EFFECT OF SHORT FIBERS ON HIGH LOAD BEARING PRESS-ON TIRE TREAD COMPOUND

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DECLARATION

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Abstract

This research work was carried out to investigate the effect of short nylon fiber on natural rubber based solid tire tread compound. It was also an objective of this work to find the best fiber loading that would produce short fiber-rubber composites of good physical and mechanical properties of vulcanized rubber. A screening procedure was carried out to identify the best fiber content.

Natural Rubber based compound was used and Nylon 6 short fibers were used. Length of fiber was selected as around 5 mm and fiber loadings were varied from 1 to 20 part per hundred rubber. The short fibers were taken from the RFL dip nylon cords but an additional dry bonding system was added to the compound to overcome the bonding failures in cutting edges of the fibers.

The Physico-mechanical properties, Dynamic properties and Cure characteristics were studied in detail. There is a positive influence on the tearing strength, cut & chip resistance and compression test (load bearing capability) with the increase in fiber content. The tensile strength and elongation properties were negatively influenced with the loading of short fiber. There was no major influence on Hardness and Specific Gravity of the composites with the increase of fiber content. At the low fiber loading, Abrasion resistance, rolling resistance and hysteretic energy loss (Resilience) of composites had positive influence on properties but beyond the fiber content of 3 parts per hundred rubbers, the properties were negatively influenced.

The Minimum torque and Mooney viscosity decreased with the fiber loading up to 5 phr but it increased with the further loading of short fiber. The scorch time increased with the fiber loading but there was no major influence on optimum curing time (t_{90})

As such the general conclusion inferred from this study favor a high potential of using short nylon fiber as reinforcing material for tire tread compounds which are used in high load bearing applications in solid tires.

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LIST OF ABBREVIATIONS

Abbreviation	Description
ASTM	American Society for Testing and Materials
BR	Butadiene Rubber
BS	British Standard
CR	Chloroprene Rubber
CTV	N - [Cyclohexylthio] Phtalimide
DIN	“Deutsches Institut für Normung” which means 'German Institute of Standardization
DMA	Dynamic Mechanical Analyzer
EPDM	Ethylene propylene diene monomer
E’	Storage modulus
E”	Loss modulus
HMT	Hexamethylene tetramine
HRH	Hydrated Silica, Resorcinol and Hexamethylene Tetramine
IRHD	International Rubber Hardness
LEFM	Linear elastic fracture mechanism
MH	Maximum torques
ML	Minimum torques
NBR	Acrylonitrile Butadiene Rubber
NR	Natural Rubber
phr	Part per Hundred Rubber
PAC	Polycyclic aromatic compound
PON	Press-On Tires
PVI	Pre Vulcanization Inhibitor

Abbreviation	**Description**
RFL	Resorcinol Formaldehyde Latex
RSS	Rib smoke sheet
SBR	Styrene Butadiene Rubber
SFRP	Short fiber reinforced polymer
ts2	Scorch time
t90	Optimum cure time
TBBS	N-Tert-Butyl-2-Benzothiazolesulfenamide
TMQ (Polymerized)	2,2 4-Trimethyl - 1,2 Dihydro Quinoline-

1 CHAPTER 01 - INTRODUCTION

1.1 Background

It is highly diversified the application of tires in the modern world. In that, Solid Tyre is older version, which is revolutionarily overtaken by Pneumatic Tyre. However Solid Tyre still plays an important role as they are used in a unique variety of industrial applications owing to their special features. Solid tires are extremely stable, puncture-resistant, and maintenance-free (A.N. & J.D, 2005). They have a high load bearing capacity and are extremely economical. These features make them ideal for forklift trucks, airport vehicles, heavy-duty transport vehicles, platform trucks, and other industrial vehicles.

Solid tires are broadly classified into three main categories, namely, Resilient tires, Press-on tires and Cured-on tires (A.K., 1985).

A press-on-band tire is made of rubber tread bonded to a mild steel band which is pressed as interference fit onto a wheel. These press-on tires are widely used in airport ground support equipment. Passenger boarding bridges are common devices placed in every airport and they are mostly comprised of press-on tires. These boarding bridges are movable connectors that extend from an airport gate to an airplane. Therefore, the tires which are used in boarding bridges require high load and high strain bearing capability. Hence, specially formulated rubber compounds are used in the manufacture of these tires.

In the competitive tire industry, rubber compound design and compounding are key elements, to get right performance into right application. Reinforcement is a main pillar in rubber compound design while vulcanizing and filling are the other main factors. Carbon black and silica are widely and successfully used as reinforcement of the rubber compound, because they are acting as fillers as well. With right usage of these Reinforcement fillers, the modules, tensile strength, tearing resistance, cut & chip resistance and abrasion resistance of final product are improved. The reinforcement mechanisms of these fillers are different from one type to another but common objective is making strong linkages between rubber chain and reinforcing fillers.

In addition to these reinforcement fillers, certain types of fibers are also used for reinforcing the rubber compound. The short fibers are used to a limited extend, generally together with a reinforcing filler to increase modulus as well as to impart anisotropy of properties where this is desired (De S.K., 1996). The fibers have higher length compared to the cross section area. Fiber reinforced compounds are characterized by physical reinforcement rather than by chemical bonds.

These fibers can be mainly classified as continuous fibers and discontinuous or short fibers (E & F, 2013). The continuous fibers are often used as a lamination with rubber compounds. There, majority of loads are taken directly by the fibers, while rubber compound is undergone less loads (E & F, 2013). Thus, those products are very strong and well suited to applications involving cyclic deformation as in pneumatic tires, belt, hoses etc. However, the preparation of the continuous fiber is complicated, multistage and is difficult to control. Due to the drawbacks of using long continuous fibers, there has been considerable interest on short fibers during the last few decades and some studies were done on discontinuous fiber reinforcement rubber compounds.

The research focus is to introduce short nylon fibers as a reinforcement agent in rubber compound for the specific application of Press-on tire (PON) category.

1.2 Research Focus

Carbon black, silica, clay and whiting are the traditional reinforcement fillers that are used in rubber industry (Rzymski, 2013). They are normally used in the particulate form. They perform well in the respective application and purpose. Short fiber in rubber compound would be beneficial for instance, in applications where high level load, modulus and hardness are required. Recent attention has been focused on the suitability of short fiber composites in high performance applications (Noodermeer, 2010). The concept of short fiber based compound is fairly new but during last three decades, it is successfully used as solid tire heel compound. Utilization of short fiber in heel compound offers economic and environmental advantages.

With the growing demand of economic competition and ecological pressure, there is an increase need for a cost effective solutions for maintaining the competitiveness of product. Therefore, the use of short fiber as reinforcing agents in rubber compound opens up a new avenue for the utilization of waste fibers, available in plenty from textile, tire and other fiber industries. The properties of short fiber containing composites depend critically on fiber content, orientation and fiber-matrix interface bond strength (Amuthakkannan P., 2013). A detailed study of the effect of these parameters on the composites properties will be highly informative.

Nylon is one of the fibers compatible with rubber compounds and performs well as short fiber, already used in tyre industry as long fibers. Fabric Calender, is the main equipment used in pneumatic tire manufacturing process, gives significant amount of unavoidable nylon rejects in each change of fiber roll during the process. One of a large pneumatic tire manufacturer in Sri Lanka disposes nylon in this way as shown in Figure 1.1 monthly. The organization has technology to use this wastage to use as reinforcement into cheap compounds in less performing areas of a tyre, there nylon fiber act more as filler than reinforcement. The scope is to focus the use of this nylon into high performing area of tyre as a reinforcement, then value addition definitely become very high. Not only waste nylon in tyre industry, but also textile waste from large number of garments daily is a very high potential candidate as source for short reinforcement fibers for tire tread compounds.

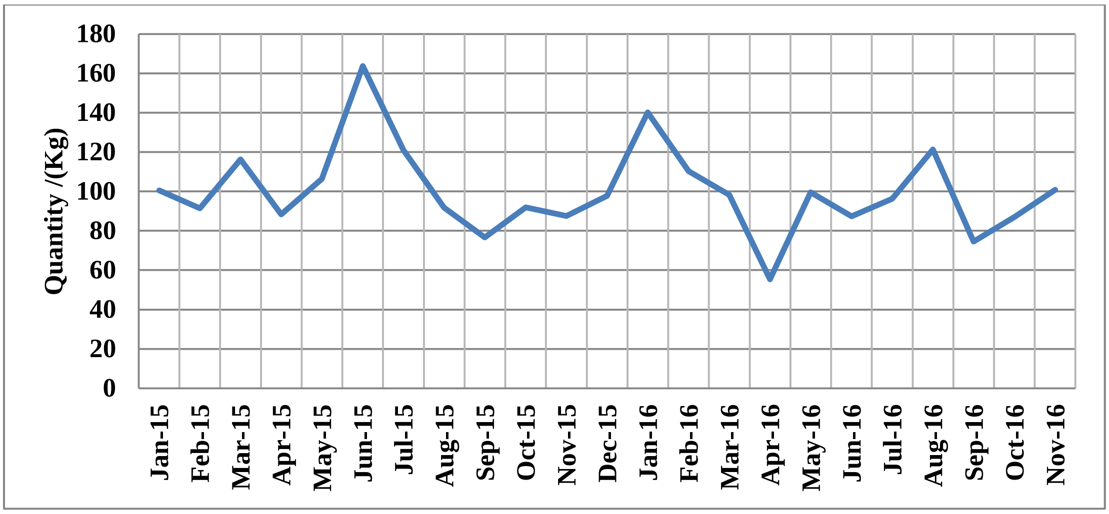


Figure 1.1 Waste Nylon Material in Fabric Calender

Tread is a potential area in solid tires for using this waste nylon as short fiber reinforcement material in the tread compound. Objective of the study is to develop short fiber reinforced tread compound for High Load PON (Press-On Tires) that are widely used in airport ground support equipment (passenger boarding bridges) that require high load performance. The current compound consumption in particular application of a leading tire manufacturer in Sri Lanka is shown in Figure 1.2.

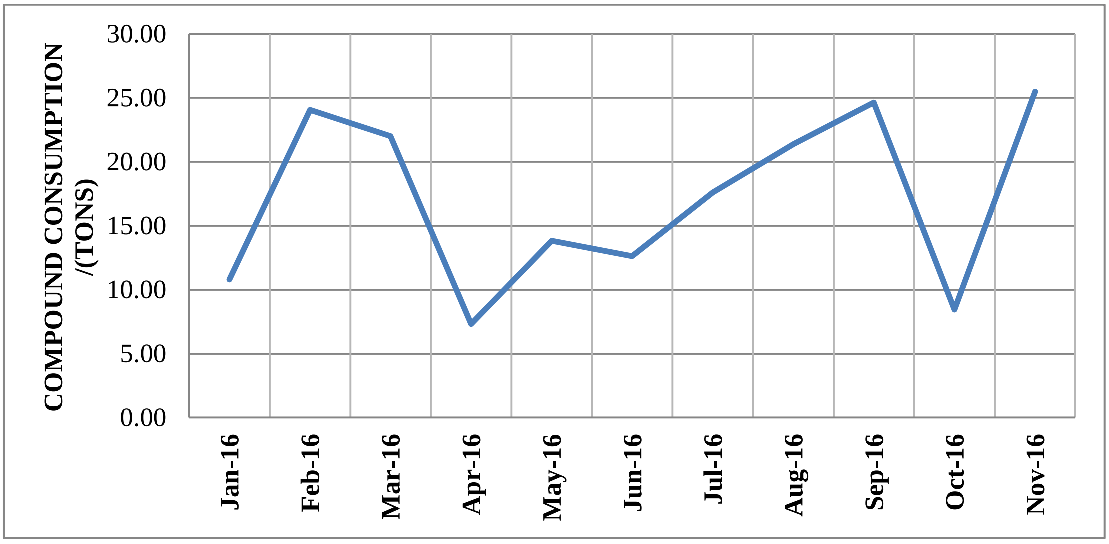


Figure 1.2 Compound Consumption

In this study, Nylon 6 material was selected because it is commonly available in tire industry as mentioned in previous paragraph. Nylon short fiber is evaluated on the properties of vulcanized product in order to use it as reinforcement material in above mentioned tread compound which is able to give a rubber compound with good performance in final product.

Following sequence is used for this study.

- Select nylon 6 short fiber material and study the effect of fiber concentration on the Rheological properties of rubber compound.
- Select nylon 6 short fiber and study the effect of fiber concentration on the physical properties of vulcanized rubber.
- Select nylon 6 short fiber and study the effect of fiber concentration on the mechanical properties of vulcanized rubber
- Select nylon 6 short fiber and study the effect of fiber concentration on the dynamic properties of vulcanized rubber.

2 CHAPTER 02 - LITERATURE SURVEY

2.1 Composites

With the expansion of material science and technology, unique and fascinating material combinations have now been developed which are commonly known as composites. A composite material is a material made from two or more constituent materials with significantly different physical or chemical properties that, when combined, produce a material with characteristics different from the individual components. The individual components remain separate and distinct within the finished structure.

Composites are one of the most advanced and adaptable engineering materials developed to date and are widely used in various industrial applications. They are heterogeneous in nature and can provide superior and unique mechanical and physical properties (Amuthakkannan P., 2013). At present, composite materials play a key role in aerospace and automobile industries because they exhibit outstanding strength to weight and modulus to weight ratio, and also because of the fibrous reinforcement they provide. High performance rigid composites are made from glass, graphite, Kevlar or silicon carbide fibers (Tsimpris C.W. and Mroczkowski T.S., 2004).

When using short fiber for reinforcing composites, high aspect ratio (length to diameter ratio) is used. Also, direction of the fiber alignment and density of the fibers make a big difference in performance.

2.2 Classification of Composites

Based on the types of reinforcement used, the composites can be classified as given in Figure 2.1.

2.2.1 Particular Reinforced Composites

Particular composites are reinforced by particles. The particles are roughly equal in all dimensions. These composites have high temperature performance, reduce friction, increase wear resistance and reduce shrinkage. The loads are uniformly shared and have improved stiffness.

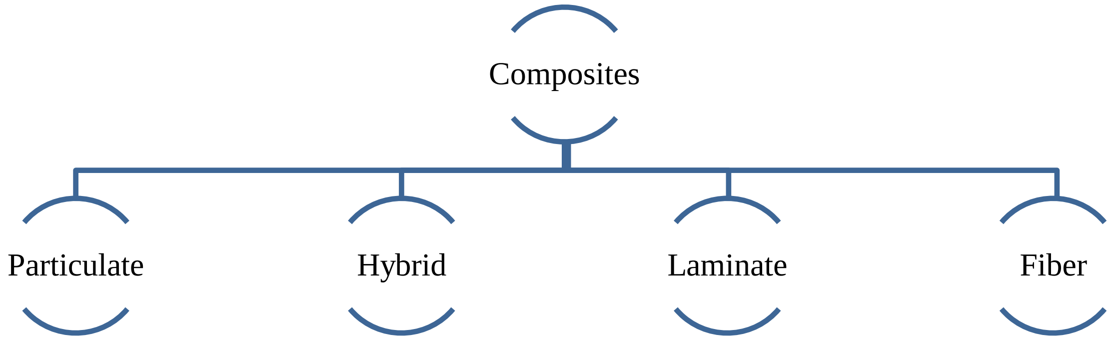


Figure 2.1 Classification of Composites

2.2.2 Hybrid Composites

Hybrid composite is made by incorporating two or more different types of fibers. These types of composites are commonly used for improving the properties and lowering the cost of conventional composites. The hybrid composites are classified according to the way of incorporating fillers.

2.2.3 Laminates

Plywood and paper are laminate composites. It is fabricated by stacking a number of layers in the thickness direction. These laminates have unidirectional or bidirectional orientation of the fiber reinforcement according to the end use of the composites. The laminate composites have good physical, mechanical and thermal behavior.

2.2.4 Fiber reinforced composites

The fibers have higher length compared to the cross section area. Fiber reinforced composites are characterized by physical reinforcement rather than by chemical bonds. They are widely used in various engineering applications.

Continuous fiber composites comprise of long fibers oriented in one direction. Discontinuous fiber composites contain short fibers embedded in the matrix. In short fiber composites, the length of short fiber are neither too long to allow neither individual fibers to entangle with each other nor too short for the fibers to lose their fibrous nature. The reinforcement is uniform in the case of composites containing well dispersed short fibers. There is a clear distinction between the behavior of short and long fiber composites.

2.3 Short Fiber-Rubber Composites

Reinforcement of rubber article with continuous cord or long fibers has been common in the industry for a long period of time. In continuous cord, reinforcing members are loaded directly by the force applied to the article and therefore the bonding between the rubber matrix and the reinforcement material is not critical (E & F, 2013). However, the preparation of the continuous fiber is complicated, multistage and is difficult to control (Floter, 2007). Due to the drawbacks of using long continuous fibers, there has been considerable interest on short fibers during the last few decades.

In the short fiber reinforcement, the fibers have a critical length which is neither too long to allow individual fibers to entangle with each other, nor too short for the fibers to lose their fibrous characteristics. The fibers are oriented uniaxially in the compound by extrusion or calendaring or milling process and have ~~a~~ good strength and stiffness (Tsimpris C.W. and Mroczkowski T.S., 2004). Short fiber composites possess several advantages over continuous fiber composites. The short fibers can be easily incorporated into the rubber compound along with other ingredients. They are amenable to standard rubber processing operations such as extrusion, calendering, milling, compression and injection moulding (Tsimpris C.W. and Mroczkowski T.S., 2004). These composites provide high green strength and high dimensional stability during fabrication. Design flexibility is another advantage of these composites. Complex shaped articles which are quite difficult to accomplish with long fiber composites can be fabricated using short fiber composites (Kavita Agarwal, 2002). Tensile strength, stiffness, low shrinkage, abrasion resistance, tears and creep resistance are greatly improved in the case of short fiber composites (Egwaikhide

A.P., 2013). On the other hand, it is a cheap material than continuous fibers. However, difficulty in achieving uniform dispersion, fiber breakdown during processing, difficult in handling and bonding are some disadvantages in short fiber composites.

