHASE:

This phase is connected to the matrix phase by bonding. It adds rigidity to the composites. Common fibers used for reinforcement include glass fibers, carbon fibers, cellulose (wood/paper fiber & straw) and high strength polymers.

Polymers are cheap and can be easily fabricated into composites. Cellulose is easily available and has good mechanical properties.

Textile industries mainly use polymers which are usually 60 – 70% and the remaining is from cellulose which is apparently being wasted. Cellulosic fibers usually
take a large amount of time, more labor and resources for production. Hence, reusing the waste textiles will help in a significant saving of time, resources and will lead to an increase in revenues.

1.4 POLYMER MATRIX COMPOSITES:

Composites made with polymers are comprised of a variety of short or continuous fibers bound together by an organic polymer matrix.

Advantages of Polymer Matrix Composites are High tensile strength, High stiffness, High Fracture Toughness, Good abrasion resistance, Good puncture resistance, Good corrosion resistance and Low cost.

Disadvantages of Polymer Matrix Composites are low thermal resistance and high coefficient of thermal expansion.

1.5 CELLULOSE REINFORCED COMPOSITES:

Cellulose fibers have been used as reinforcing materials in combination with polymeric materials for over 3000 years. The price of natural fibers is $200-1000 per metric ton. Cellulose fibers are broadly classified into three types, depending on the part of the plant from which they are extracted. Like bast or stem fibers (jute, flax, hemp, ramie), leaf fibers (sisal, banana, manila, hemp, pineapple) and seed fibers (cotton, coir, oil palm)

Natural fiber-reinforced polymer composites represent one of today’s fastest growing industries. Natural fibers are gradually gaining significant importance in technical applications and are used as an alternative to manmade fibers such as carbon, glass or aramid.

Natural fibers have some of the special characteristics which cannot be found in
synthetic fibers, so by reinforcing polymers with natural fibers there is a scope for improvement in mechanical properties such as stiffness and strength. These mechanical properties differ from material to material depending on the source. Availability, price, and performance are main factors to be considered while using cellulosic fibers. Despite the variability in mechanical properties, natural fibers have more advantages than thermoplastics such as low specific weight and low production cost. But cellulosic fibers have a low thermal stability that results in the exclusion of some manufacturing processes and also limits the use of the composites to low temperature applications.

Composites from polymers have been gaining in popularity due to their inherent processing advantages. Among thermoplastics, polypropylene (PP) and polyethylene terephthalate (PET) have outstanding properties like low density, good flex life, good surface hardness, very good abrasion resistance and excellent electrical properties.

The composites prepared using cellulosic and thermoplastic materials are undergoing some problems like the incompatibility between polar - hygroscopic fiber and nonpolar - hydrophobic matrices.
Chapter 2: OBJECTIVES

Main objective is to develop composites from PET – cotton with good mechanical properties. The goal is to use plasticization and hydrolysis treatment to reduce the melting temperature of PET which further helps in ensuring better tensile stress, elongation and work of rupture. This study aims to form at composites at lower temperatures to ensure less damage to cotton fabrics.
Chapter 3: LITERATURE REVIEW

An intensive literature review aided me understand that there isn’t enough research being pursued in direction of reducing the melting point of PET in cotton composites. On the other hand, significant amount of work has been done in the development of composites from PET or cotton with polypropylene, HDPE, banana, kapok, etc.

In this research, our primary focus was on using waste textiles such as cotton and PET to prepare composites at a lower temperature with the intention of protecting the cotton properties by reducing melting temperature of PET, so that resulting composites have better mechanical properties.

Yi Zou et al (Yi Zou et al., 2011 - “Reusing polyester/cotton blend fabrics for composites”) were successful in producing composites from polyester and cotton blend fabrics without using additional matrix or reinforcement materials. PET/cotton blend composites were formed by treating with plasticizers like 2- Phenyl phenol and glycerol. This process was successful in producing composites at 260 °C, 270 °C, 280 °C and 290 °C. The aim was to compare composites developed from untreated PET-cotton blend fabrics to plasticizer-treated composites to reduce the time taken to fabricate composites.

Studies were able to produce regenerated cellulosic fibers from waste PET and cotton blend textiles. N-methyl-morpholine N-oxide was used as a solvent to dissolve cellulose materials and thereby separating from synthetic materials contained in the original composition to obtain a cellulose solution according to Kazuyuki Yabuki et al (Kazuyuki Yabuki et al., 2003 – “Process for producing regenerated cellulosic fibers”). This cellulosic solution was used to produce high quality regenerated cellulosic fiber
through the high-speed spinning process. However, this process resulted in wastage of valuable synthetic which could instead be reused thereby reducing the contribution towards environmental pollution.

Until now, the ways of reusing cotton and PET waste textiles together was described but they can also be reused separately. Cotton waste was usually reused in shredded form. Shredded cotton was usually used as filling materials or to produce regenerated cellulosic fibers or to produce composites using molten polymers. This makes reuse of cotton easier compared to PET. This may be an effective way for using cotton but, the lack of continuity of the fibers will have a negative impact on the mechanical properties of the future materials. On the other hand, waste polymeric materials like PET or PP (Leonard Y.Mwaikambo et al., 2000 – “Kapok/cotton fabric-polypropylene composites”), (Maries Idicula et al., 2006 – “Thermophysical properties of natural fiber reinforced polyester composites”) are melted and then used to develop composites using natural fibers like banana, sisal. This process is not preferred due to its lower mechanical properties resulting from poor interfacial adhesion between matrix and reinforcement.

The study titled “Polyester cotton composites with graphite modified cotton as reinforcement” by Hashmi et al (S.A.R.Hashmi et al., 2007) has shown that cotton undergoes graphite modification which was used as reinforcement to polyester which can be used to fabricate composites.

Review done by Layth Mohammed et al (Layth Mohammed et al., 2015 – “A review on natural fiber reinforced polymer composites and its applications”) comparing properties of composites prepared from cellulosic/cellulosic and cellulosic/synthetic
fibers. Composites prepared with two cellulosic fibers *i.e.*, cellulosic/cellulosic are less common when compared to cellulosic/synthetic fibers. Composites fabricated with cellulosic/synthetic fibers are economical and retains better processing, mechanical, physical, electrical, thermal and dynamic mechanical properties.