During last few decades, several investigations were done on short fiber – Rubber composites. Characterization of fiber orientation in short fiber reinforcement composites with an image processing techniques was studied in various researches (Lee Y.H., 2002). . A special technique was used and good results were obtained. The measurements method in this study was effective in the case of random fiber distribution as well as unidirectional distribution. The compression modulus of elastomers that is important performance properties in bridge bearing and base isolation bearing for structures located in seismic zones were studied in depth (Fediuc D.O., 2013). A Zwick hardness tester was mainly used to determine the properties of elastomers. The main aim of this study was to investigate and define the relationship between compression and shear modulus, hardness and shape factor of the composites (Fediuc D.O., 2013). Dynamic visco elastic properties of nylon short fiber reinforced composites were studied in some research (Li Chen, 1989). In this study, the storage modulus and loss modulus increases with fiber loading were reported. Special elastomers were developed with short fiber for tire reinforcement by Dupont (Tsimpris C.W. and Mroczkowski T.S., 2004). Low strain modulus, tear resistance and slow tear propagation without significant increase in hysteresis were greatly observed in the development. The fracture toughness characterization of random fiber reinforced composites had been investigated by (Todd M.M. and Victor C., 1987). In this study, classical linear elastic fracture mechanism (LEFM) approached was used. The composites with long fibers (24-50 mm), inelastic behavior ahead of crack tips in the process zone occurs to such an extent that the small scale yielding requirement of LEFM was not satisfied. In composites randomly reinforced with short fibers (0.5 mm) the process zone was sufficiently small that LEFM toughness tests were valid with standard specimen size. A truck tire tread compound reinforcement with short aramid fibers was studied (Noodermeer, 2010). In his study, it was clearly showed that contrary to the NR-sulfur system, in the NRperoxide system the adhesion effect to RFL was improved, although the reinforcing degree was not as good as an EPDM compound due to the poorer dispersion of fibers in NR. Also, effect of short nylon fibers on the mechanical properties of Natural Rubber (NR) vulcanizates were studied (Senapati A.K., 1988). Also, some researches were done on real applications to check the suitability of using short nylon fibers on V-belt application. V-belt is another potential application and detail analysis was done in past researchers (King Chang Kee, 1990).

A short fiber composite comprises of the two main constituents of short fibers and rubber matrix

2.4 Rubber Matrix

Different types of elastomers have been used as the rubber matrix for short fiber reinforcement. Typically, the matrix has considerably lower density, stiffness and strength than the fiber (Burrowes, 2005). After incorporating fibers into rubber matrix, the final product shows high strength and stiffness. Low density of the matrix helps to mix both materials together better. In a composition, the rubber matrix is required to fulfill the following functions (De S.K., 1996):

- To bind the fibers together by virtue of its cohesive and adhesive characteristics
- To protect them from adverse handling and impacts of the environment
- To disperse the fibers and maintain the desired fiber orientation and spacing
- To be chemically and thermally compatible with reinforcing fibers

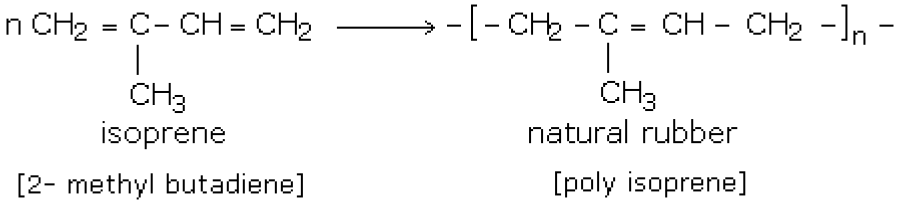
Following elastomers or blend of elastomers is widely used as rubber matrix.

- Natural Rubber (NR)
- Styrene Butadiene Rubber (SBR)
- Acrylonitrile Butadiene Rubber (NBR)
- Chloroprene Rubber (CR)

2.5 Natural Rubber

2.5.1 Structure and Properties of Natural Rubber (NR)

The natural rubber latex is obtained from the milky secretion of the tropical tree called Hevea Brasiliensis. Its latex is collected by making a cut in the tree and it is coagulated with acetic or formic acid. It may then be squeezed, rolled, milled and vulcanized. It is an excellent example of a natural polymer. It is a linear long chain molecular made up of an unsaturated isoprene (C5 H8) repeat units. There may be many as 11,000 to 20,000 isoprene units in a polymer chain of natural rubber (S.R.O., 2007).



$$n\,CH_2 = \underset{\displaystyle CH_3}{\underset{|}{C}} - CH = CH_2 \longrightarrow -[-CH_2 - \underset{\displaystyle CH_3}{\underset{|}{C}} = CH - CH_2 -]_n -$$

Figure 2.2 Chemical Structure of Natural Rubber

Properties of Natural Rubber (Renner, 2011)

- Natural rubber is tough and elastic material. It becomes soft and sticky when the temperature rises. After vulcanization, it is comparatively hard and non-sticky.
- It has a coil like structure and when stretched, it expands and attains its original state when released. The molecules straighten out when stretched and when released, they coil up again.
- Raw natural rubber has low tensile strength and abrasion resistance but vulcanized rubber has high tensile and high abrasion resistance.
- Raw natural rubber can be used over a narrow range of temperature from 10 to 60 degree centigrade but after vulcanization, it can be used over a wide range of temperature from -40 to 100 degree centigrade.
- It is insoluble in water, alcohol, acetone, dilute acids and alkalis.
- It is soluble in ether, carbon disulphide, carbon tetrachloride, petrol and turpentine.

- Raw natural rubber absorbs a large amount of water but vulcanized rubber absorbs only a small amount of water.

NR can be hardened by addition fillers during the compounding and vulcanization process. The common fillers used extensively are carbon black and silica which makes it more durable and an economical product.

2.5.2 Application of Natural Rubber

Natural Rubber can be used to manufacture engineering products such as tires, tubes, other automobile components, conveyor belts, gloves, rubber bands etc. .Due to the high strength and high elongation, natural rubber is widely used in special thin wall articles like surgical gloves, condoms, balloons etc (James, 2012).

2.6 Fibers

Length to diameter ratio of a fiber is greater than 10. Other characteristic of fibers are flexibility and fineness. Fibers have a fine hair like structure and they are of animal, vegetable, mineral or synthetic origin. Fibers can be classified as follows (De S.K., 1996):

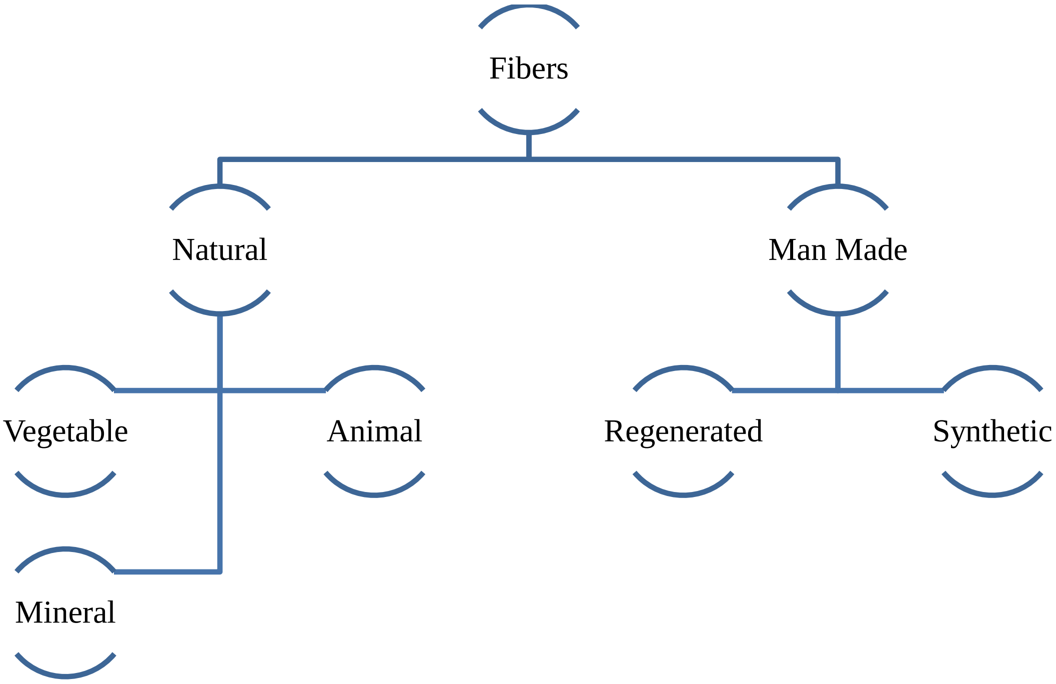


Figure 2.3 Classification of Fibers

2.6.1 Nylon Fiber

In 1931, American chemist Wallace Carothers reported on research carried out in the laboratories of the DuPont Company and it is the first successful innovation to introduce Nylon. Nylon is the first synthetic fiber to be commercialized in 1939 (Rushton, 1995). It is a polyamide fiber derived from a diamine and a dicarboxylic acid. The most common versions are nylon 6 and nylon 6,6 which is widely used as fiber and is made from adipic acid and hexamethylene diamine. The commercial production of Nylon 6 begins with carprolactum. Nylon fibers have outstanding durability and excellent physical properties. The main features are,

- Exceptional strength
- High elastic recovery
- High abrasion resistance
- Washability
- Resistance to damage from oil and resistance to many chemicals
- High Resilience
- High Melting point
- Light weight
- Long lasting fiber

Most common nylon fiber is continuous fibers. But, during last few decades, discontinuous or short nylon fibers have been a subject of a number of investigations and gradually, it is used in industrial products (Rzymski, 2013). Short fibers are available in pulp and staple forms (Monthey S. , 2013). The staples are usually greater than 5 mm long and are either precision cut, with almost all fibers having the same length or produced via a stretch-break process or a discontinuous method, which gives a random length distribution. Pulp is the traditional name for the cellulose in the paper markers’ slurries. Traditional pulp was made from flax, hamp, rise straws or asbestos. The same kind of milled organic structures are made from synthetic organic fibers. The morphology of pulp is characterized by the large array of curled fibril-like appendages attached to the original fiber core. The fibrillated structure gives rise to tremendously increased surface area, which is beneficial for the adhesion between fibers and rubber matrix.

2.7 Factors Affecting the Properties of Short Fiber – Rubber Composites

Over the last few decades, with the increased emphasis on the subject, several researches had been conducted on short fiber-rubber composites. It was highlighted that short fiber–rubber composites have gained ~~a~~ wide importance due to the advantages in processing and in the remarkable improvement in their mechanical properties (De S.K., 1996). The primary effect of short fiber reinforcement on the mechanical properties is to increase modules, increase strength, decrease elongation at rupture, increase hardness, improve cut, tear and punch resistance. In past researches, the following factors have been identified as influencing factors on the properties of short fiber–rubber composites (Amuthakkannan P., 2013).

- Fiber Length
- Fiber Aspect Ratio
- Fiber Content
- Fiber Orientation
- Fiber-Rubber Matrix Adhesion

2.7.1 Fiber Length

Importance of length and its influence on the properties of the composites were studied by several researchers. The fiber length in the short fiber-rubber matrix plays a major role. If the fiber length is too long, it is difficult to maintain the shape of the original fiber due to the entanglement and overlapping of short fiber during the mixing process (Lauke, 1996). As a result of this, poor dispersion can occur and uneven distribution of short fiber in the cured product may result. But a very small length of fiber does not offer sufficient stress transfer from the matrix to the fiber. In the mixing operation, further breakage can occur and it depends on the strength of the fiber (Kavita Agarwal, 2002). On the other hand, if the rubber matrix viscosity is high, more shear will be generated during mixing thereby exceeding the bending stress of the fiber which eventually results in further breakages.

The effects of the fiber length of basalt fiber on mechanical properties of composites were studied (Amuthakkannan P., 2013) . Different lengths were investigated and defined the fiber length 10 mm as the most appropriate for better tensile and flexural strength (Amuthakkannan P., 2013). The impact strength of basalt fiber reinforced composites of 50 mm fiber length denoted the highest impact energy.

The effect of fiber length and fiber orientation distribution for predicting the tensile strength of short fiber reinforced composites was studied (Lauke, 1996). In their study, the effects of the mean fiber length, the most probable length (mode length), the critical fiber length, the mean fiber orientation, the most probable fiber orientation and the fiber orientation coefficient on the tensile strength of SFRP were studied in detail. The results showed that the strength of SFRP increase rapidly with the increase of the mean fiber length at small mean fiber lengths and approaches a plateau level as the mean fiber length increase for the case of large mean fiber length.

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2.7.2 Fiber Aspect Ratio

Fiber aspect ratio is defined as the ratio between the length and the diameter of the fiber. The aspect ratio of the fibers is a major parameter that controls the fiber dispersion and fiber matrix adhesion which gives the optimum performance of the short fiber-rubber matrix (Lee D.J. and Ryu, 2008). If the aspect ratio of the fiber is lower than the critical aspect ratio, insufficient stress will be transferred and the reinforcement will be inefficient. On the other hand, short fibers that are too long entangle with one another causing dispersion problems. Several researchers have suggested the optimum level of aspect ratio based on their studies of this phenomenon.

Both tensile and tear properties of short nylon 66 fiber reinforced rubber have been investigated as a function of fiber aspect ratio, fiber content and bonding agents by (Lee D.J. and Ryu, 2008). They further identified the increasing rate of torque and that the hardness of short fiber reinforced rubber is slowed with a fiber aspect ratio of

200-300. The ultimate tensile strength, tensile modulus and tearing energy until crack initiation showed a maximum value with the fiber aspect ratio at about 300. The tearing energy until final rupture showed a maximum value with the fiber aspect ratio at 300-400. Senapati et al have shown that an aspect ratio of 170 gives good mechanical strength in the case of nylon 6 short fibers reinforced in NR compounds.

In the mixing and milling operation, quite often the fibers undergo size reduction. The fiber breakage with synthetic fibers like nylon 6 and PET during mixing into rubber on a mixing mill is almost negligible (Senapati A.K., 1988). Nylon fibers are very high flexible material and very high resistance to break at bending. Therefore, the fibers do not break at high shear force generated during the mixing operation.

2.7.3 Fiber Content

Fiber content has a significant influence on the strength and other properties (De S.K., 1996). A lower concentration of fibers gives lower mechanical strength and predominate the properties of the rubber matrix. At high fiber concentration, the matrix is sufficiently restrained and the higher level of tensile properties gradually improves to give strength higher than the rubber matrix. The concentration of fibers beyond which the properties of the composites improve above the original matrix strength is known as the optimum fiber concentration.

2.7.4 Fiber Orientation

Fiber orientation is a key parameter in performance of short fiber reinforced rubber composite. During the processing of short fiber reinforced rubber compound, the fibers tend to orient along the flow direction causing mechanical properties to vary in different directions. The milling, calendaring and extrusion are most commonly used processing methods in which fibers tend to orient along the direction of flow (Tsimpris C.W. and Mroczkowski T.S., 2004). A large shear force during these operations helps to orient the material in the direction of the flow. A high degree of fiber orientation could be achieved by reducing the mill nip. A small mill nip and single pass in the mill is one of the best practice for proper aligning (Akthar S., De P.P., De S.K.,, 1986). Two passes through tight mill nip were given optimum mechanical properties for Nylon 6/NR composites (Senapati A.K., 1988).

Friction ratio of the mill rolls and temperature of the mill rolls are other factors that can affect the orientation of the short fiber in a composite. Too low and too high friction ratio of mill rolls is also not good for proper orientation of fibers. If the friction ratio is around 1.05 or less, the shear force is not sufficient to orient the fibers in the flow direction. Also, if the friction ratio is around 1.75, there is a slip on the roll surface, as a result of which the fibers are not sufficiently oriented in the milling direction. The researchers have highlighted that 1.15 is the optimum friction ratio that the high tensile and tearing strength exhibits.

The effect of fiber orientation on the toughening of short fiber-reinforced composites was studied (Norman R.E. and Robertson D.A., 2003). The effect of fiber orientation on the toughening of composites by short glass fibers generally below their critical length was investigated using specimen with either well-aligned or randomly oriented fibers. According to the study, the fibers perpendicular to the fracture plane provided the greatest toughening and fibers within the fracture plane provided the least toughening.

2.7.5 Fiber – Rubber Matrix Adhesion

Bond between fibers – rubber matrix plays a major role in the composites since it is through this interface that the load is transferred to the fiber. Fibers are load bearing membranes in the composites and the load transfer depends on fiber – rubber matrix adhesions. Rubber matrix is low modulus polymer and fibers are high modulus. Due to the different modulus of both materials, deformation of the fiber – rubber matrix interface is more critical. If the interface has better adhesion, the composites work as a single component and gives better mechanical and rheological characteristic of the composites. Therefore, the most important parameter in the composites is selecting the most suitable adhesion between fiber and rubber matrix.

There are different techniques that have been employed to achieve a strong interfacial bond between fiber and rubber matrix. They are as follows:

- **Adsorption and Wetting**

This mechanism works due to the physical attraction between the surfaces, which is better understood by considering the wetting of solid surfaces by liquids. The surface roughness prevents the wetting except at isolated points. When the fiber surface is contaminated, the effective surface energy decreases. This hinders a strong physical bond between fiber and rubber matrix interface.

- **Interdiffusion**

In a composite, we can identify polymer molecules that diffuse into the molecular network of the fiber surface. The bond strength depends on the amount of molecular conformation, constituents involved and the ease of molecular motion.

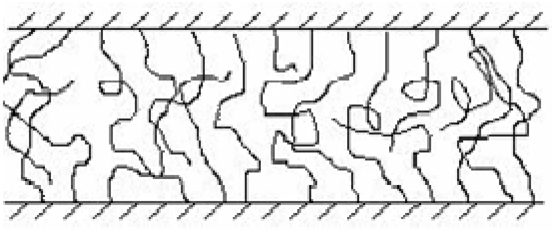

Figure 2.4 Polymer molecules diffuse into the molecular network of the fiber surface

- **Electrostatic Attraction**

This mechanism works when there is an electrostatic interaction at the interface available. It is necessary to have ~~a~~ charge differences at the interface. If there is anionic and cationic species present at the fiber and rubber matrix phases, there will be an electrostatic attraction and will make a proper bond at interface. Introduction of coupling agents at the interface can enhance bonding through the attraction of cationic functional group by anionic surface and vice versa.

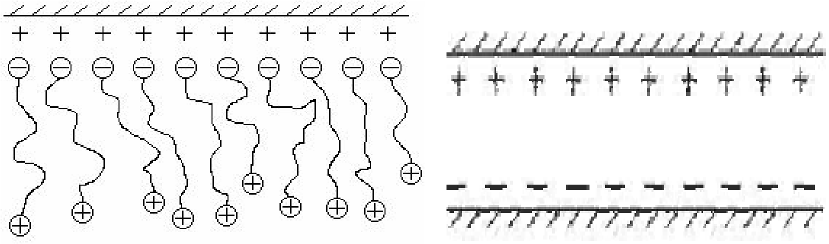

Figure 2.5 Electrostatic interaction at the interface

- **Chemical Bonding**

A chemical bond in the composite occurs when there are compatible chemical bonds in both fiber and rubber matrix. There are weak chemical bonds as well as some strong bonds. The type of bond determines the strength of interface. Interfacial chemical bonding can increase the adhesive bond strength by preventing molecular slipping at a sharp interface during fracture and by increasing the fracture energy by increasing the interfacial attraction.

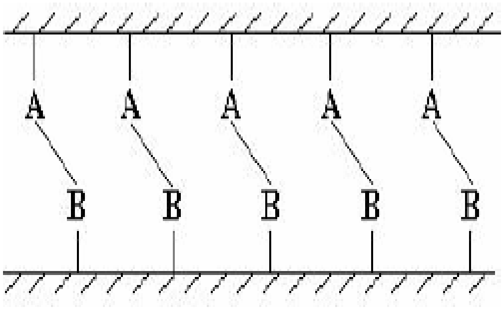

Figure 2.6 Chemical bonds in both fiber and rubber matrix

- **Mechanical Adhesion**

Roughness of the fiber surface plays an important role in mechanical adhesion. If the surface roughness of the fiber is high, a significant bond will be available at the interface. The surface roughness can increase by promoting wetting or providing mechanical anchoring sites. Natural fibers such as cotton have short whiskers protruding from the surface, which help to give a physical bond when mixed in rubber. Nylon, polyester and rayon have smooth surfaces and adhesion of these fibers to the rubber matrix is comparatively poor.

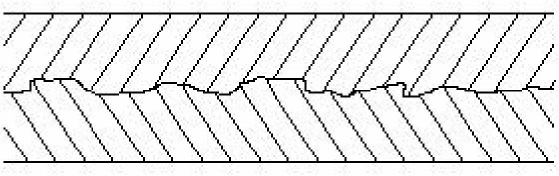

Figure 2.7 Mechanical adhesion due to surface roughness

There is a lot of research done on fiber adhesion to rubber compounds. The improvement in adhesion by enhancing fiber-rubber matrix adhesion through the incorporation of a bonding system has been studied by (Suhara F., 1998). Evaluation of the three component bonding system (HRM system) for various fibers was done by (DerrigerD.C., 1971)

HRM Dry Bonding System

Hydrated Silica, Resorcinol and Hexamethylene Tetramine are main components in HRM bonding system. The dry bonding system is commonly used in rubber to create adhesion between fiber and rubber matrix. Dry bonding system is comparably easier than adhesive dipping. For the continuous fibers, adhesive dipping is common practice but in short fiber, dry bonding system is more economical. This is because the constituents of the dry bonding system can be added to the rubber matrix like any other compound ingredients. Then, extra operation like dipping and drying can be avoided.

(Soltani S., 2010) studied the effect of the bonding agent on mechanical properties & rheological properties of short nylon fiber NR/SBR composites. The composite were prepared with virgin and waste short nylon fiber using a two roll mill. According to the study, the adhesion between fiber and rubber was enhanced by using a dry bonding agents consisting of resorcinol, hexamethylenetetramine and hydrated silica (HRH). The bonding agent provided a shorter cure time and enhances mechanical properties. Further, he observed, the mechanical properties were higher in the longitudinal direction compared to the transverse direction. Also, the increase in the storage modulus and complex viscosity were found to be larger in the composites prepared with virgin fibers compared to the waste fibers composites. .

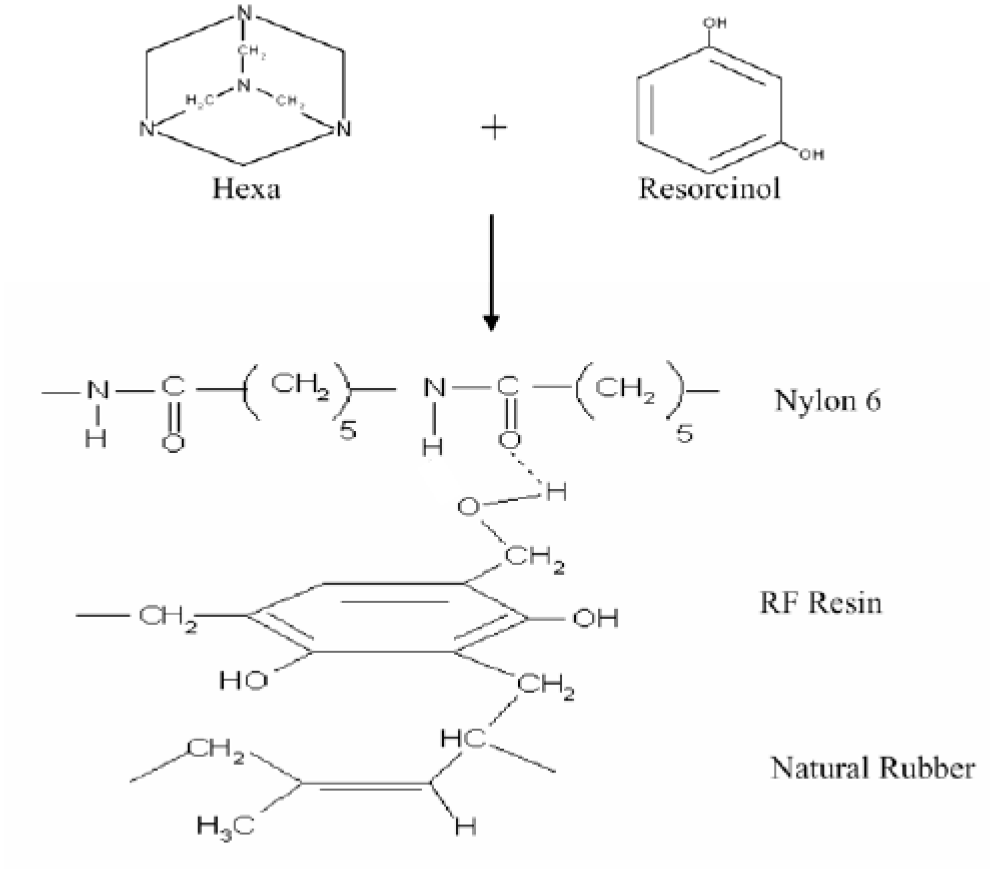

Figure 2.8 Chemical Reaction of HRH

(Dunnom, 1967) has observed the improvement of adhesion level by adding silica to a compound containing resorcinol and hexa. In addition to Dunnom, several other researchers have described various aspects of short fiber adhesion to rubber matrix in the presence of the dry bonding system. Also, researches were done by replacing silica with carbon black. (Ramayya A.P., 1986) replaced silica by carbon black in the system and there was shown improvement in the bonding strength between rayon and natural rubber. It was highlighted that the major disadvantages of HRM system in short fiber adhesion is the requirement of long curing time as per Akthar et al.

Pretreating of fibers is another approach for enhancing interfacial adhesion. (Zhou Y.H., 1993) studies a pretreating method for Nylon short fibers and it was proven that the interfacial adhesion of short nylon fiber – rubber composite was strengthened by pretreating the fibers by coagulating a mix of coupling agents or adhesives.

Evaluation of adhesion level of fiber-rubber matrix is done not only by observing the value but also with the observation of the interface surfaces. Therefore, it is a quantitative as well as a qualitative measurement.

2.8 Manufacturing of fibers

2.8.1 Continuous fibers

Continuous fibers are manufactured by some sort of spinning method. Often, it is a wet spinning. In the wet spinning process molten polymer is filtered and pumped through a spinnerette containing a large number of very fine holes. A positive displacement pump is used to give an extremely accurate and constant flow through the spinnerette. The extruded semi-molten polymer is stretched to about 25 times its original length while solidifying in a cool air stream. The solidified bundle is then treated with a spin finish to lubricate the filaments and cold-draw over a series of take-up rolls. The drawing process elongates the filaments by several hundred percent while the polymer is still above its glass transition temperature. This procedure increases the strength, modulus and reduces the breaking elongation. Controlled feed rate and draw speed are necessary to ensure filament uniformity and control linear density. Finally, the filament bundles are gathered, lightly twisted and stored on beams. It is called yarns. In the cord production, yarns are removed from beams and cords are formed by cable twisting when two or more yarns are back twisted. The twist levels are important for tire performance (Plotkin, 2009).

2.8.2 Discontinuous fibers

Discontinuous fibers are produced by centrifugal spinning with different types of set-ups of rotating wheels. These methods are generally considerably less expensive than the continuous methods. When using a centrifugal spinning method for production of discontinuous fiber it is inevitable that the diameter and length of the fibers vary (Khan, 2012).

2.9 Processing of Short fibers – Rubber composites

The processing of fibers into rubber matrix is very similar to ordinary rubber compounding. The fibers can be incorporated into the rubber mix basically the same way as other ingredients. The same processing techniques such as milling, calendering, and extrusion can be used. The steps of the process can be listed out as follows (Khan, 2012).

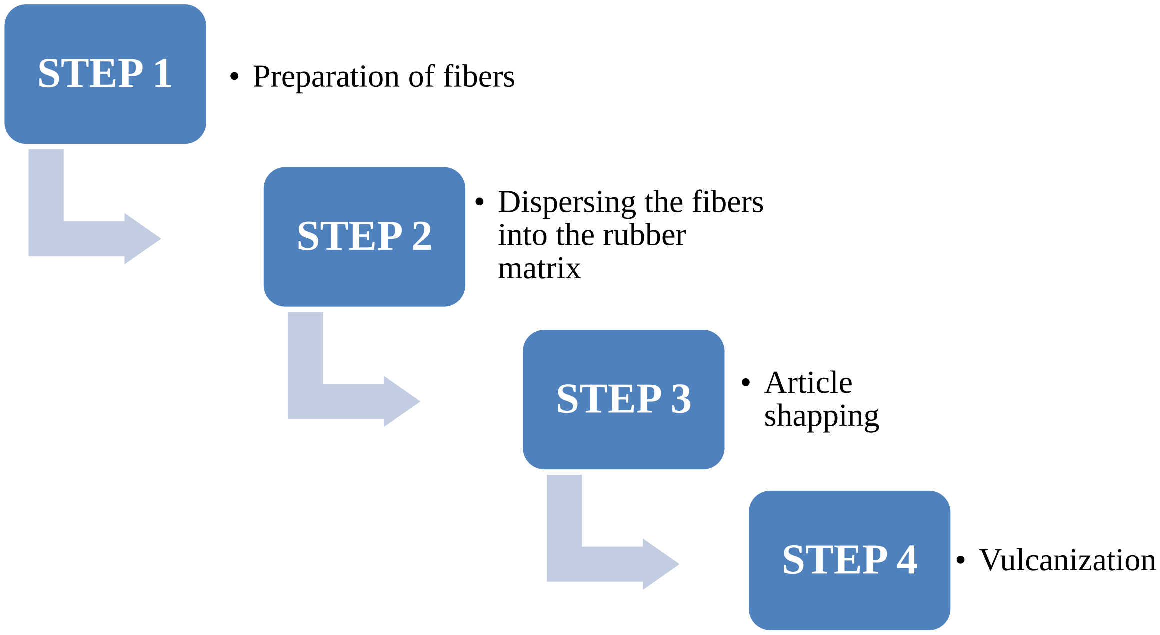

Figure 2.9 Process flow chart of Product Manufacturing with short fiber

2.10 Drawback of short fiber loaded compounds in processing

The short fiber compounding is done in ordinary processing machineries (Floter, 2007). When, adding more and more fibers into compound, it is negatively influence on processing in conventional machinery due to following drawbacks. Therefore, it is very important to consider following properties at compound development stage.

- Yield strength and modulus : It is an important factor when handling a rubber stock in the milling process
- Cut Resistance: It might be so high with fiber reinforcement that the mill rolls have to be stopped to allow the worker to cut off the sheet (Foldi, 1996)
- Tack: the ability for the compound to stick to itself, is somewhat reduced by the presence of fiber above 5-10phr range.
- Mooney Viscosity: A measure of how easily the green rubber will fill a cavity mould upon heating. (Foldi, 1996)

2.11 Reinforcing Mechanism of Short Fibers

For the continuous fiber and rubber composite, the following equation and relationship can be derived.

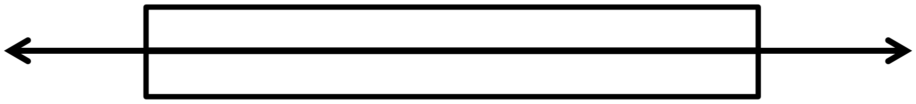

Figure 2.10 Force apply on continuouse Fiber

When a tensile or compressive load is applied parallel to the fiber direction, the strain in composites, fiber and rubber matrix are equal at the perfect condition of adhesion between fiber and rubber matrix.

$\in C = \in F = \in M$ -> Equation 1

If it is assumed that both fiber and rubber matrix behave elastically, then we can write the following equations.

$\sigma F = EF\ \epsilon F$ -> Equation 2

$\sigma M = \ EM\ \epsilon M$ -> Equation 3

Where σF and σM is the stress generated in the fiber and rubber matrix, and EF and EM are elastic modulus of fiber and rubber matrix respectively.

Considering equations 2 and 3

$\sigma F/EF = \sigma M/EM$

Generally, elastic modulus of fiber is greater than the elastic modulus of rubber matrix. Therefore stress developed in fiber is higher than the rubber matrix. As a result of this, fiber can bear a major part of the applied load in the fiber-rubber composite.

When we come to short fiber and rubber composites, the situation is different. Aspect ratio of the short fiber is considerably lower than that of the continuous fiber. The end effect of long fibers can be neglected but its effect is more significant in the short fibers. As a result of the reduction of fiber length, the performance of the fiber-rubber composites will drop and can cause a premature failure in the composites (Kavita Agarwal, 2002).

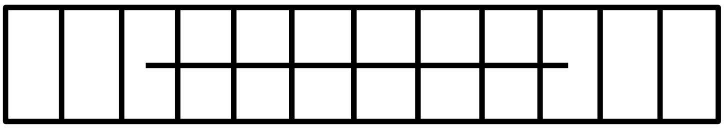

Figure 2.11 Short Fiber in a Rubber Matrix

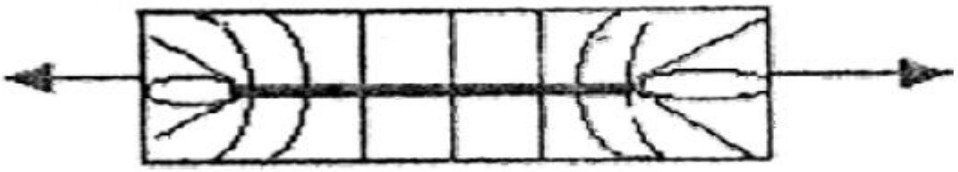

Figure 2.12 Force Apply on a Short Fiber

As shown in Figure 2.11 Short Fiber in a Rubber Matrix, a single short fiber is embedded in a low modulus matrix. At the unloaded condition, the imaginary lines running perpendicular to the fiber direction is unchanged. The fiber and rubber matrix will experience different strains due to their different moduli. When the composite is loaded axially, the longitudinal strain in the rubber matrix will be higher than that in the adjacent fiber due to lower modulus. When a force is applied, the imaginary vertical lines in the short fiber composite will be distorted as shown in Figure 2.12 Force Apply on a Short Fiber. The longitudinal strains create a shear stress distribution around the fibers in the direction of the fiber axis and the fiber is stressed in tension. The applied load is transferred from matrix to fiber across the interface because of this shear stress distribution.

When a mechanical force is applied to the polymeric material, it spreads smoothly through the matrix until it reaches the fiber-matrix interface. If the interface is strong or well bonded, the stress is transferred into fiber and spread throughout the fiber. This process occurs at all the short fiber-rubber composites and the perfect bond between two components enhances the reinforcing efficiency.

3 CHAPTER 03 - EXPERIMENTAL

This chapter is allocated for defining the material used, the method of sample preparation and experimental techniques adopted in the investigation.

3.1 Material

In the formula, Natural Rubber (RSS 3) was used. It was obtained from Thai Hua Rubber Company – Thailand. The molecular weight, molecular weight distribution and non-rubber constituents of natural rubber are affected by clonal variation, season and method of preparation. Therefore, the natural rubber was taken from same batch to minimize the variation.

In this study N339 carbon grade was used as filler and was taken from an approved supplier name Phillip India. Silica was also used in this formulation as fillers and it was also taken from SKI Carbon - India.

Softeners are used in rubber compounds principally as processing aids and to improve tack or stickiness of vulcanized compound. Petroleum oil is common processing aid in rubber compounding. In recent years the carcinogenicity of polycyclic aromatic hydrocarbons comes to be regarded as important. Oils containing 3% or more of polycyclic aromatic compounds (PCA) are obliged to indicate that they are toxic and there is a trend towards regulation of their use. For this study, the oil that contain less than 3% PCA was used and it is called low PAH oil. The low PHA petroleum oil was taken from Nynas – Sweden.

Waxes, antioxidants and antiozonants are added to rubber compounds to help protect compound against deterioration by ozone, oxygen and heat. 6PPD and HS were used as antioxidants and was taken from Sinorghen - China.

During vulcanization or curing, the polymer chains become linked, transforming the viscous compounds into strong, elastic material. Sulfur along with accelerators and activators help achieve the desired properties. Sulphur was used as curing agent because of its easy available & affordable of price. Sulfur is commonly used in tire industry and it is used in more than 80% of dry rubber compounds. The material was supplied by Miwon Commercial – Korea. High purity Zinc Oxide was used as activator because the content of metal of copper, Manganese and Ferrous can cause degradation of polymer. This interference may lead to affect final results of study.

Accelerators speed up the cross linking reaction but it negatively influence on processing. With faster accelerators, the cross linking process may begin in the heat history during the processing of compound and is often termed premature vulcanization. Pre Vulcanization Inhibitor (PVI) was used in experiment to avoid premature vulcanization risk in compounding. (Yanggu Hutai – China)

In addition to that, Nylon 6 materials were used as short fibers and it was taken from supplier called Junma – China. The material was cut to approximately 5 mm in length to easy incorporation of other ingredients.

More attention was given to choose specific material sources throughout the experiment to ensure the minimum influence of material quality on final results. Also, when the experiment was designed, the materials were reserved from same batch to minimize batch to batch variation. The specifications of the materials are in ANNEX 1.

3.2 Preparation of Compound

3.2.1 Compound formulation

In order to match with the research objectives, compound formulation was developed. Only short-fiber content was varied while keeping other ingredient constant to see the effect of short fiber alone. The base formulation was derived from a standard industrial solid tire tread compound.

The formulas used in the preparation of the different short fiber loaded compounds are given in following **Error! Reference source not found.**.

Table 1 First Stage Compound Formulation

Material	Parts Per Hundred Rubber
RSS 3	100
Carbon Black - N339	58
Zinc Oxide (ZnO)	5
Stearic acid	2
Antioxidant & Antiozonant - 6PPD	2
Antioxidant – TMQ (Vulkanox HS)	1
Low PAHS rubber process oil	5
Bonding agent – Resorcine 80 SBR	1.5
Precipitated Silica filler	5
Nylon 6 short fiber	0, 1.0, 1.5, 2.0, 2.5, 3.0, 5.0, 10.0, 15.0, 20.0

Table 2 Second Stage Compound Formulation

Material	Parts Per Hundred Rubber
Sulphur	2
Accelerator - TBBS	1
Pre-vulcanization inhibitor CTP (PVI)	0.3
Accelerator - HMT	0.4

In every formulation, around four test samples were prepared and tested. The average of each samples were taken as the final results. In addition to that, the compound that is currently used for this application was used as a reference samples..

3.3 Processing

3.3.1 Mixing in Internal Mixer

Mixing was done in Farrel BR1600 Banbury mixer. In developing a rubber compound, it is essential to mix the raw dry rubber with various compounding ingredients. The process of sequentially adding the ingredients into the raw rubber is termed ‘compounding’ and the resulting final homogeneous mix is referred to as the ‘rubber compound’.

The mixing process was done in two stages as the 1st stage and the 2nd stage. During the 1st stage mixing, dry rubber, carbon black, short fibers and other ingredients were added under the pre-defined sequence. In the 2nd stage mixing, the entire curatives were added. At the time of designing the experiment, mixing time was defined and all compound samples were prepared according to the mixing time.