The study “Recycling of polyethylene terephthalate” by M.Evstatiev et al (M.Evstatiev et al., 2002) helps in understanding the process of recycling the polyester, methods, and characterization techniques to know the recycled polyester properties. *Hybrid Ramie Cotton Fabric Composites* by C.Z Paiva Júnior (C.Z Paiva Júnior et al., 2004), Natural fiber reinforced PET composites, Natural fiber reinforcement on other polymers to form composites were few other studies which helped us in understanding the fabrication of composites made from natural fibers with PET.

Most of the composites reported in the literature survey were formed at 260 °C. At such a high temperature, there may be an adverse effect on the mechanical properties of the constituent materials or the composites may become weak, mainly when using natural fibers like cotton to develop composites. Also, the composites formed with high amounts of chemicals such as alkalis, plasticizers etc. during the depolymerization process, with an idea of lowering the melting point, may increase the treatment cost and more importantly, affect the environment.

Continuous and dedicated research to reclaim valuable PET has used in many physical and chemical techniques to recycle PET. The approach to reusing PET includes melting the waste PET bottles to reproduce new products. Among all types of PET, polyester fibers have good mechanical properties and therefore are easy to be reused when compared to bottles or flakes. The chemical methods of recycling PET involve
depolymerizing the PET to obtain monomers and oligomers by hydrolysis, methanolysis and ammonolysis. Among these methods, hydrolysis is the easy process and causes less environmental problems.

Considering the achievements and the drawbacks of the various techniques studied in the literature review, this research has harnessed a method for the development of composites using 100% PET and 100% cotton waste textiles. Hydrolysis and Plasticization are the types of treatment selected in our technique for decreasing the melting point so that the composites can be prepared at a lower temperature thereby reducing the damage to cotton from high temperatures.
1.1 THEORETICAL MODEL

Procedure considered for designing the theoretical model

100% PET and 100% cotton are the materials selected to develop composites. PET has a melting point is 260°C and whereas cotton starts damages after 146°C. So the main idea is to reduce the melting point of PET to protect cotton. PET undergoes plasticization and hydrolysis to reduce its melting temperature. PET is tested with DSC (Differential scanning calorimetry) which is a thermal analysis to check whether the melting point is decreased or not. Treated PET and cotton are compressed into a composites using compression molding. Composites is tested for mechanical properties like strength, elongation, work of rupture, Flexural rigidity etc.
Chapter 4: MATERIALS AND METHODS

4.1 MATERIALS

Plain woven fabrics of 100% cotton and 100% PET are considered in fabricating the composites. The fabrics are supplied by Limian Textile Company, Wuxi, China. Cotton is used as reinforcement whereas polyester is used as a matrix in fabricating the composites. Alkali and plasticizers are used to treat PET, which are reagent grade chemicals from VWR international, Bristol, CT. The Table 4.1 presented below lists the chemicals used in plasticization and hydrolysis processes and Table 4.2 presents the properties of chemicals listed in Table 4.1.

TABLE 4.1: LIST OF PLASTICIZERS AND ALKALI

<table>
<thead>
<tr>
<th>For plasticization</th>
<th>For hydrolysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 Phenyl phenol (2PP)</td>
<td>Dimethyl Sulfoxide (DMSO)</td>
</tr>
<tr>
<td>Benzyl Butyl Phthalate (BBP)</td>
<td>Tetra methyl ammonium hydroxide (TMAH)</td>
</tr>
<tr>
<td>Diallyl Phthalate (DAP)</td>
<td></td>
</tr>
<tr>
<td>Benzoic Acid</td>
<td></td>
</tr>
<tr>
<td>Chemicals</td>
<td>Chemical formula</td>
</tr>
<tr>
<td>---------------------------------</td>
<td>------------------------------------------------------</td>
</tr>
<tr>
<td>2 Phenyl phenol (2PP)</td>
<td>C₁₂H₁₀O</td>
</tr>
<tr>
<td>Butyl benzyl phthalate (BBP)</td>
<td>CH₃(CH₂)₃OOC₆C₄H₄COOCH₂C₆H₅</td>
</tr>
<tr>
<td>Diallyl phthalate (DAP)</td>
<td>C₆H₄(COOCH₂CH =CH₂₂)</td>
</tr>
<tr>
<td>Benzoic Acid (BA)</td>
<td>C₇H₆O₂</td>
</tr>
<tr>
<td>Dimethyl sulfoxide (DMSO)</td>
<td>C₂H₆OS</td>
</tr>
<tr>
<td>Tetramethylammonium hydroxide (TMAH)</td>
<td>C₄H₁₃NO</td>
</tr>
</tbody>
</table>

The Chemicals used are:

**Plasticizers for plasticization:**

**Phenyl Phenol (2PP)** is an organic compound containing 2 benzene rings linked together with a hydroxyl group

**Benzyl butyl phthalate (BBP)** is an ester of phthalic acid, benzyl alcohol, and n-butanol.

**Diallyl phthalate (DAP)** is a Diallyl ester of o-phthalate acid which can be used as a plasticizer and for polymerization.

**Benzoic Acid** is a colorless crystalline solid with a simple aromatic carboxylic acid.

**Alkali for hydrolysis:**

**Tetra methyl ammonium hydroxide** is a quaternary ammonium salt and is a stable solid form only as a pentahydrate.
**Dimethyl sulfoxide** is an organosulfur compound. It is a colorless liquid which can dissolve both polar and nonpolar compounds and can be dissolved in a wide range of organic solvents as well as water.

Hydrolysis treatment was not effective in reducing the melting point of PET whereas plasticizers were able to reduce the melting point of PET. The main advantage of this is that the composites formed at lower temperature retain better mechanical properties when compared to composites prepared with no treatment. Therefore it is better to fabricate composites at lower temperatures as lower temperatures leave lesser damage on cellulosic materials.