3.3.2 Compound Mixing Cycles

The sequence of the mixing is called the “mixing cycle” and it was carried out in two stages. It is called 1st stage mixing cycle and 2nd stage mixing cycle. Mixing cycle was designed by considering the proper incorporation of short fibers into the compound

The mixing cycle that used in the preparation of compounds is given in **Error! Reference source not found.**.

Table 3 First Stage Compound Mixing Cycle

RAM DOWN TIME /(Sec)	TOTAL TIME /(Sec)	DESCRIPTION
0	0	RSS + 1/2 Short Nylon Fiber
60	65	Carbon + Chem + Oil + 1/2 Short Nylon Fiber
60	165	Ram Cleaning
60	213	Dump

2nd Stage Mixing Cycle

Table 4 Second Stage Compound Mixing Cycle

RAM DOWN TIME /(Sec)	TOTAL TIME /(Sec)	DESCRIPTION
0	0	1st Stage compound
30	35	2nd Stage Chemical add
60	70	Dump

3.3.3 Mixing on Mill

Mill mixing was done on two roll laboratory size mixing mill. Mill size is 200 X 400 mm. speed of the mill rolls can be adjusted independently and 1:1.2 friction ratios was used for the experiment. The lump taken from the internal mixer was passed six times through the tight nip to get a smooth band on mill rolls. Finally, the compound was sheeted out at a fixed nip gap so as to orient the fibers preferentially in one direction.

3.3.4 Cure Characteristic

Cure Characteristics were measured by using Alpha Rheometer (ODR 2000). The machine has two directly heated, opposed biconical dies that are designed to achieve a constant shear gradient over the entire sample chamber. The specimen was rectangular in shape and kept at the top and bottom of the oscillating die. The oscillating angle is +/-3 deg and a 150 degree centigrade temperature was maintained in the mould cavity. The specimens had to under-go 30 minute testing time. The test was done according to the ASTM D 2084. The torque transducer on the upper die sense the force being transmitted through rubber and following graph is taken in the test.

The following data can be taken from the graph

- Maximum Torque (M_H) : It is the torque recorded when curing of the compound is completed
- Minimum Torque (M_L) : It is the lowest torque shown by the compound at the test temperature before the onset of cure
- Scorch Time (t_{S2}) : It is taken as the time for 2 units rise in torque from the minimum torques
- Cure Time (t_{90}) : The optimum cure time corresponds to the time to achieve 90% of maximum cure

3.3.5 Moulding and Vulcanization of Test Specimens

Test specimens were prepared in both the milling direction and the transverse direction. Vulcanization was done at 150 +/-2 ^{0}C and at a pressure of 180 kg/cm^{3} in an electrically heated hydraulic press to their respective cure times. The curing times are defined according to the geometry of specimens (width, thickness etc.).

3.4 Physical Properties

Physical properties of different fiber loadings were tested and the tensile strength, elongation at break, modulus, tear strength, loss modulus, storage modulus, tan δ, abrasion resistance, compression load and cut and chip resistance values obtained experimentally. Tests were done to determine how the fibers were oriented along and across the sample length.

3.4.1 Determination of Tensile Strength, Modulus and Elongation

The tensile testing was done in Gotech GT-7010 - AE testing machine according to the ASTM D 412 A. The samples were punched out from the moulded sheets both along and across the fiber direction. The shape of the specimen is called dumb bell. The schematic representation of fiber orientation in tensile samples (Dumb bell) is given in Figure 3.1 Fiber Orientation in Tensile Test Specimen. The Test Pieces are acclimatized at the test temperature 27 +/- 2 ºC for 24 hours before testing.

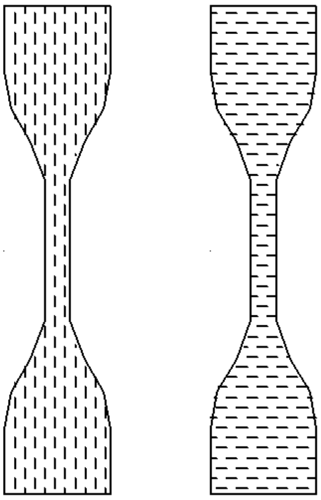

Figure 3.1 Fiber Orientation in Tensile Test Specimen

The tensile properties of all rubber compounds were tested at room temperature. The specimen's ends were firmly clamped and the one inch distances were marked on the specimens. The rate of grip separation speed was 500 mm/min. During the test, loads and elongation were taken as direct measurements and the following equations were used to calculate other results:

$$\text{Tensile Strength } (kg/cm^2) = \frac{\text{Load at Break}}{\text{Original cross section area}}$$

$$\text{Modules at 300\% Elongation } (kg/cm^2) = \frac{\text{Load at 300\% Elongation}}{\text{Original cross section area}}$$

$$\text{Elongation at Break (\%)} = \frac{(\text{Length at Break} - \text{Original length}) \times 100}{\text{Original Length}}$$

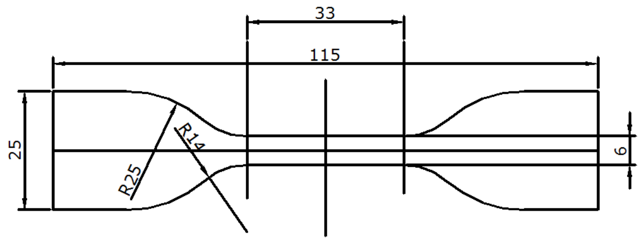

Figure 3.2 Dimensions of Dumb Bell

3.4.2 Determination of Tear Strength

The specimens were cut using the die cutter stand in accordance with ASTM D 624 – Die C. The cut was done along the fiber direction and in the transverse direction. The schematic representation of fiber orientation in tear samples is given in Figure 3.3 Fiber Orientation in Tearing Test Specimen.

The specimen was conditioned at least 16 hours before starting the test. The forced required propagating at 90 deg original angle of the test piece was measured as direct measurements and same tensile machine (Tensiometer) was used for this test. The test speed was 500 mm/min and test was done until the test specimen was broken. The thickness measurements were done to the 2nd decimal point in centimeter.

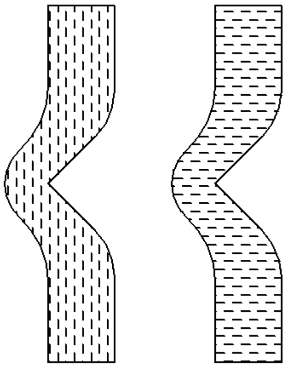

Figure 3.3 Fiber Orientation in Tearing Test Specimen

The following equation is used for calculating tear strength.

$$\text{Tear Strength (kg/cm)} = \frac{\text{Maximum Load at Break (kg)}}{\text{Thickness of specimen (cm)}}$$

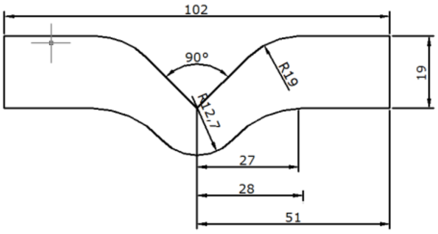

Figure 3.4 Dimensions of the Tearing Piece

3.4.3 Determination of Density

Density of the samples was measured according to the ASTM D 297. Alfa Mirage MD 300S Electronic Densimeter was used for the test. The samples were made free of porosities or air entrapped and weight of the sample was around 7 g. A direct measurement is given in Electronic Densimeter.

Indirectly, we can calculate the density by taking the weight of the samples in the air and in water. Density of the sample was calculated by using following equation:

$$\text{Density} = \frac{\text{Weight of the sample in Air x Density of Water}}{\text{Loss of weight in water}}$$

3.4.4 Hardness

Hardness is the property of a material that expresses the degree of resistance to scratching, puncturing or deformation. Hardness is measured in Shore Hardness, International Rubber Hardness (IRHD) or British Standard (BS).

Resistance to indentation of rubber sample was measured by using the Durometer Rubber hardness tester. ASTM D 2240 test procedure was followed to measure hardness of test samples.

3.5 Mechanical Properties

3.5.1 Determination of Abrasion Resistance

Abrasion resistances of the samples were measured using a DIN abrader according to ASTM D 5963 (Testing Method B and Calculation Method A). A sample having a diameter of 16 +/- 2 mm and a thickness of 7 mm was placed on a rotating holder. The specimen was conditioned at least 16 hours under room temperature after curing the compound sheet. A load of 10 N was applied on the specimen against ~~to~~ a rotating drum. The abrasion resistance is measured by moving a test piece across the surface of an abrasive sheet mounted to a revolving drum. The special abrasive sheet is made of Aluminum Oxide of grit 60 bonded to a sheet in average thickness of 1 mm. A standard rubber specimen is used to calibrate the abrasive sheet to ensure that the value is within the specified range of 180 mg to 220 mg. The sample weight is taken before the test and the final weight is taken after the test. The weight measurements were done to the nearest 1mg. The difference in weight is the weight loss of the test piece after its travel through 40m abrasive distance. The results are expressed as volume loss in cubic millimeter. The rotating speed of the drum is 40 +/- 1 rpm and diameter of the drum is 150 +/- 0.2 mm. Three specimens were tested in each compound and the average value was taken.

$$\text{Abrasion Loss } A_A = \frac{\Delta mt \text{ X } S_0}{dt \text{ X } S}$$

Where,

A_A - Abrasion loss in mm^3

Δmt – Mass loss of the test piece in mg

dt - Density of the test compound in mg/mm3

S_0 - Nominal Abrasiveness = 200 mg

S – Abrasiveness in mg

3.5.2 Cut and Chip

.

The Cut and Chip Tester is designed to measure the relative service life of off-the-road, farm and heavy duty treads that are subjected to the rigors of traveling on hard and rough surfaces. It also measures the chipping and tearing of the tread when subjected to off-the-road surfaces by reduction in sample weight due to chips. The Cut and Chip Tester provides a simple, quick laboratory method to evaluate cutting and chipping resistance of rubber compounds subjected to harsh mechanical environments. Rpm 750 for sample rotating test time 10 min.

Cylindrical shape specimen is used. Diameter of the sample is around 50 mm and thickness is 13+-0.5 mm.

% weight loss calculation

$$\%\ \text{weight loss} = \frac{(\text{Initial Weight} - \text{Final Weight}) \text{ X } 100\%}{(\text{Initial Weight})}$$

% Diameter reduction calculation

$$\%\ \text{Diameter reduction} = \frac{(\text{Initial average diameter} - \text{Final average Diameter}) \text{ X } 100\%}{(\text{Initial average diameter})}$$

3.5.3 Rebound Resilience

Rebound resilience is the ratio of energy returned to energy applied. Rebound resilience shall be measured using a one-degree-of-freedom mechanical oscillatory device. The device consists of a stand with an anvil, a test piece holder, a pendulum with an indenter and an indicating device. The pendulum consists of an arm and a hammer incorporating an indenter with a hemispherical surface. The pendulum is suspended so that it oscillates circularly under the effect of gravity. During the test, the arm is raised an angle of 90^0 from its rest position and released. With the arm in the vertical position, the indenter shall just touch the test piece surface, the direction of impact of the indenter being perpendicular to the test piece surface.

3.6 Set Properties

3.6.1 Compression Load

ASTM D575 is a testing specification that determines the compression-deflection characteristics of rubber compounds. This test method is applicable to all rubber compounds other than hard rubber and sponge rubber.

Deflection is the change in thickness of the specimen upon application of a compressive force. This method involves one kind of compression stiffness measurement.

Procedure A: Compression Test of Specified Deflection- A compression test in which the force required to cause a specified deflection is determined. The dimensions of the test specimen are 300 mm height and 600 mm in diameter.

3.7 Dynamic Properties

3.7.1 Dynamic Mechanical Analysis (DMA)

DMA was done using a circular test specimen. The test was done under Compression mode. Heat rate is 2 ^{0}C/min. The frequency was 1 Hz under temperature sweep mode at a rate of 2 ^{0}C/ min. The samples were subjected to dynamic strain amplitude of 0.05%. The dynamic storage modulus (E’) and loss modulus (E”) of the composites were monitored. Test specimen was cylindrical in shape and height & diameters were 25 and 16.5 mm respectively. Temperature range is 30 ^{0}C 100 ^{0}C

3.7.2 Heat Build-up

BF Goodrich Flexometer was used to carry out the heat build-up as per the ASTM D-623 - Method A. The test specimen was in cylindrical shape. Diameter of the sample was 17,8 +/-0.1 mm and height was 25 +/-0.15 mm. Specimen was conditioned at least 24 hours under the room temperature after the curing.

Oven temperature was kept constant at 100 ^{0}C. The frequency was set 30Hz. The temperature of heat chamber was set at 100 ^{0}C and kept to stabilize the temperature. Then the samples were preconditioned at the test temperature in the oven for 30 minute prior to the test. Then the specimen was placed on base and allowed to cool down to 96 ^{0}C. The heat development at the base of the sample was sensed by a thermocouple and relayed to a digital temperature indicator. The temperature rise at the end of the specified time interval was taken at the heat buildup.

There are two testing procedures. They are the standard test and blow-out tests.

In the standard test, the stroke height is 4.45 mm and load on specimen is 245 N. The testing time is 30 minute and the temperature difference was taken at the end of the test.

In the blow out test, the stroke height is 6.35 mm and load on specimen is 489 N. The test is done until the specimen blows out. At the end of the test, the temperature difference is measured.

4 CHAPTER 04 - RESULTS AND DISCUSSION

This chapter focuses on a systematic analysis of the results obtained in the study and hence interprets the performance of short fiber loading.

4.1 Rheological Properties

Oscillating disc rheometer and Mooney viscometer were used for measurements of the curing properties. The initial and final Mooney, maximum and minimum torques, scorch time, optimum curing time etc. are evaluated in the following graphs.

4.1.1 Mooney Viscosity

Final Mooney viscosity of 2nd stage compound

The results of Mooney viscosity measurements give a good indication of processability of the compound. Generally, the higher the value of Mooney viscosity, the greater is the power or energy required to process the material in extrusion, injection moulding, milling etc. however some caution should be used in relating Mooney viscosity to the behavior of compound in the extrusion or injection or milling process because the shear rates uncounted are much greater as compared to Mooney viscometer results. The measuring conditions in Mooney viscometer were defined such a way that the preheat time 1 minute and a set time of 4 minute at 100 ^{0}C with small rotor, which is expressed as MS (1+4) @ 100. Small rotor was used due to the stiffness of compound. Mooney viscosity is the results obtained from Mooney viscometer and the values indicate processability of the compound.

According to the Figure 4.1 indicate that the Mooney viscosity gradually drop to a certain fiber loading level and again increase with increasing loading of short nylon fibers. At the initial loading of short fiber, the Mooney viscosity value of the compound drops upto 5 phr loading and the lowest torque was shown at 5 phr. It indicates, the stiffness of the compound gradually drops with the fiber loading and it is a great help for processing of the compound. As mentioned in previous paragraph, energy consumption for the processing of compound will reduce and it is a great economical advantage. The results shown in Figure 4.1, the Mooney viscosity increases with increasing fiber loading beyond 5 phr. In the low loading of the short fiber, natural rubber is the predominate factor for controlling the compound stiffness and at a certain level of fiber loading, the fiber acts as the predominant factor for compound stiffness. This is an expected result because of the short fibers increases the stiffness of uncured rubber composite which leads to greater resistance against the shearing action of the rotor of the Mooney viscosity. .

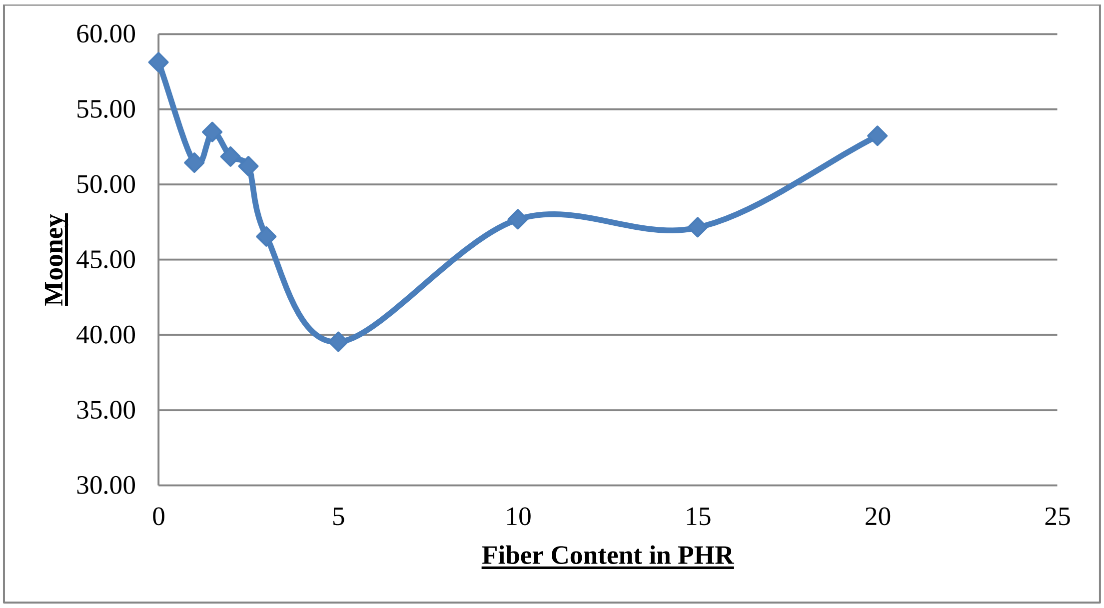

Figure 4.1 Final Mooney Viscosity

4.1.2 Rheometer Properties

Minimum Torque

Variation of minimum torque (M_L) with short fiber loading is shown in Figure 4.2. As shown in the graph, the minimum torque decreases with fiber loading up to 5 phr and again it starts to increase. The increasing or decreasing of torque, which is a scale of interior shear stress, is due to the physical and chemical reactions of the composites. It indicates the reinforcing effect. In the control sample, the ML is closer to 30 dNm and it is gradually decreases closer to 20 dNm at 5 phr fiber content. When we increase the fiber content proportionally, the rubber content was reduced. As a result of this, the ML ~~is~~ gradually drops up to 5 phr. With lower fiber content, the ML is dominated by the rubber and reinforcing fibers that act as the network defects. At higher fiber content, fiber dominates the properties and ML increases with the fiber content. (Lee D.J. and Ryu, 2008)

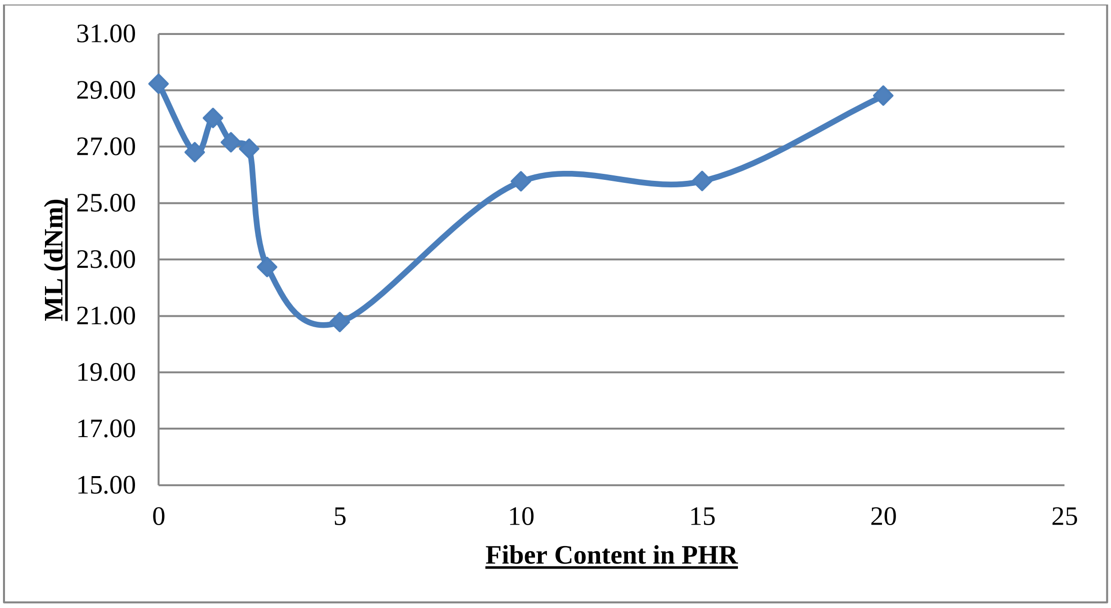

Figure 4.2 Vulcanizing Properties - ML

Scorch Time (t_{s2})

The scorch time is the time required at a specified temperature for a rubber compound to form incipient cross links. When a scorch point is reached after a compound is exposed to a given heat history from factory processing, the compound cannot be processed further by milling, extruding, calendaring etc. Therefore, scorch measurement is very important in determining whether a given compound can be processed in a particular operation. Figure 4.3 gives the variation of scorch time with short fiber content. While increasing the short fiber content, scorch time also increases and it is good for scorch safety. The scorch time varies between 2.3 & 3.6 minute. The delayed start of cure reaction is also important to allow for the flowing of the material in every article in complex tread cavities in the mould before the reactions start.

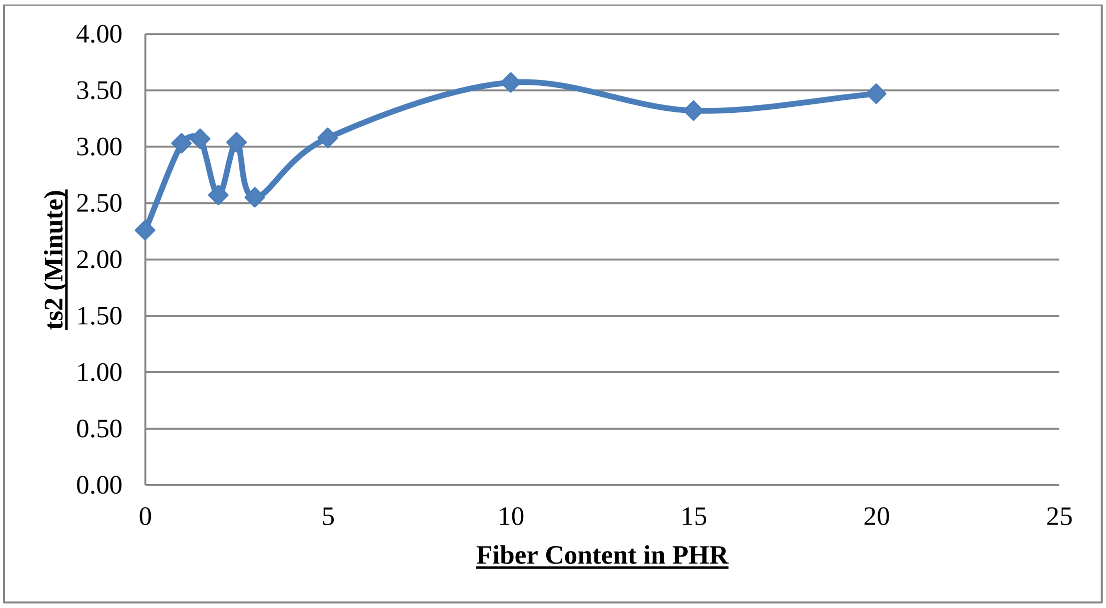

Figure 4.3 Vulcanizing Properties - ts2

Optimum Cure Time (t_{90})

This is the time taken for obtaining 90% of the maximum torque. As shown in the Figure 4.4, the optimum curing times of all formulations are more or less similar. Therefore, there is no risk in extending the curing cycle while adding the short fibers.

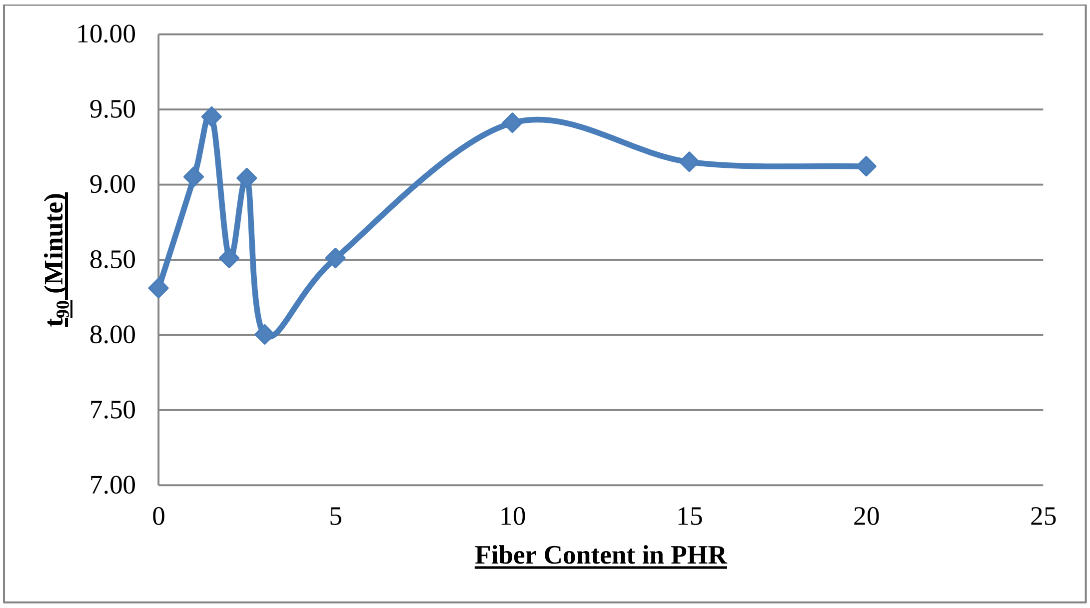

Figure 4.4 Vulcanizing Properties - t90

4.2 Microscope Studies of the compound samples

The orientation of the fiber was accomplished by compound milling operation and it is helped to enhance the performance. Microscope studies were done to identify the orientation of the fibers in the compound.

Figure 4.5 is shown the orientation of the in milling direction. The microscope pictures were taken from the samples that made with 2phr short fiber loading.

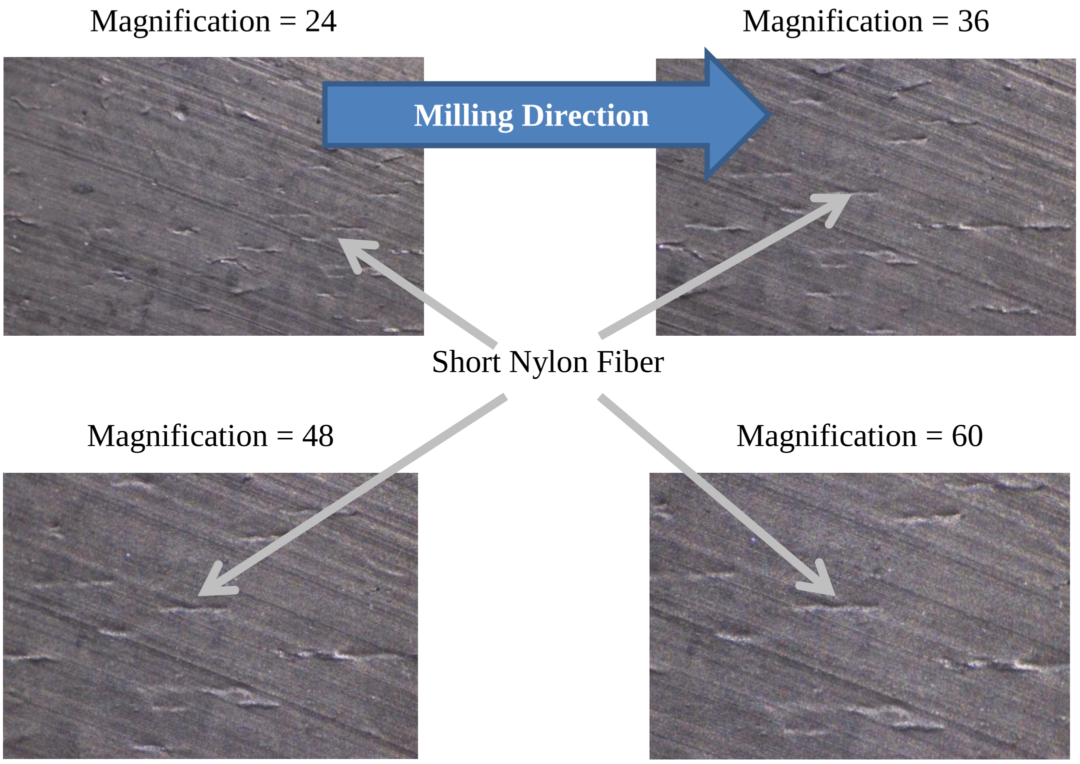

Figure 4.5 Microscope Picture in Different magnitudes

4.3 Physical Properties

4.3.1 Tensile Strength

Figure 4.6 shows the variation of tensile strength with fiber loading. The tensile strength is almost linearly decreased with fiber loading. The highest tensile strength was observed in gum compound that does not consist of short fiber. The tensile test was done in both longitudinal and transverse directions of compound sheet. The fiber-rubber matrix interface adhesion is important for the tensile strength and most probably, the reason for the decreasing tensile strength is poor adhesion in the interface. The interface adhesion can be improved by mechanical interaction as well as by chemical interaction. Previous studies were shown that the influence of a bonding agent to improve the short fiber–rubber matrix interfaces strength (Lee D.J. and Ryu, 2008). They have demonstrated that the dry bonding system consisting of hydrated silica, resorcinol and hexamethylene became the best interface bond model. In a similar way, a dry bonding system was used in compounding in this study, but it was not performed well. Therefore, we can see a drop of tensile strength while increasing the short fiber loading in the compound. 150 kg/cm^2 is the minimum requirement of tread compound and up to 5phr, the minimum requirement is achieved in both parallel and transverse directions.

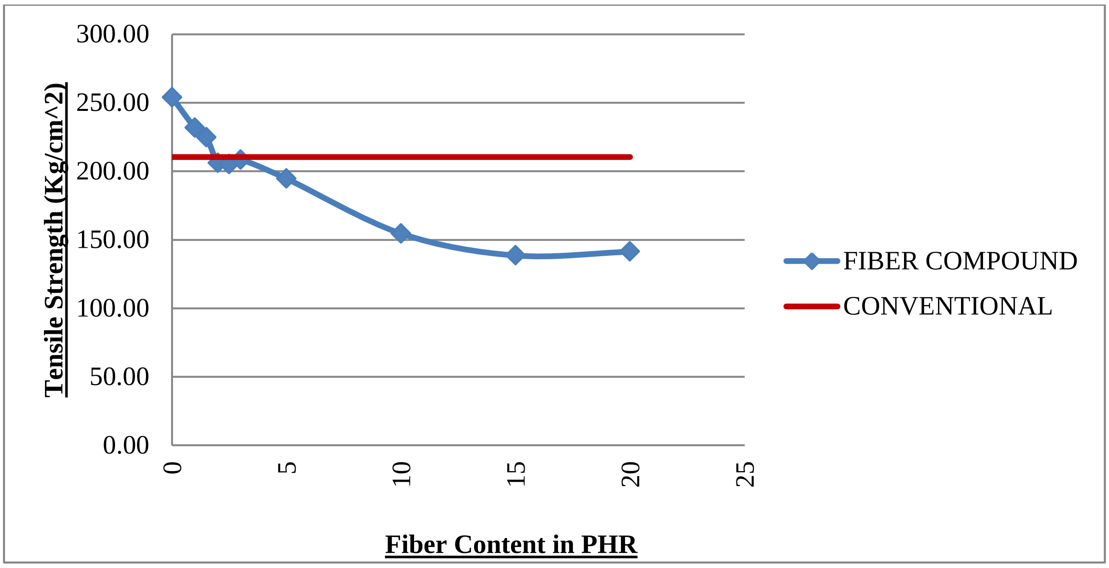

Figure 4.6 Tensile Strength - Longitudinal Direction

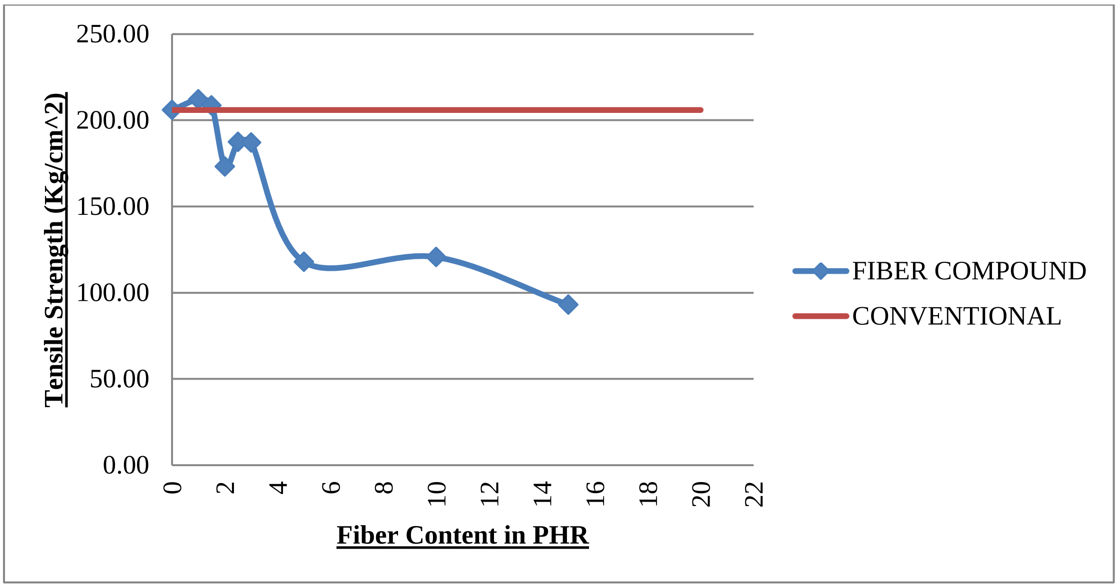

Figure 4.7 Tensile Strength – (Transverse Direction) vs fiber loading.

4.3.2 Tear Strength

Three types of tearing patterns were observed (Lee D.J. and Ryu, 2008). They are straight, stable and unstable. In the case of straight tearing, the tearing force fluctuates only slightly and the rate of tear propagation is basically constant and the cracks grow continuously. The stable and unstable tearing patterns are mostly observed in short fiber reinforced compounds. In the stable tear, crack propagation is advanced with a certain angle from the pre-crack direction. The angle of propagation is dependent on the fiber content. With the further increase of fiber content, unstable tearing patterns can be observed.

Figure 4.8 shows the tearing strength of different fiber loading in milling direction. The trend of the graph depicts that tearing strength was increased with short fiber content. Instead of steady tear propagation, the tear growth was irregular. It was observed that the crack arrested and re-initiated at fairly regular intervals. These irregularities increased with fiber content. The alignment of the fiber has interrupted the crack propagation of the short fiber reinforced rubber and as a result of this phenomena, the tear strength has increased with short fiber loading.

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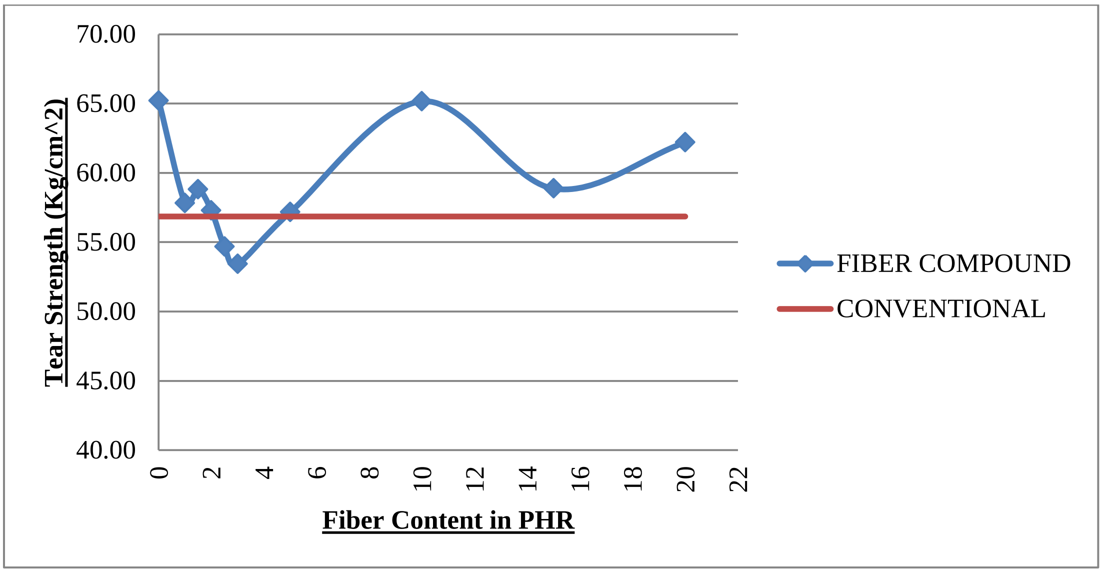

Figure 4.8 Tear Strength - Longitudinal Direction

Figure 4.9 shows the tearing strength distribution with the fiber loading in perpendicular direction to the milling direction. Tearing strength in perpendicular direction has different trend than the milling direction. As shown in Figure 4.9, the tearing strength was gradually increased and after 3 phr loading, it was decreased. When fiber volume fracture is low, there are more interactions between fiber & compound molecules and the fibers play the role of reinforcement. This is why a certain fiber loading, the tearing strength of the composite increase with the increasing of the fiber content. As the fiber loading become higher, there will be more interaction between fibers which may lead to reduce the bond. Hence, the tearing strength starts to decrease as fiber loading increase above a critical value. But, all fiber reinforced compounds have higher tearing strength than the control that was formulated without short fiber.