### 4.2 COMPOSITES FABRICATION:

100% cotton and 100% polyester were cut into the required sizes and were treated with plasticizer and alkali. To achieve good penetration level, polyester fabric is treated using the techniques described below:

#### 4.2.1 PLASTICIZATION:

In this procedure, PET was treated with plasticizers which have high density, so the penetration of the chemicals into the PET fabrics is difficult when treated. The amount of plasticizer required for the treatment is selected based on the weight of the fabrics. For our experimentation, 10% of Plasticizer was selected. Fabric to liquor ratio was about 1:3. Ethanol was selected as solvent to dissolve the plasticizer. Once the plasticizer is dissolved completely, it is sprayed on the fabrics followed by removal of solvent.
4.2.2 HYDROLYSIS:

In this procedure, PET was treated with alkali selected on the weight of the fabrics. For this study, 10% of the alkali was selected. Water was used as solvent selected to dissolve the alkali. Once the alkali was completely dissolved in water, the alkali solution and fabrics are sealed in a canister. The canister was then placed in an oven for 30 min at a temperature of 130°C. The fabric was then removed from the canisters and rinsed completely.

FLOW CHART 4.2: HYDROLYSIS TREATMENT

After the fabric was completely dried, 7 samples of treated polyester and 5 samples of cotton were stacked together alternatively and placed between two aluminum sheets coated with Teflon. This is to ensure that the melted polyester will not stick to the compressing plates.
Mechanical hot press or laboratory scale compression molding press (Carver, Inc., Wabash, IN, USA) was selected from developing composites post-treatment. Hot press was preheated to the desired temperature to make the composites. For this study, the selected temperatures were 240°C, 250°C, 260°C and 270°C. Treated fabrics are hot pressed at 240°C, 250°C, 260°C and 270°C whereas untreated fabrics are formed into composites at 260°C and 270°C. The amount of time in the hot press also affects the mechanical properties and flexural properties of the sample. Samples are developed at 3.5mins (210 seconds) (here time refers to the amount of time a sample is held inside the hot press.)

![Flow Chart 4.3: Composite Fabrication](image)

**FLOW CHART 4.3: COMPOSITE FABRICATION**

The layers of the fabrics were evenly arranged and placed between 2 plates and pressure of 10,000 pounds or 200 psi was applied to them for the 3.5 mins (210 seconds). Finally,
the compressing molding press was turned off and cold tap water was turned on until the machine cools down. The composites were carefully removed and cut into samples as per dimensions for further tests.

4.3 MATERIAL CHARACTERISTICS

4.3.1 MECHANICAL PROPERTIES

4.3.1.1 STATIC TENSILE ANALYSIS

Tensile stress is defined as the expansion caused due to the equal and opposite force applied on a body in a tensile direction thereby causing expansion in the samples. The principle used is the constant rate of traverse. The graphs were formed using this data to compare the difference between samples which helps us to do time and temperature study.

Tensile strain was used to test the elastic nature of the composites. The comparison study of elongation for treated PET-cotton composites against untreated PET-cotton composites was done. Higher tensile strain makes the composites more durable, thereby making the composites effective for various purposes.

Work of rupture is defined as the energy required in breaking a material. It helps us to know the amount of stress and strain a sample can take before it gets ruptured.

Composites were conditioned in a standard testing atmosphere of 21 °C and 65% relative humidity for at least 24 hours before performing the test. Each data point was average of 5 or higher tested samples which are taken from different composites prepared under similar conditions.

Tensile test of PET-cotton composites was carried out on an MTS tester (QTest/10) according to the procedure of ASTM D638-03 using a 2000 pounds load cell.
The samples are cut in dog bone shape with a length of 165 mm, with a width of the widest section as 19 mm, width at the narrow section as 13 mm, and gauge length for testing as 115 mm.

Samples were carefully placed between the clamps of the tensile testing machine which runs under the principle of the constant rate of traverse as previously mentioned. Once the sample was tested the software helps us in generating the data automatically. The output data required can be selected ahead like stress, strain, modulus, energy etc. The output data received from the software was imported into an Excel file and all the means are calculated and graphs were developed which helps us to evaluate the mechanical properties of the composites.

4.3.1.2 STATIC FLEXURAL ANALYSIS

Flexible rigidity was the study of flexible nature of composites. The composites were cut into sample sizes and then tested for flexibility.

After the composites were cut into required sizes, an MTS Qtest/10 tester was used to determine the flexural properties of PET – cotton composites. According to the procedure ASTM D790-03, the size of samples was 20.3 cm x 7.6 cm with support length of 15.2 cm, and load of the cell was 2000 pounds with a crosshead speed of 10mm/min for the three-point-bend tests. The data was then imported into Excel and required graphs were developed to analyze the efficiency of the composites.

4.3.2 THERMAL ANALYSIS

DIFFERENTIAL SCANNING CALORIMETRY

A mettle Toledo DSC is used in this study, which enables us to study the effect of alkali and plasticizers on PET under a wide range of temperatures. The samples are
sealed in the aluminum cans, with both the treated and untreated samples. The weight of sample is less than 5 mg. The experiment is conducted from 25 °C to 280 °C with a heating rate of 25 °C/min and 280 °C to 25 °C with a cooling rate of 15°C/min. This experiment is carried out under a nitrogen atmosphere.

4.3.3 MOLECULAR CHARACTERIZATION

INTRINSIC VISCOSITY

Intrinsic viscosity was used to measure the solute’s (cotton) concentration in the viscosity of the solution. Cotton was hot pressed at four different temperatures 240 °C, 250 °C, 260 °C and 270 °C. Solvent selected to dissolve cotton was copper ethylene diamine. The solution was continuous stirred for 36-48 hours to ensure uniformity. A large water bath was prepared and temperature of water was maintained at 25 °C. Dissolved solution was taken into Viscometer. The time required for the solution to flow between two points was recorded. This recorded time helps us to calculate viscosity using the viscosity table which helps us to measure the Degree of polymerization (DP). Using the DP values the extent of damage in the cotton fabrics due to hot press can be estimated.