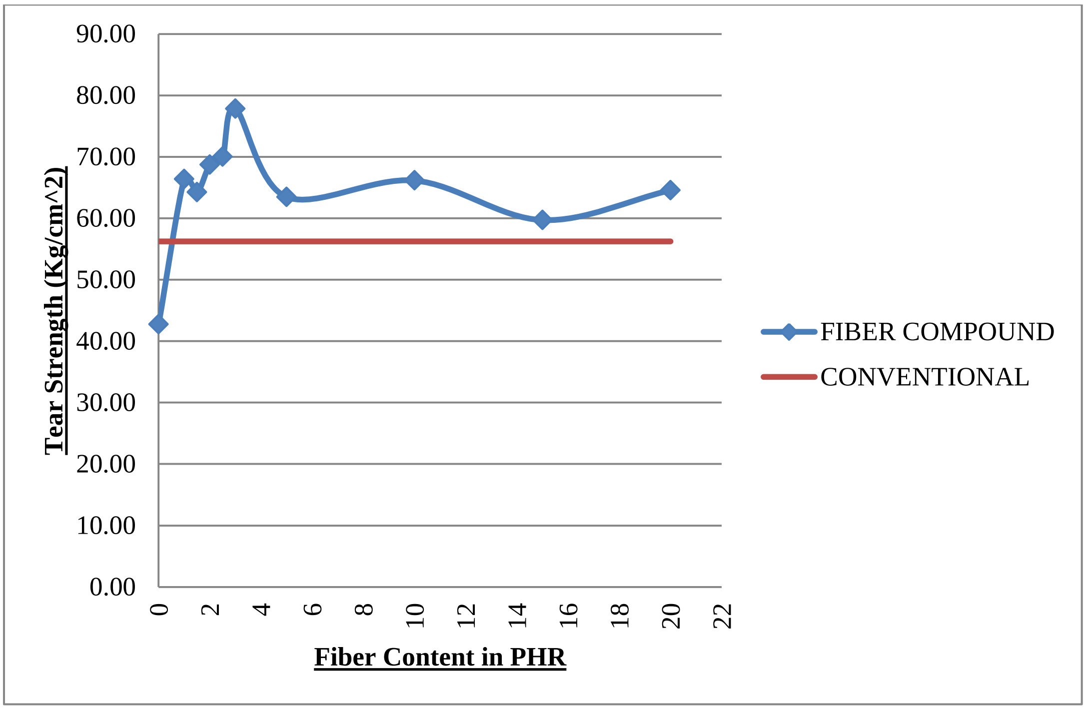

Figure 4.9 Tear Strength - Transvers Direction

4.3.3 Modulus

Figure 4.10 is given the 300% of modules. With the fiber loading, the modules will drastically drop due to the stiffness of the compound. Further increase of short fiber, the 300% modulus is not achieved beyond 10 phr compound. .

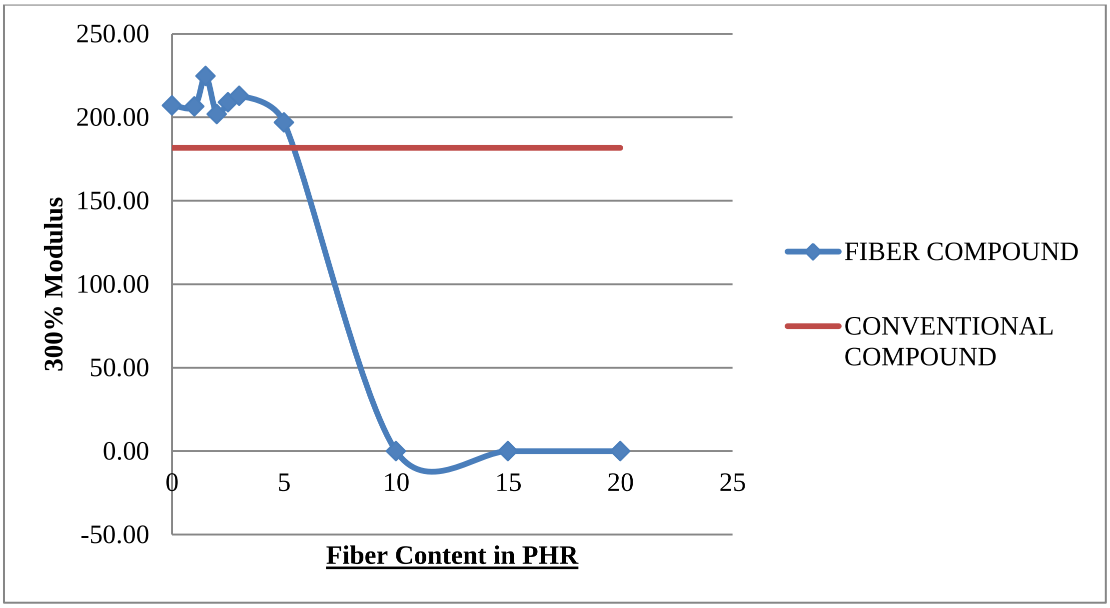

Figure 4.10 300% Modulus

4.3.4 Elongation at Break

Longitudinal Direction

Figure 4.11 shows variation of elongation at break in longitudinal direction with short fiber loading. The elongation at break values were decreased with the increase in short fiber loading. There is a gradual reduction from 354 at control (without fiber) to 83 at 20 phr of short fiber loading. There is a drastic drop 10 hpr onward. The presence of poor adhesion between fiber – rubber matrix interface causes the sample to fall at low elongation as it also evident from the lower tensile strength. The drop of the elongation in longitudinal direction is 77%.

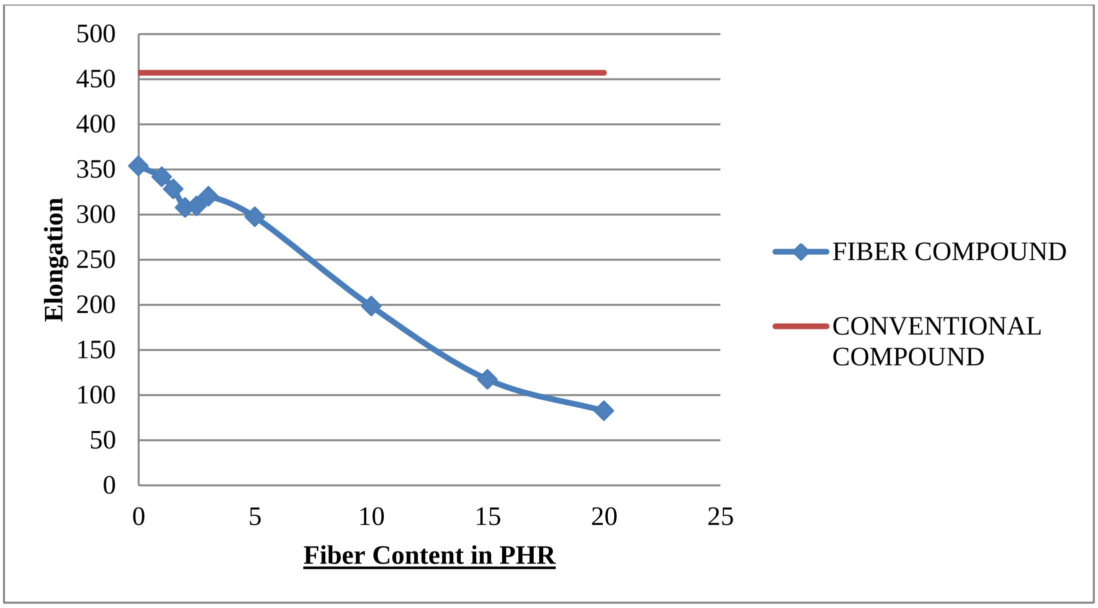

Figure 4.11 Elongation at Break - Longitudinal Direction

Transverse Direction

Figure 4.12 shows the elongation at break in transverse direction with the short fiber loading. The value was decreased with the loading of short fiber and it was dropped from 336 at control to 170 at 20 phr short fibers loading. The percentage of elongation drop is only 46%. Elongations at break values of the samples with transversely oriented fibers are higher than that of the samples with longitudinally oriented fibers. The fibers inhibit the orientation of molecular chains and hence the ultimate elongation decreases substantially on introduction of fibers.

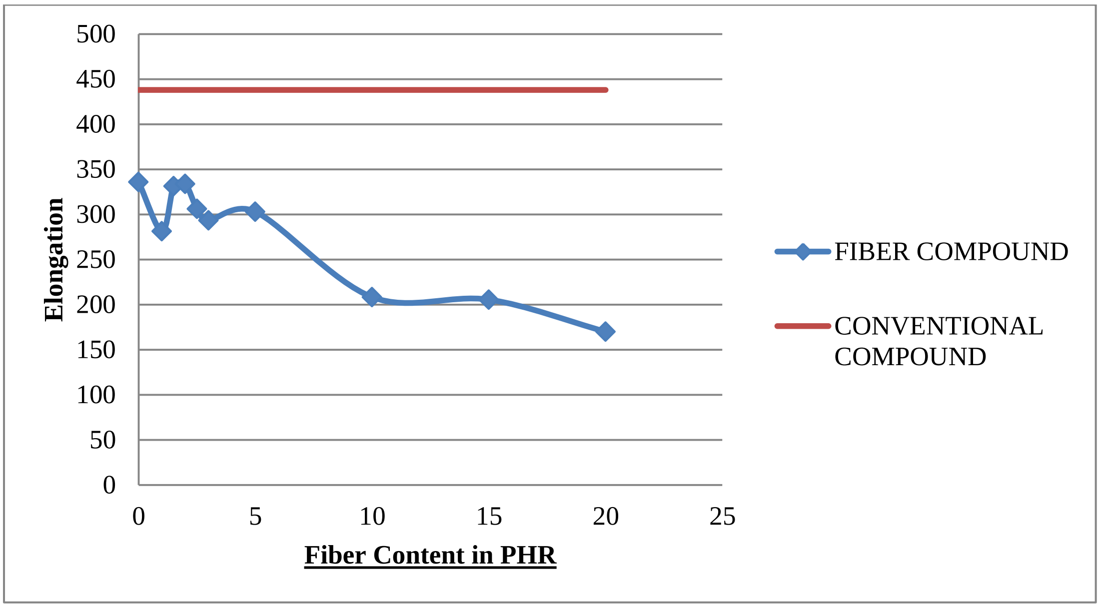

Figure 4.12 Elongation at Break - Transverse Direction

4.3.5 Hardness

The hardness measurements results were as predicted and did not render any unexpected changes. In general, compounds with higher fiber loading gave higher hardness. As shown in the Figure 4.13, the hardness was dropped by one unit compared to the control but it was recovered 3 phr onward. At low fiber loading, rubber matrix dominated the results but it was changed after 3 phr. Then, hardness was increased with respect to increase of short fiber loading.

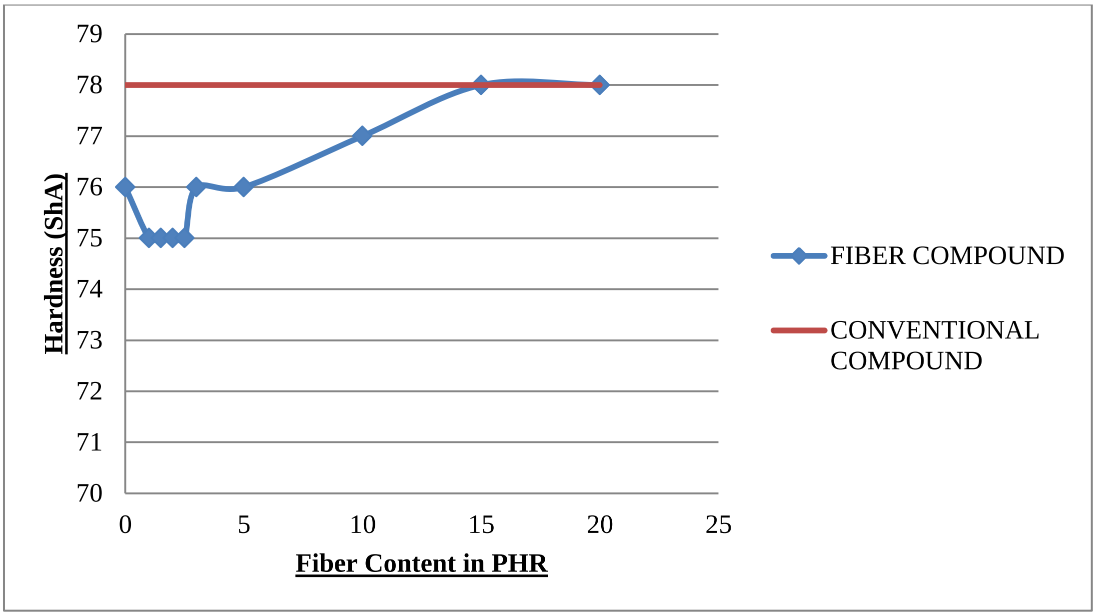

Figure 4.13 Hardness

4.3.6 Density

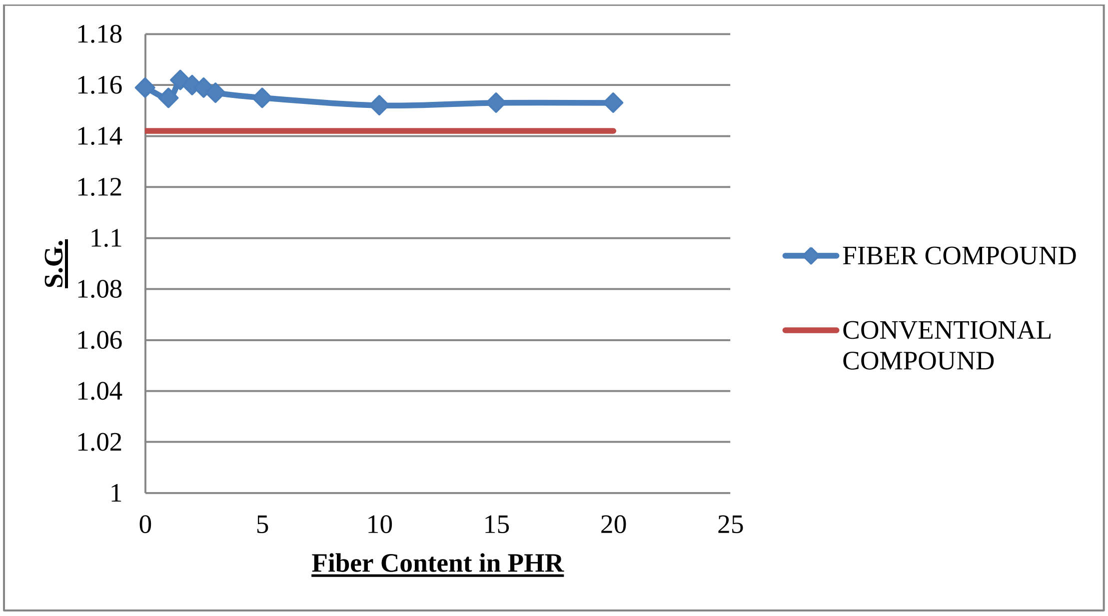

Figure 4.14 Specific Gravity of compound

The specific gravity of the compound was changed while increasing the fiber loading. It is almost similar and it is a good indication for predicting the product cost.

4.4 Mechanical Properties

4.4.1 Abrasion

Although the preceding section has emphasized the favourable effect of the short nylon fiber – rubber matrix on the tear strength, some judgement on the composites by determining their abrasion properties was considered as essential and relevant in servies of tyres. To obtain a useful measure of the rather complicated relationship between the fiber content and the abrasion characteristic, a series of DIN abrasion tests were done.

The results of the abrasion test are shown in Figure 4.15. The specimens were prepared according to the DIN procedure and an average volume loss of three samples were taken and a graph was plotted. All the specimens were prepared and tested under the same conditions. Lower the abrasion value, lower is the volume loss and it is more favorable for better performance. As shown in the graph, up to 3 phr, more or less of a similar volume loss was maintained and when compared with the the control and after that, the volume loss was rapidly increasing. Two important factors in studying abrasion behavior of rubbers are modulus and crosslink density. High modulus and crosslinking density lead to the enhancement of abrasion strength of rubber compounds [Ramin Z., et al.]. As shown in Figure 4.10, 300% modulus was around 200 Kg/cm^2 up to 5 phr fiber content and after that the compound became very stiff and did not achieve 300% elongation. As a result of high modulus, the abrasion resistance was poor in samples that were made in 10, 15 and 20 phr.

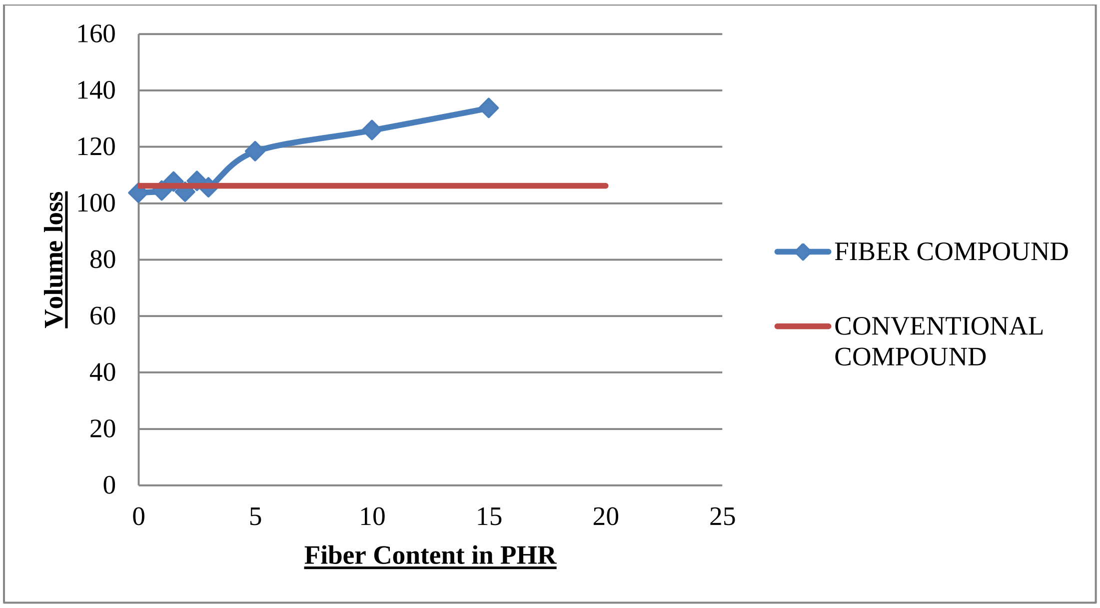

Figure 4.15 Abrasion

4.4.2 Compression Test

Figure 4.16 shows variation of compression set at constant strain. The compression set increased with the increasing of fiber concentration but it ~~is~~ adversely affected the elongation properties.

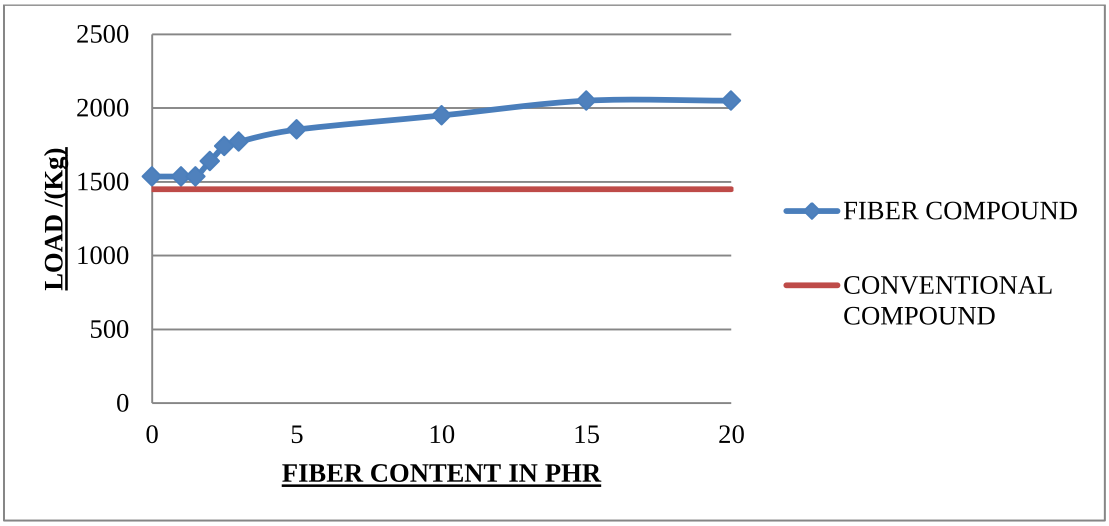

Figure 4.16 Compression Test

4.4.3 Rebound Resilience

Rebound resilience is the ratio of the energy of the indenter after impact to its energy before impact expressed as a percentage. As shown in Figure 4.17, the rebound resilience values did not show significant effect on different loading of short fibers. However, this could only be true up to 10 phr fiber loading but a slight drop could be seen after that. At 15 phr fiber loading, the hardness increases by 1. Perhaps this could be due to the increase of hardness value and hence would have contributed to the lower value of rebound resilience. (Rios S.R., 2001) found that there was an inverse relationship between hardness and rebound resilience. If the hardness achieved was high, the rebound resilience decreased because there were more points of slippage between the reinforcement and rubber matrix and also because of the reinforcing material tends to clump together so that particles touch one another instead of being totally embedded in the rubber matrix.

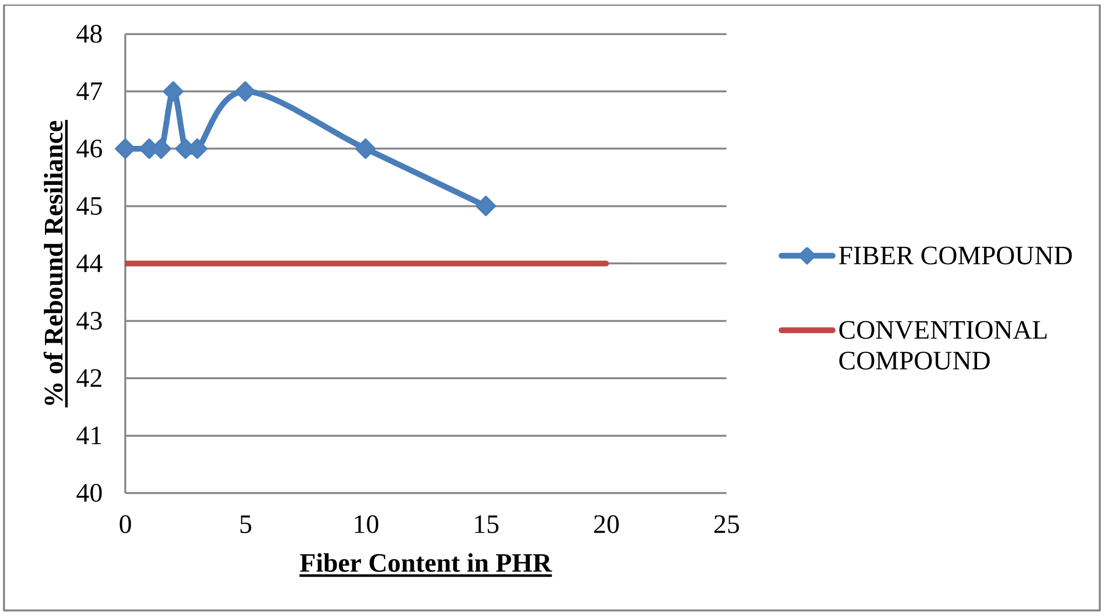

Figure 4.17 Rebound Resilience

4.4.4 Cut and Chip

The tires used in the industrial application are subjected to tread damage commonly referred to as cutting and often chipping, both of which are a major problem for such tires. Cutting takes place when the tire strikes a sharp object with such force that the surface is penetrated or cut. Chipping is the fragmentation of the damaged part of the tread usually after tearing. This test was planned to avoid lengthy and costly field test.

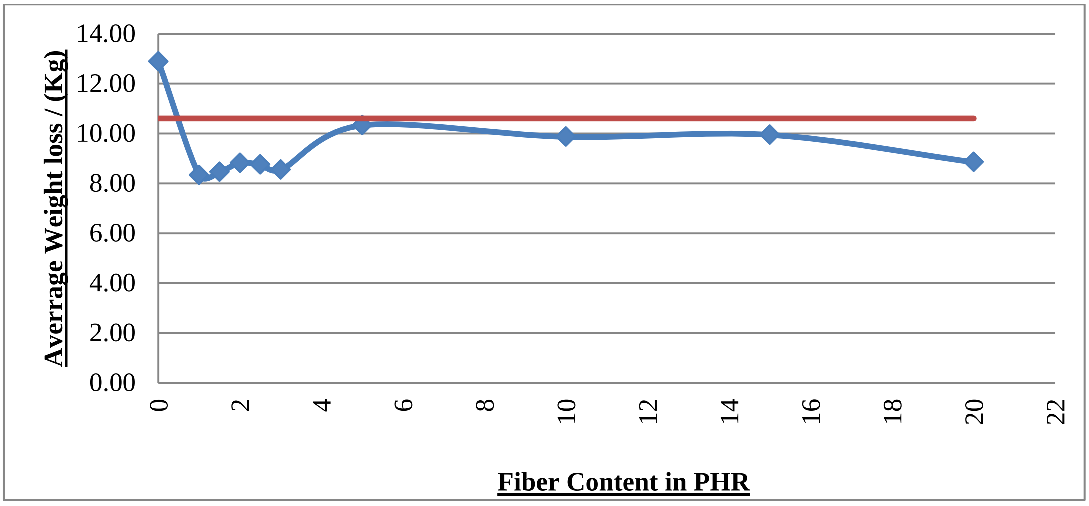

Figure 4.18 Average Weight Loss %

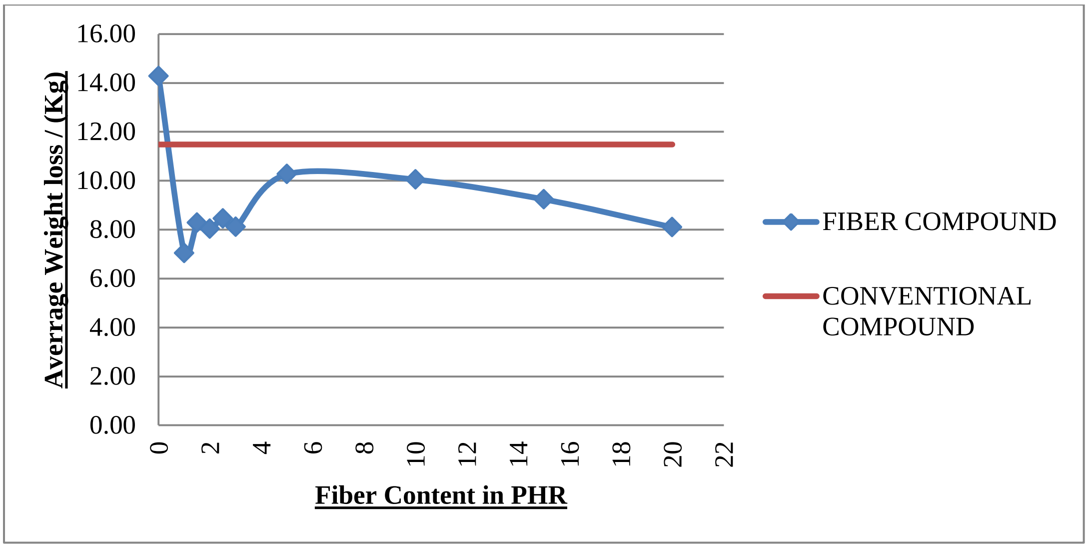

Figure 4.19 Average Diameter Loss %

4.5 Dynamic Properties

4.5.1 Goodrich Flexometer Heat Build-Up

The Goodrich Flexometer is widely used to determine the internal heat generation of a compound under flexing compression. Although the standard has pointed out that no simple correlation can be found between the accelerated test and service performance, several reports have revealed that a good prediction is possible. The Goodrich flexometer heat build-up values of different fiber loading are given in Figure 4.20. The heat build-up increases with increase in the amount of fiber in the composites. Due to the stiffness of the fiber loaded composites, it generates more heat since the dissipation of energy depends upon both stress and stain amplitude.

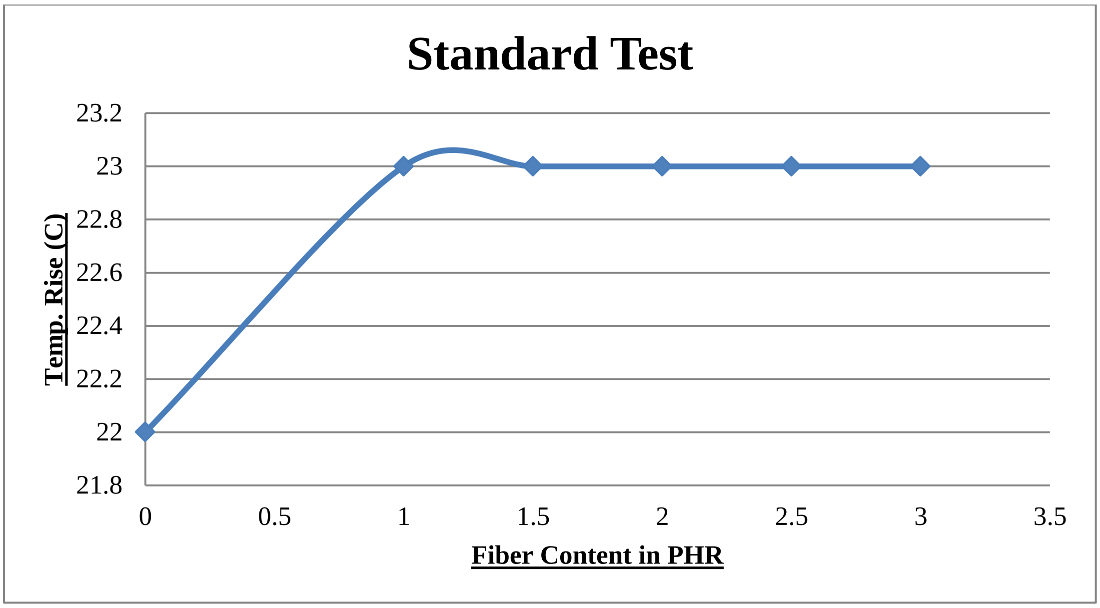

Figure 4.20 Goodrich Flexometer Heat Build-Up - Standard Test

Blowout Test

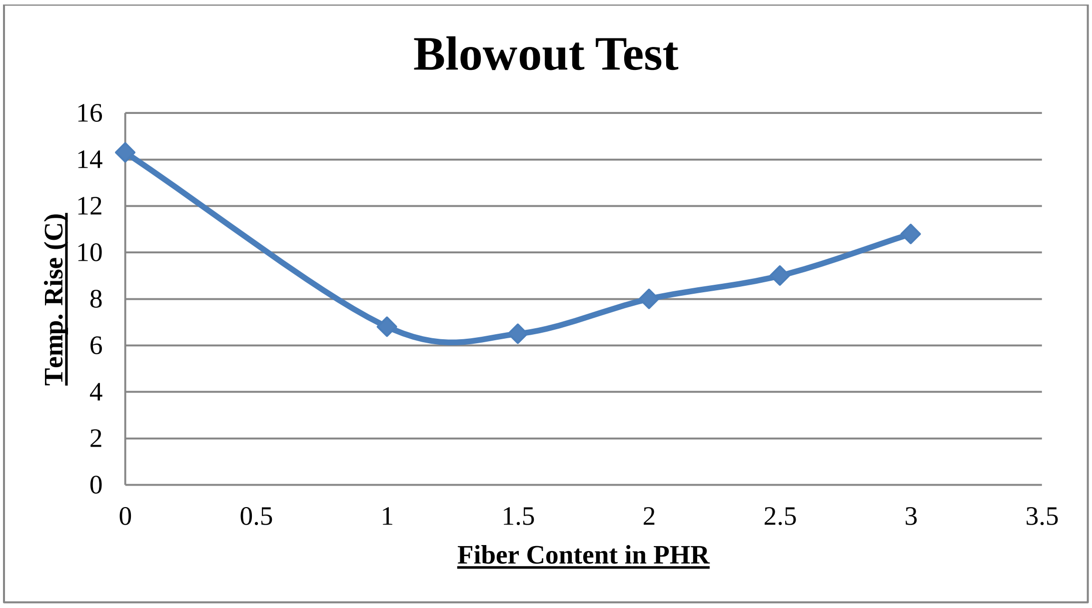

Figure 4.21 Goodrich Flexometer Heat Build-Up - Blowout Test

4.5.2 Dynamic Mechanical Analysis (DMA)

A Metravib DMA+150 Dynamic Mechanical Analyzer was used to complete temperature sweeps using the shear mode. The temperature sweep in shear was performed with 2^0 C/minute heating rate from 30^0 C to 100^0 C at 1 Hz with 0.0005 dynamic strains.

The main focus of this analysis was to closely observe the visco-elastic properties of the tyre tread compound. Tangent δ at 60^0C is used as a predictor of the tread compound's contribution to tyre rolling resistance, while Tangent δ at 0^0 C is used as a predictor of tread compound's wet traction. However, the test conditions for Tangent δ at 0^0 C are not available in-house presently, and therefore, the practical test for that was not performed. Also, within the scope of this research, wet traction analysis is of a lower precedence as it is not the main performance criterion for high load press-on tyres. Figure 4.22, shows the Tangent δ at 60^0C. As can be seen by the graph, the first six samples given are similar in value and from 5 phr onward, the value has drastically increased.

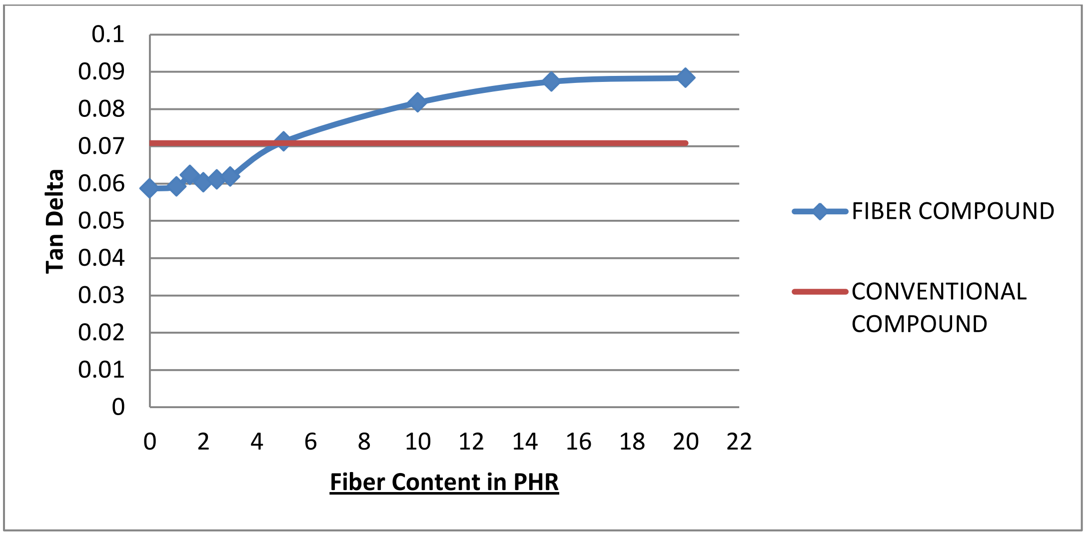

Figure 4.22 Tangent δ at 60 ^{0}C Temperature

4.6 Overall Performance in individual fiber loading

1 phr fiber loading vs control sample

As shown in the Figure 4.23 cut & chip and tear strength in transverse direction shown big improvement but there is a drastically drop blowout performance. Other results are more or less similar to control specification.

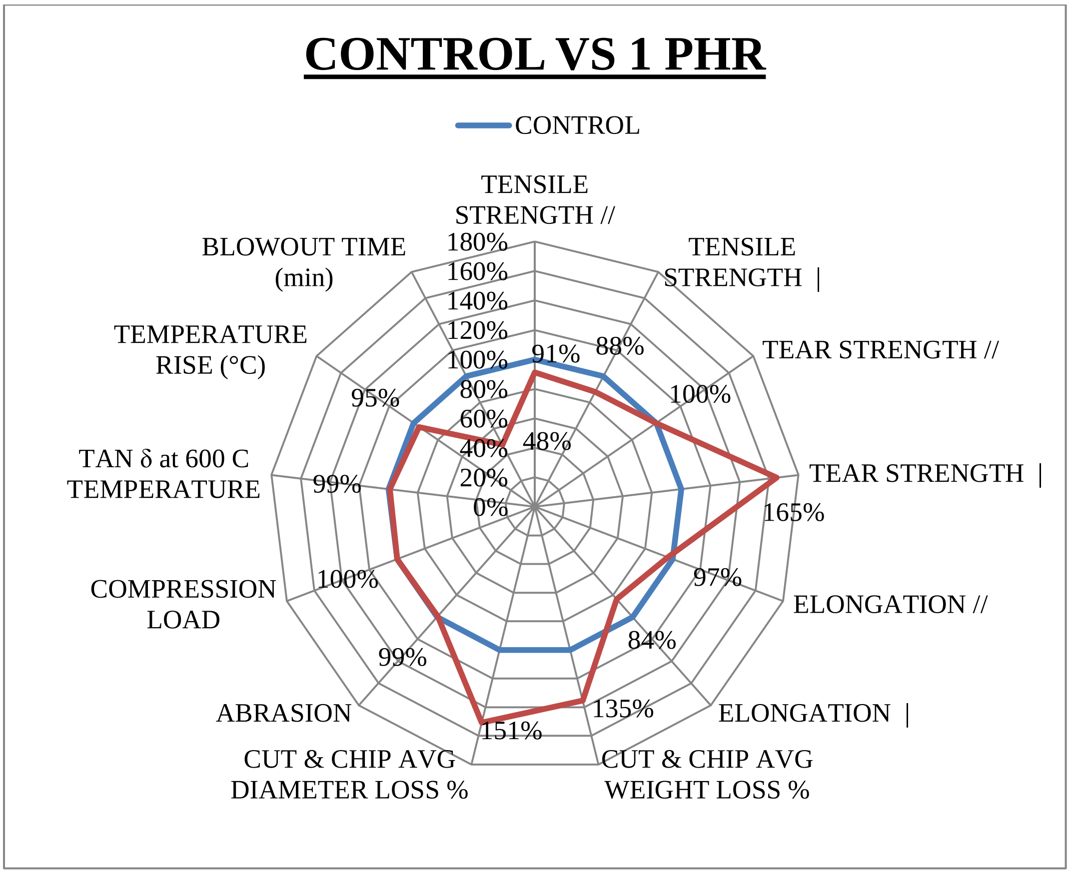

Figure 4.23 Control Vs 1 Phr

1.5 phr fiber loading vs control sample.