Example: The longer the time, higher is the degree of polymerization of the solution. This means the length of cellulose molecules is longer. On the other hand, if it takes less time than DP is less and therefore, length of cellulose molecules is shorter.

4.3.4 STATISTICAL ANALYSIS

Fisher’s least significant difference (LSD) provided by the SAS (SAS Institute Inc., NC) software was used in order to analyze the results of composites prepared from polyester & cotton under various temperatures and times.
Chapter 5: RESULTS AND DISCUSSION

PET-COTTON COMPOSITES:

Results from the test performed on fabricated composites fall under three different categories as follows:

5.1 COTTON EFFECT – THERMAL DEGRADATION OF COTTON

Cotton effect is studied to understand the effect of temperature on cotton fabrics. Mechanical properties and molecular characterization selected to analyze cotton behavior.

5.2 PLASTICIZER EFFECT

Plasticizer effect was studied using thermal analysis using DSC, mechanical properties are categorized into static tensile analysis and flexural rigidity, interfacial morphology and statistical analysis.

5.3 HYDROLYSIS EFFECT

Hydrolysis effect is studied using thermal analysis using DSC and mechanical properties by static tensile analysis.
5.1 COTTON EFFECT – THERMAL DEGRADATION OF COTTON

5.1.1 Mechanical Properties

TABLE 5.1: VALUES OF MECHANICAL PROPERTIES OF COTTON FABRICS

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Tensile stress (MPa)</th>
<th>Tensile strain (%)</th>
<th>Work of Rupture (J/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ORIGINAL</td>
<td>77.51    3.79   11.62</td>
<td>0.89  58.53</td>
<td>4.81</td>
</tr>
<tr>
<td>240</td>
<td>65.62  5.6     10.98</td>
<td>0.69 44.36</td>
<td>6.125</td>
</tr>
<tr>
<td>250</td>
<td>62.96  4.36  9.52</td>
<td>0.33 36.08</td>
<td>3.89</td>
</tr>
<tr>
<td>260</td>
<td>53.83  2.46  8.95</td>
<td>0.31 28.71</td>
<td>1.63</td>
</tr>
<tr>
<td>270</td>
<td>47.41  2.88  7.71</td>
<td>0.47 21.18</td>
<td>2.50</td>
</tr>
</tbody>
</table>

10

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Tensile Stress (MPa)</th>
<th>Tensile Strain (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ORIGINAL</td>
<td>77.51    3.79   11.62</td>
<td>0.89</td>
</tr>
<tr>
<td>240</td>
<td>65.62  5.6     10.98</td>
<td>0.69 44.36</td>
</tr>
<tr>
<td>250</td>
<td>62.96  4.36  9.52</td>
<td>0.33 36.08</td>
</tr>
<tr>
<td>260</td>
<td>53.83  2.46  8.95</td>
<td>0.31 28.71</td>
</tr>
<tr>
<td>270</td>
<td>47.41  2.88  7.71</td>
<td>0.47 21.18</td>
</tr>
</tbody>
</table>
Figure 5.1: MECHANICAL PROPERTIES OF COTTON FABRIC

For testing mechanical properties five different conditions are considered. Out of those four of them are hot pressed at a temperature of 240 °C, 250 °C, 260 °C and 270 °C for a time of 1 minute and the other is the control which has no heat treatment. The samples are prepared according ASTM standard which is 75mm length and 15mm width. These samples are carefully placed between the clamps of MTS QTest/10 which runs on the principle of the constant rate of traverse. For testing the samples 30 samples in each condition are considered to reduce the standard error. These results help us understand the effect of cotton fabrics on the final composites. Poor mechanical properties of cotton fabrics influence the mechanical behaviour of composites. From Table 5.1 the values of tensile stress, tensile strain and work of rupture reduces as temperature increases were shown. So this implies poor mechanical properties when compared to control. Figure 5.1
helps us understand the trend of decrease in the properties.

Tensile stress indicates the strength of the fabrics, from the graph the strength of control is more when compared to samples which are hot pressed. Strength decreases due to the damage to the cotton fabrics while hot press. So as the temperature increases the contribution of strength by cotton fabrics towards composites tend to decrease. The same trend was observed in the tensile strain and work of rupture. In case of tensile strain, elongation decreases as the treatment temperature increases resulting in poor elongation when compared to control. As observed in the tensile stress graph, strength of the fabric is reducing with temperature which means less amount of energy to break it. This cements the results observed in Work of Rupture graph, an inverse relationship between amount of energy and temperature was observed.
5.1.2 Molecular Characteristics - Intrinsic Viscosity

<table>
<thead>
<tr>
<th>Degree of Polymerization</th>
<th>Mean</th>
<th>SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>No trt</td>
<td>1997.706</td>
<td>52.18434</td>
</tr>
<tr>
<td>240</td>
<td>1075.007</td>
<td>38.98624</td>
</tr>
<tr>
<td>250</td>
<td>1048.372</td>
<td>48.5001</td>
</tr>
<tr>
<td>260</td>
<td>666.8011</td>
<td>6.057978</td>
</tr>
<tr>
<td>270</td>
<td>550.8225</td>
<td>33.55057</td>
</tr>
</tbody>
</table>

Figure 5.2: INTRINSIC VISCOSITY OF COTTON

Molecular characteristics of cotton were studied to understand the reason behind the poor mechanical properties of cotton fabrics. For molecular characterization intrinsic viscosity was selected. Cotton fabrics were dissolved in copper ethylene diamine and
tested for the viscosity. For the testing, five different fabrics were tested in each category and each fabric is tested thrice to reduce the standard error. From the Table 5.2 decrease in DP was observed as temperature increases.

Figure 5.2 helps us understand the damage to cotton caused by temperature effect due to hot press at higher temperatures i.e., 240 °C, 250 °C, 260 °C and 270 °C when compared to control (No trt). The gradual decrease in the Degree of Polymerization (DP) which helps us understand that the number of monomers or the polymer length is decreasing as the treatment temperature increases resulting in weak cotton fabrics. DP helps us understand the length of polymer chain, lesser the DP shorter the fibers and higher DP indicates longer fibers hence resulting in good mechanical properties. From Figure 5.2 the DP of control (No trt) is 1997.706 which is very high. When compared this value to samples treated at 270 °C DP is 550.8225 implying an almost 1/4th reduce in the length of the polymer. At 240 °C the value is about 1075.007 and at 250 °C it is 1048.372 which are relatively higher DP than fabrics treated at 270 °C. Therefore composites fabricated at 240 °C and 250 °C have less damage to cotton fabrics. Hence, lesser DP results in shorter fiber length or less polymer length resulting in the poor mechanical properties of composites.
5.2 PLASTICIZER EFFECT:

PET fabrics are treated using plasticizers i.e., 2PP (2 phenyl phenol), BBP (Benzyl butyl phthalate), DAP (Diallyl phthalate) and BA (Benzoic acid). Test methods used for these composites are thermal analysis using DSC (Differential scanning calorimetry), Mechanical properties like static tensile analysis and Flexural analysis using MTS QTest/10, Interfacial Morphology using SEM (scanning electron microscope) and statistical analysis is done for the above results using least significant difference. The above results will help us understand which plasticizer exhibits better properties.

5.2.1 THERMAL ANALYSIS - DIFFERENTIAL SCANNING CALORIMETRY:

![Figure 5.3: THERMAL ANALYSIS OF PET WITH PLASTICIZERS](image)

No treatment

2PP

BBP

Benzoic Acid

DAP
Figure 5.3 helps us to understand the change in the melting point by plasticizer treated PET. The graph shows melting peaks of PET samples that underwent different treatments. The first graph is a PET sample with no treatment where the melting peak is at 260 °C, which can be considered the control. The second graph shows PET treated with 2PP for which melting peak is at 230 °C which is significantly lower than the control. The remaining graphs show PET treated with BBP, DAP and BA respectively where the melting peaks are all 240 °C, which are also less than the control. Therefore the plasticizer treatment of PET significantly reduces its melting point. Further study of mechanical properties will help us understand the effect of Plasticizers on composites fabricated at lower temperatures.

From the study done by Yi Zou the melting point of PET was influenced by plasticization treatment using 2PP and glycerol. Using 2PP for the treatment and increased our search in finding plasticizer whose melting point and chemical structure are similar to PET. The chemicals with higher melting point are selected which are mentioned in Table 4.2.
### 5.2.2 STATIC TENSILE ANALYSIS

**TABLE 5.3: VALUES OF MECHANICAL PROPERTIES OF PLASTICIZERS**

<table>
<thead>
<tr>
<th>Tensile Stress</th>
<th>Tensile Strain</th>
<th>Work of Rupture</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean</td>
<td>SD</td>
</tr>
<tr>
<td>240 2PP</td>
<td>43.589</td>
<td>1.991</td>
</tr>
<tr>
<td>240 BBP</td>
<td>45.088</td>
<td>5.837</td>
</tr>
<tr>
<td>240 DAP</td>
<td>35.981</td>
<td>4.901</td>
</tr>
<tr>
<td>240 BA</td>
<td>39.867</td>
<td>5.764</td>
</tr>
<tr>
<td>250 2PP</td>
<td>36.24</td>
<td>6.519</td>
</tr>
<tr>
<td>250 BBP</td>
<td>40.166</td>
<td>4</td>
</tr>
<tr>
<td>250 DAP</td>
<td>30.489</td>
<td>6.234</td>
</tr>
<tr>
<td>250 BA</td>
<td>30.8742</td>
<td>4.19</td>
</tr>
<tr>
<td>260 2PP</td>
<td>36.608</td>
<td>3.913</td>
</tr>
<tr>
<td>260 BBP</td>
<td>12.882</td>
<td>0.619</td>
</tr>
<tr>
<td>260 DAP</td>
<td>24.848</td>
<td>9.342</td>
</tr>
<tr>
<td>260 BA</td>
<td>19.182</td>
<td>2.634</td>
</tr>
<tr>
<td>260 no trt</td>
<td>9.051</td>
<td>0.866</td>
</tr>
<tr>
<td>270 2PP</td>
<td>28.091</td>
<td>1.659</td>
</tr>
<tr>
<td>270 BBP</td>
<td>17.76</td>
<td>2.231</td>
</tr>
<tr>
<td>270 DAP</td>
<td>12.344</td>
<td>8.732</td>
</tr>
<tr>
<td>270 BA</td>
<td>10.53</td>
<td>4.276</td>
</tr>
<tr>
<td>270 no trt</td>
<td>8.34</td>
<td>1.055</td>
</tr>
</tbody>
</table>
For mechanical properties, composites are fabricated at four different temperatures 240 °C, 250 °C, 260 °C and 270 °C for 3.5 mins with a pressure of 10,000 pounds or 2,000 psi. Composites are developed under five categories which includes plasticizer treatments with 2PP, BBP, DAP, BA and no treatment. The samples are tested for static tensile test like tensile stress, tensile strain and work of rupture. Samples are prepared by following ASTM standards which are dog bone in shape. The dimensions of the samples are length of 165 mm, width of the widest section is 19 mm and width at the narrow section is 13 mm. From the table above it can be observed that composites prepared at 240 °C and 250 °C tend to have better properties when compared to composites fabricated at no treatment. Composites prepared at lower temperature and good mechanical properties are considered to be better composites.
5.2.2.1 TENSILE STRESS

Figure 5.4: TEMPERATURE STUDY FOR TENSILE STRESS

TABLE 5.4: STATISTICAL ANALYSIS OF TENSILE STRESS

<table>
<thead>
<tr>
<th>T Grouping for temp(trt)</th>
<th>Least Squares Means (Alpha = 0.05)</th>
<th>LS-means with the same letter are not significantly different.</th>
</tr>
</thead>
<tbody>
<tr>
<td>trt</td>
<td>temp</td>
<td>Estimate</td>
</tr>
<tr>
<td>BBP</td>
<td>240</td>
<td>45.0880</td>
</tr>
<tr>
<td>2PP</td>
<td>240</td>
<td>43.5841</td>
</tr>
<tr>
<td>2PP</td>
<td>250</td>
<td>41.2435</td>
</tr>
<tr>
<td>BBP</td>
<td>250</td>
<td>40.1670</td>
</tr>
<tr>
<td>BA</td>
<td>240</td>
<td>39.8676</td>
</tr>
<tr>
<td>2PP</td>
<td>260</td>
<td>36.6080</td>
</tr>
</tbody>
</table>
In **Figure 5.4** comparing all the plasticizer treated composites with the control (no trt) which indeed helps us understand the effect of the chemical treatment on the composites.

<table>
<thead>
<tr>
<th>Plasticizer</th>
<th>Temperature</th>
<th>Stress LS Mean 95% confidence limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>DAP</td>
<td>240</td>
<td>35.9809 E</td>
</tr>
<tr>
<td>BBP</td>
<td>260</td>
<td>33.1038 F</td>
</tr>
<tr>
<td>BA</td>
<td>250</td>
<td>32.8743 F</td>
</tr>
<tr>
<td>DAP</td>
<td>250</td>
<td>31.4897 G</td>
</tr>
<tr>
<td>BA</td>
<td>260</td>
<td>29.1825 H</td>
</tr>
<tr>
<td>2PP</td>
<td>270</td>
<td>28.0913 I</td>
</tr>
<tr>
<td>DAP</td>
<td>260</td>
<td>24.8487 J</td>
</tr>
<tr>
<td>BBP</td>
<td>270</td>
<td>17.7607 K</td>
</tr>
<tr>
<td>BA</td>
<td>270</td>
<td>15.5350 L</td>
</tr>
<tr>
<td>DAP</td>
<td>270</td>
<td>12.3446 M</td>
</tr>
<tr>
<td>none</td>
<td>260</td>
<td>12.0512 M</td>
</tr>
<tr>
<td>none</td>
<td>270</td>
<td>8.3411 N</td>
</tr>
</tbody>
</table>

---

**Figure 5.5: TENSILE STRESS - LS Mean 95% confidence limit**
Tensile stress values using Table 5.4 and Figure 5.5 show that 240-BPP gives the best result with the highest value at 45.0880 (standard error = 0.3642) and is not significantly different (estimate = 3.4172, standard error = 0.5151, p-value = <.0001) from 240-2PP at 43.5841 (standard error = 0.03642). These two values perform significantly better than the other values as shown in the Least Squares Means table above and also shown by the average of the two highest values being significantly different from the next highest value, 240-2PP and BPP vs 250-BPP (estimate = 8.3382, standard error = 0.8922, p-value < 0.0001). The highest value is significantly different from the lowest value, 240-BPP vs none-270 (estimate = 35.2431, standard error = 0.5151, p-value < 0.0001). Compare the estimates like 2PP vs BBP (estimate = 13.4075, standard error = 1.0401, p-value ≤ 0.0001) which has the least difference when compared to the others like 2PP vs BA (estimate = 32.0675, standard error = 1.0302, p-value ≤ 0.0001), 2PP vs DAP (estimate = 44.8631, standard error = 1.0302, p-value ≤ 0.0001) and 2PP vs none (estimate = 75.1404, standard error = 1.2618, p-value ≤ 0.0001) which are estimated to have a huge difference between the treatments.

From these analyses, the composites prepared with plasticizer treatments of 2PP and BBP have better strength and the significant difference is very less whereas when compared to other composites they have poor strength and difference between the estimates is high. Higher strength is due to higher interfacial bonding between PET and cotton and less damage to the cotton fabric resulting in a better mechanical property.

There is a decrease in the strength of the composites as the treatment temperature increases. At 260°C and 270°C, plasticizer treated composites has better strength than the other composites and composites fabricated with no treatment tend to have lowest
strength. Overall composites prepared with BBP and 2PP have higher strength when fabricated at lower temperature but overall composites prepared with plasticizer treatment tend to exhibit higher strength than composites fabricated with no treatment.
5.2.2.2 TENSILE STRAIN:

Figure 5.6: TEMPERATURE STUDY FOR TENSILE STRAIN

TABLE 5.5: STATISTICAL ANALYSIS OF TENSILE STRAIN

<table>
<thead>
<tr>
<th>trt</th>
<th>temp</th>
<th>Estimate</th>
<th>Letter</th>
</tr>
</thead>
<tbody>
<tr>
<td>BBP</td>
<td>240</td>
<td>3.0100</td>
<td>A</td>
</tr>
<tr>
<td>2PP</td>
<td>240</td>
<td>3.0027</td>
<td>A</td>
</tr>
<tr>
<td>BBP</td>
<td>250</td>
<td>2.4813</td>
<td>B</td>
</tr>
<tr>
<td>BA</td>
<td>240</td>
<td>2.4453</td>
<td>B</td>
</tr>
<tr>
<td>BBP</td>
<td>260</td>
<td>2.2884</td>
<td>C</td>
</tr>
<tr>
<td>DAP</td>
<td>240</td>
<td>2.2100</td>
<td>C</td>
</tr>
<tr>
<td>BA</td>
<td>250</td>
<td>2.1087</td>
<td>D</td>
</tr>
</tbody>
</table>

T Grouping for temp(trt)
Least Squares Means (Alpha=0.05)

LS-means with the same letter are not significantly different.
Figure 5.7 helps us understand the amount of elongation a sample can undergo before it breaks. Composites prepared at 240°C have the highest elongation when compared to the composites fabricated at other temperatures.

From Table 5.5 and Figure 5.7, tensile strain of 240-BPP performs the best with the highest value at 3.01 (standard error = 0.0331) and is not significantly different

<table>
<thead>
<tr>
<th></th>
<th>temp</th>
<th>strain LS-Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>DAP 250</td>
<td></td>
<td>1.8433 E</td>
</tr>
<tr>
<td>2PP 260</td>
<td></td>
<td>1.8423 E</td>
</tr>
<tr>
<td>BA 260</td>
<td></td>
<td>1.7653 E</td>
</tr>
<tr>
<td>2PP 250</td>
<td></td>
<td>1.7553 E</td>
</tr>
<tr>
<td>BBP 270</td>
<td></td>
<td>1.5427 F</td>
</tr>
<tr>
<td>none 260</td>
<td></td>
<td>1.5353 F</td>
</tr>
<tr>
<td>2PP 270</td>
<td></td>
<td>1.4887 F</td>
</tr>
<tr>
<td>DAP 260</td>
<td></td>
<td>1.2850 G</td>
</tr>
<tr>
<td>DAP 270</td>
<td></td>
<td>1.0120 H</td>
</tr>
<tr>
<td>BA 270</td>
<td></td>
<td>0.9789 H</td>
</tr>
<tr>
<td>none 270</td>
<td></td>
<td>0.7961 I</td>
</tr>
</tbody>
</table>
(estimate = .007, standard error = 0.04, p-value = 0.8756) from 240-2PP at 3.0027 (standard error = 0.0331). These two values perform significantly better than the other values as shown in the least squares means Table 5.5 and also shown by the average of the two highest values being significantly different from the next highest value, 240-2PP and BPP vs 250-BPP (estimate = 1.05, standard error = 0.08, p-value < 0.0001). The highest value is significantly different from the lowest value, 240-BPP vs none-270 (estimate = 2.21, standard error = 0.046, p-value < 0.0001). Comparing the estimates like 2PP vs BBP (estimate = 0.5213, standard error = 0.04681, p-value < 0.0001) have least significant difference when compared to 2PP vs BA (estimate = 0.7908, standard error = 0.09362, p-value < 0.0001), 2PP vs DAP (estimate = 1.7387, standard error = 0.09362, p-value < 0.0001) and 2PP vs none (estimate = 3.4950, standard error = 0.1147, p-value < 0.0001) which are estimated to have a huge difference between treatments.

From this analysis, 2PP and BBP treated composites exhibit better elongation and the significant difference between both is less which makes them fall under same range. Composites prepared from other plasticizers like BA and DAP also have good elongation than control but less when compared to 2PP and BBP. Composites prepared at 270 °C with DAP and no treatment have poor elongation.
5.2.2.3 WORK OF RUPTURE:

Figure 5.8: TEMPERATURE STUDY FOR WORK OF RUPTURE

TABLE 5.6: STATISTICAL ANALYSIS OF WORK OF RUPTURE

<table>
<thead>
<tr>
<th>T Grouping for temp(trt)</th>
<th>Least Squares Means (Alpha=0.05)</th>
</tr>
</thead>
<tbody>
<tr>
<td>trt</td>
<td>temp</td>
</tr>
<tr>
<td>BBP</td>
<td>240</td>
</tr>
<tr>
<td>2PP</td>
<td>240</td>
</tr>
<tr>
<td>DAP</td>
<td>240</td>
</tr>
<tr>
<td>BA</td>
<td>240</td>
</tr>
<tr>
<td>BBP</td>
<td>250</td>
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<tr>
<td>DAP</td>
<td>250</td>
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<tr>
<td>BA</td>
<td>250</td>
</tr>
<tr>
<td>BBP</td>
<td>260</td>
</tr>
<tr>
<td>BA</td>
<td>260</td>
</tr>
<tr>
<td>DAP</td>
<td>260</td>
</tr>
<tr>
<td>2PP</td>
<td>250</td>
</tr>
</tbody>
</table>
Amount of energy required to break the composite is measured from Figure 5.8. Composites prepared with at 240 °C BBP need more energy to rupture the composites whereas DAP and BA require less energy. At 250 °C BBP, BA and DAP require more energy to break and 2PP requires less. At 260 °C and 270 °C composites with treatment require more energy when compared to control. Composites fabricated with treatment
tend to show better work of rupture indicating that treatment is effective in protecting the cotton and developing better composites.

Work of rupture according to Table 5.6 and Figure 5.9 show that 240-BPP is performing the best with the highest value at 144.84 (standard error = 0.6038) and is not significantly different (estimate = -0.007, standard error = 0.04, p-value = 0.8756) from 240-2PP at 143.80 (standard error = 0.6038). Estimates help us understand the results better. Comparing 2PP at 240 and BBP at 250 (estimate = 23.6478, standard error = 0.8540, p-value = <.0001), 2PP vs BA (estimate = -25.9189, standard error =1.7079, p-value = <.0001), 2PP vs BPP (estimate = -68.3369, standard error =1.7079, p-value = <.0001), 2PP vs DAP (estimate = -32.4663, standard error =1.7079, p-value = <.0001), 2PP vs none (estimate = 114.50, standard error =2.9018, p-value = <.0001), 240 vs 250 (estimate = 175.91, standard error = 1.7079, p-value = <.0001) and 240 vs 260 (estimate = 290.63, standard error = 1.6203, p-value = <.0001) above results helps us to understand that the composites fabricated at 240 °C had better properties than composites at other temperatures.

From this analysis, composites developed at 240 °C with BBP and 2PP require more energy to break the composites compared to composites formed at other temperatures. Overall composites fabricated at lower temperature tend to require more energy to break than the composites developed at higher temperature because at that high temperature there is large impact on cotton causing it to damage resulting in reduction of mechanical properties of the composites.

Static tensile analysis calculated from above figures helps us understand that composites fabricated at lower temperature with plasticization treatments are better when
compared to composites by Yi Zou “Reusing polyester/cotton blend fabrics for composites” - composites were fabricated at higher temperatures and exhibits poor mechanical properties. The poor mechanical properties is due to the damage caused to the cotton which was proved by mechanical and molecular characteristics of cotton fabrics calculated in cotton effect.

Composites using plasticizer 2PP were developed in Yi Zou’s research but they were fabricated at higher temperature, composites are developed at lower temperature which was not focusing in there research. Composites fabricated at lower temperature exhibit higher strength in case on BBP at 260 °C tensile stress 33 MPa and the control at 260 °C is 12 MPa. There was a 175% increase in the strength due to the plasticizer treatment. In the same way the elongation and work or rupture are influenced by treating them with plasticizers. Indicating that composited developed at lower temperature with plasticization treatment exhibit better mechanical properties when compared to composites fabricated with no treatment.
5.2.3 STATIC FLEXURAL ANALYSIS:

Flexural rigidity is studied measuring peak stress using testing machine MTS Qtest/10. Samples are prepared according to standard ASTM and conditioned for 24 hours before testing.

From the above Figure 5.10, composites fabricated at 250 °C show more flexibility and as the temperature increases the flexibility tends to decrease. Composites prepared with plasticizer treatment have higher flexibility when compared to control (no trt). Among all the plasticizers BA tend to show lower flexibility. At 260 °C the highest flexibility is for BBP whereas least is for BA and no treatment which explains us that composites have low flexibility as the temperature increases causing poor mechanical properties.
5.2.4 INTERFACIAL MORPHOLOGY

Figure 5.11 – SEM TO STUDY THE INTERFACIAL MORPHOLOGY

SEM helps us to understand the interfacial bonding and structure of individual compounds during different temperatures of hot press. On the left side, long cotton yarns which are intact with melted polyester or PET at a temperature of 240 °C for 3.5 mins. Whereas the picture on the right is taken at 260 °C for 3.5 min, it clearly shows in figure 5.11 that cotton fibers are broken into smaller pieces which help us to understand the effect of temperature on the cotton. Cotton fibers are damaged or broken as temperatures increase which gives us an explanation behind the poor mechanical properties of the composites. Composites prepared at 240 °C have longer cotton fibers which contribute to better mechanical properties when compared to the composites fabricated at 260 °C. The presence of continuous cotton fibers provides better mechanical properties to composites prepared at 240 °C compared to the ones prepared at 260 °C.
5.3 HYDROLYSIS EFFECT

5.3.1 TETRA METHYL AMMONIUM HYDROXIDE - TMAH

5.3.1.1 Thermal Analysis: Differential Scanning Calorimetry

From Figure 5.12 DSC graph gives as melting peak which helps us to study the thermal behavior of the composites. In the above graph appears to be at 260 °C which means that there is not shift in the melting point of PET but hydrolysis treatment. Hydrolysis treatment using TMAH was not successful in reducing the melting point of PET and composites cannot be fabricated at a temperature lower than 260 °C.
5.3.1.2 Mechanical Analysis – Static Tensile Analysis

Mechanical properties of composites which were fabricated at 260 °C for 1.5 min are studied. Composites were prepared with 0% TMAH and 10% TMAH. Mechanical properties like strength, elongation, and work of rupture are tested.

From Figure 5.13 the composites prepared without treatment exhibit good mechanical properties when compared to the composites with treatment. In the case of tensile stress, there was a decrease in strength due to hydrolysis treatment which indicated that composites are being damaged and same trend is observed in case of elongation and work of rupture. So hydrolysis treatment using TMAH was causing damage to the composites resulting in poor mechanical properties.
5.3.2 DI METHYL SULPHOXIDE - DMSO

5.3.2.1 Thermal Analysis: Differential Scanning Calorimetry

PET fabrics are treated with DMSO using hydrolysis treatment procedure. The samples undergo thermal analysis to check the shift in the melting point using Differential scanning calorimetry (DSC).

According to Figure 5.14, the melting peak is at 260 °C which means that composites cannot be prepared at a lower temperature below than 260 °C. By observing the shape of the curve it can help us understand the crystalline and amorphous nature in PET after treatment. The peak is narrow which means that the amount of amorphous nature in PET is less. The amorphous nature helps in melting of PET so as the amount of amorphous nature in PET decreases temperature required for melting PET is more which is 260 °C.
5.3.2.2 Mechanical Analysis: Static Tensile Analysis

From Figure 5.15, the effect of DMSO on strength, elongation and energy was interpreted. Tensile stress which represents strength decreases as the percentage of DMSO increase. Strength of composites prepared with 0% DMSO has better properties. Tensile stress is directly proportional to strain so same trend is experienced in elongation so as the percentage of DMSO increases elongation decreases. Strength influences the work the rupture because work of rupture represents the amount of energy required to break the composite so as the composites strength decreases less amount of energy is required to break the composites. Overall, DMSO treatments results in decreasing the mechanical properties decreases.

Figure 5.15: DMSO EFFECT ON THE COMPOSITE FABRICATION
Chapter 6: CONCLUSIONS

Waste PET and cotton textiles were successfully fabricated into composites. Plasticization and hydrolysis treatment were selected to reduce the melting point of PET. Plasticizer treatment was effective in reducing the melting point so they are developed at lower temperatures i.e., 240 °C and 250 °C. Among plasticizers 2PP and BBP exhibits better mechanical properties. Overall composites developed with plasticizer treatment have better mechanical properties when compared to control.

Successfully in fabricating the composites at lower temperature tend to have better mechanical properties due to less damage caused to cotton fabric. In order to corroborate our findings, molecular characterization and interfacial morphology to study the damage done to cotton fabrics when treated at higher temperatures.

Hydrolysis treatment was found not successful in decreasing the melting point of PET. This was proved by the generated DSC graphs from thermal analysis. Additionally, the hydrolysis effect caused damage to PET resulting in poor mechanical properties than composites fabricated without treatment.
Chapter 7: FUTURE WORK

From this research, successful fabrication of composites at lower temperature with better properties but there are several suggestions for further study which might help to understand this process better.

PET/Cotton blend fabrics can be considered instead of 100% PET 100% cotton for fabricating the composites by selecting chemicals which can help reduce the melting point of polyester and protect the cotton properties. Cotton can also be treated with coupling agents to protect its properties when fabricated at higher temperatures.

This study used TMAH and DMSO during hydrolysis treatment for reducing the melting. Alternative alkali can be selected for the hydrolysis study. Plasticizer treatment was successful in reducing the melting point of PET but have not studied the structural modification of PET. Structural modification of PET can be studied for understanding the changes occurred in PET. Another property that should be studied is the intermolecular bonding or the interfacial attraction for all the chemical treatments.

Alternative methods of thermal analysis like TGA (thermal gravimetric analysis) can be studied for understanding the results. Also, mechanical properties of the composites like abrasion resistance, impact resistance can also be studied, which were not included in this study.
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