This is also similar behavior as 1 phr but there is a significant improvement in tearing strength in longitudinal direction.

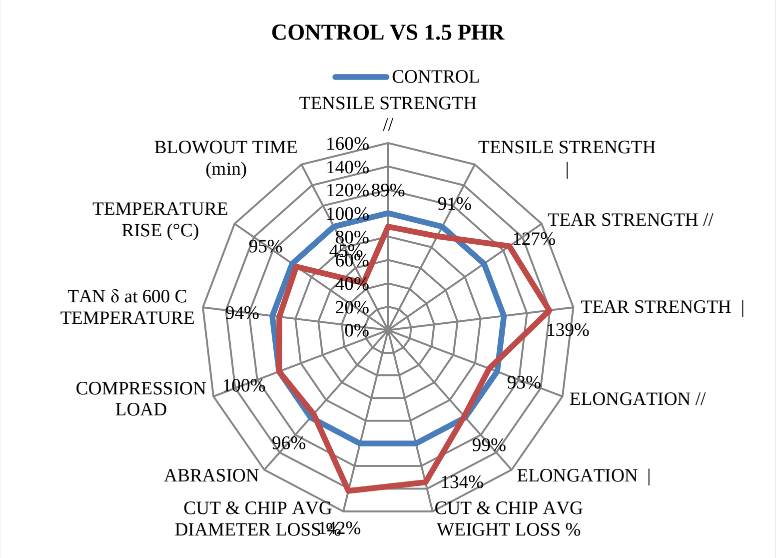

Figure 4.24 Control Vs 1.5 Phr

2 phr fiber loading vs control sample.

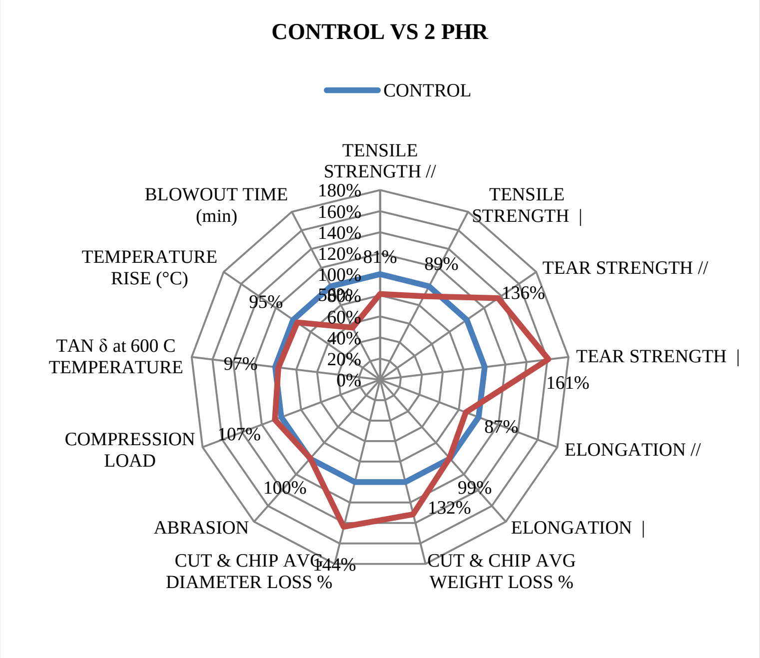

Figure 4.25 Control vs 2 phr

2.5 phr fiber loading vs control sample.

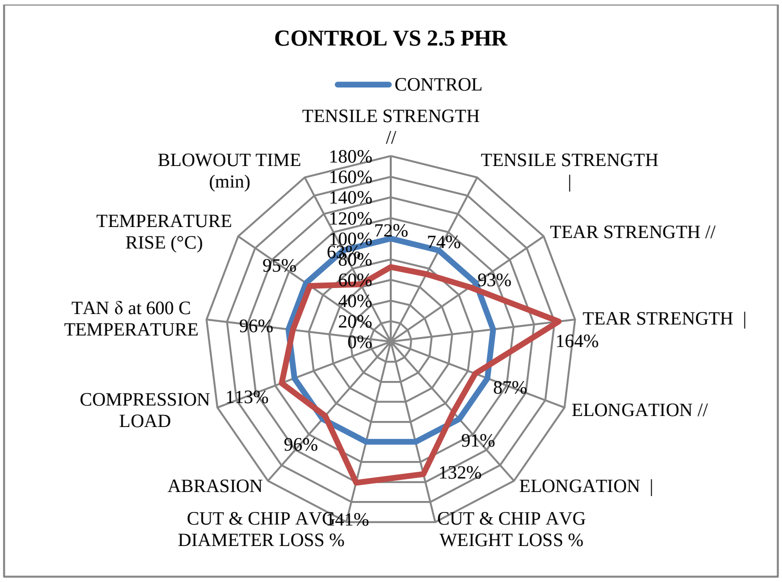

Figure 4.26 Control vs 2.5 phr

3 phr fiber loading vs control sample.

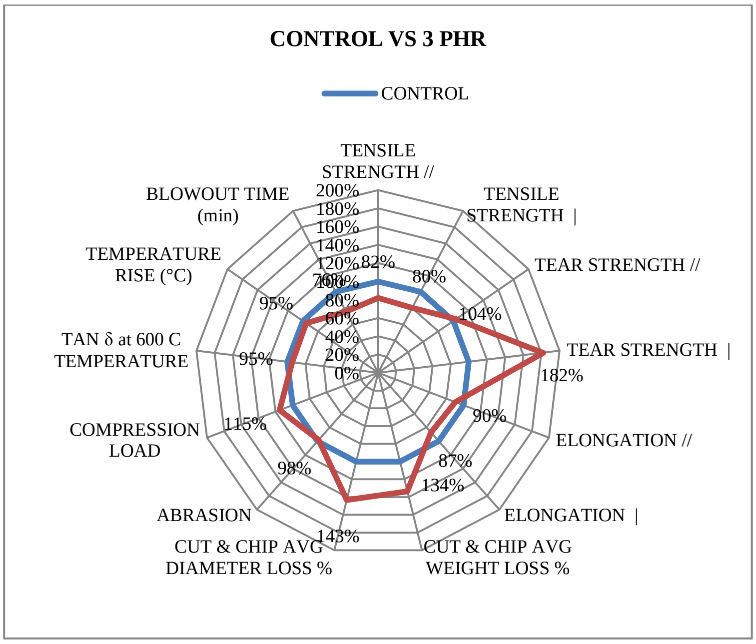

Figure 4.27 Control vs 3 phr

5 phr fiber loading vs control sample.

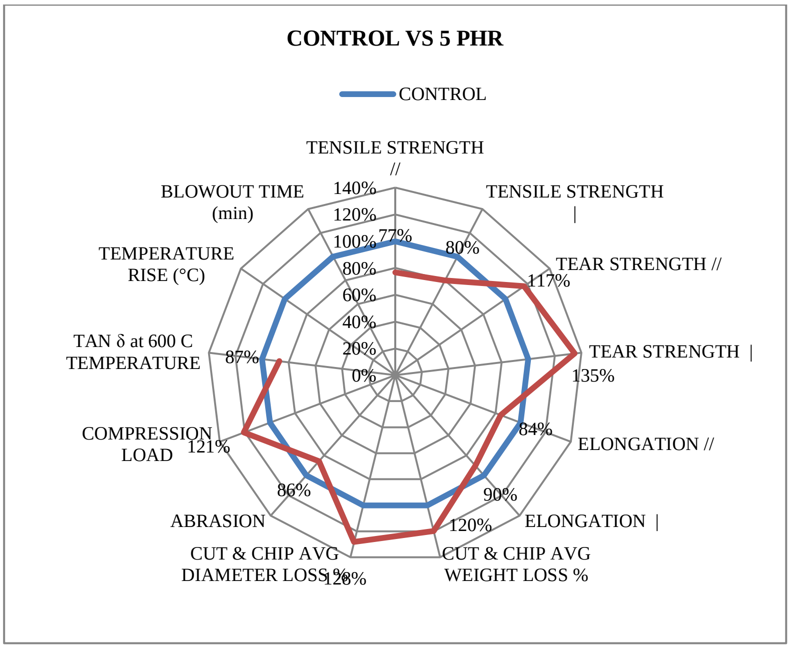

Figure 5.1 Control vs 5 phr

10 phr fiber loading vs control sample.

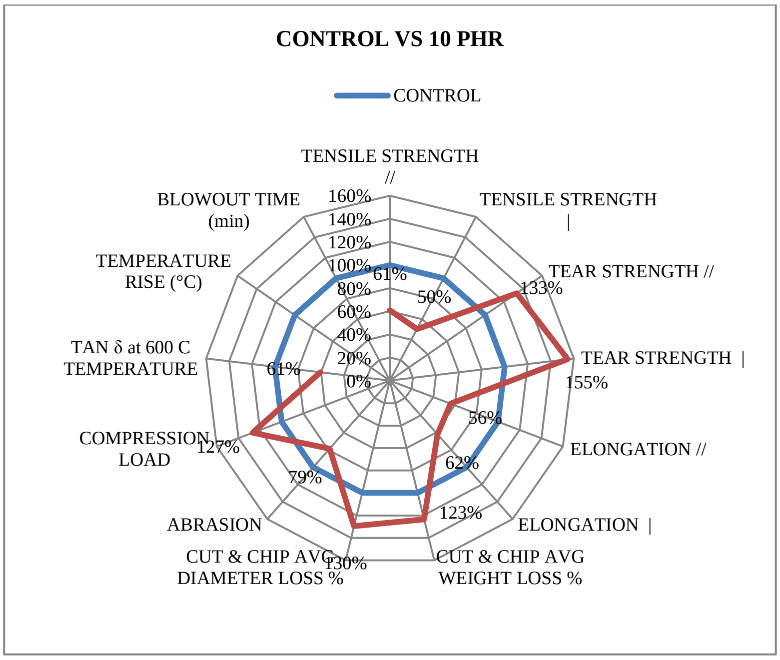

Figure 5.2 Control vs 10 phr

5 phr fiber loading vs control sample.

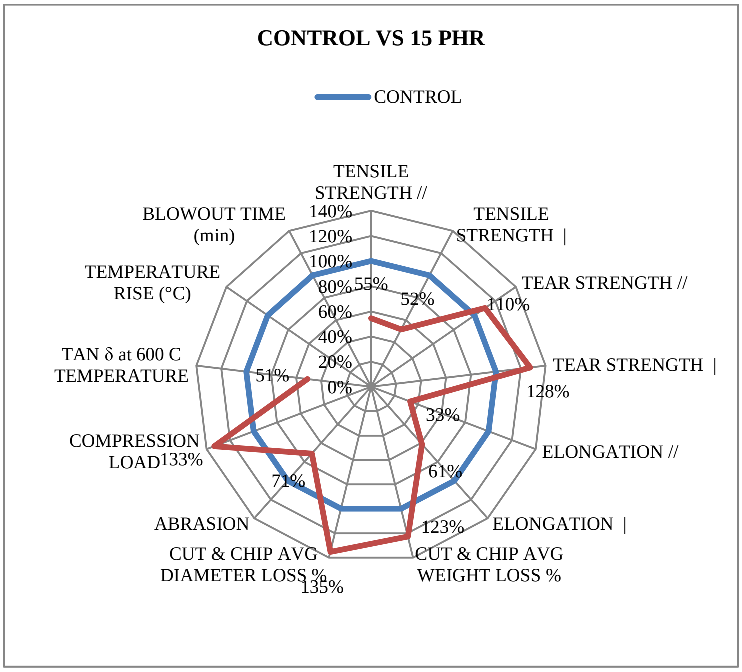

Figure 5.3 Control vs 15 phr

20 phr fiber loading vs control sample.

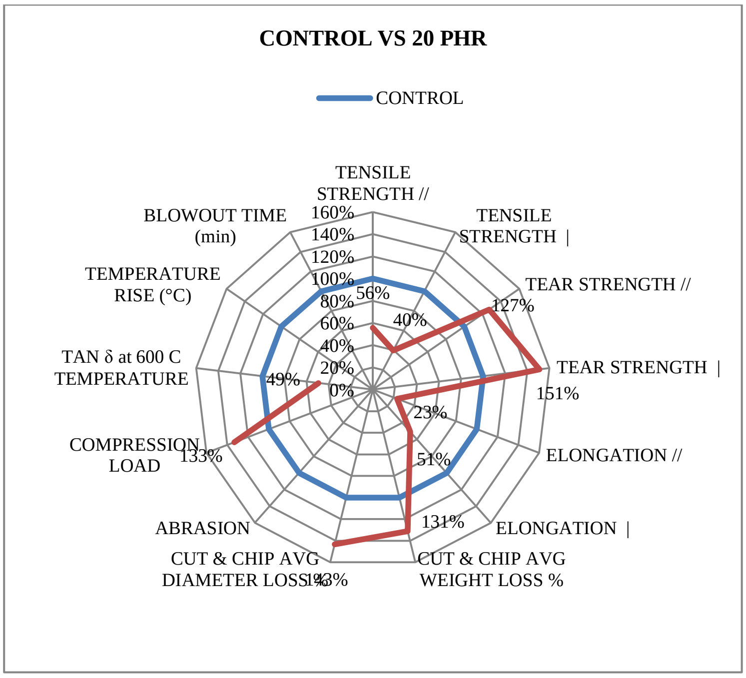

Figure 5.4 Control vs 20 phr

5.1 Hypothesis test for selecting best combination

As shown in overall rating, the highest values are represented in 2phr and 3 phr. Therefore, a hypothesis test was planned to select the best option out these two combinations. Minitab software was used to do the hypothesis test. The summary of hypothesis test is shown in Table 5. The details of Minitab results are in Annex 2.

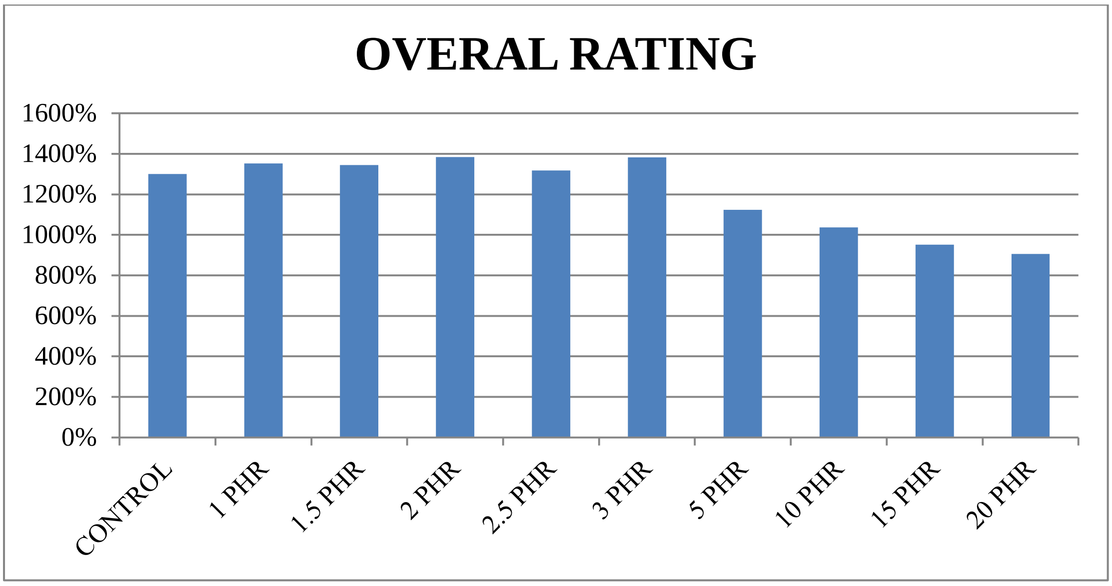

Figure 5.5 Overall Rating

Table 5 Summery of Hypothesis Test

Parameter	**Hypothetical Assumption**	**Conclusion**	**Selection**	
Tensile Strength//	Tensile St.of the 2 PHR nylon is difference from tensile st of the 3 PHR	There is not enough evidence to conclude that the means differ at the 0.05 level of significance.	Both same	Fig-16
Tensile Strength |	Tensile St.of the 2 PHR nylon is grather than tensile st of the3 PHR	conclude that the mean of 2 PHR NY-TEN is greater than 3PHR NY-T at the 0.05 level of significance.	2 PHR Nylon	Fig-17
Tear Strength //	Tear St.of the 2 PHR nylon is grather than tear st of the 3 PHR	Conclude that the mean of 2 PHR NY is greater than 3PHR NY at the 0.05 level of significance.	2 PHR Nylon	Fig-18
Tear Strength |	Tear St.of the 2 PHR nylon is difference from tear st of the 3 PHR	There is not enough evidence to conclude that the means differ at the 0.05 level of significance.	Both same	Fig-19
Cut & Chip (Avg weight loss)	Cut & Chip-Avr weight loss of the 2 PHR nylon is difference from Cut & Chip-Avr weight loss st of the 3PHR	There is not enough evidence to conclude that the means differ at the 0.05 level of significance.	Both same	Fig-20
Cut & Chip (Avg diam.	Cut & Chip-Avr diameter loss of the	There is not enough evidence to conclude	Both same	Fig-21

loss)	2 PHR nylon is difference from Cut & Chip avg diameter loss of the 3 PHR	that the means differ at the 0.05 level of significance.		
Abrasion	Abrasionof the 2 PHR nylon is difference from Abrasion of the 3 PHR	There is not enough evidence to conclude that the means differ at the 0.05 level of significance.	Both same	Fig-22
Compression Test	Compression load.of the 2 PHR nylon is difference from Compression load of the 3 PHR	There is not enough evidence to conclude that the means differ at the 0.05 level of significance.	Both same	Fig-23

6 CHAPTER 05 - CONCLUSION AND RECOMMENDATIONS FOR FUTURE STUDIES

In recent years, rubber composites are becoming increasingly important. Short fibers are added as fillers to improve the strength and stiffness of the composites. This study was carried out with the objective of systematically analyzing the effect of short nylon fibre on the properties of natural rubber vulcanizate, so as to assess the feasibility of using it as filler for rubber resulting in rubber composites with good physical, mechanical and dynamic properties.

6.1 Conclusion

6.1.1 Physical Properties

a. The presence of short nylon fiber in Natural rubber base compound makes it superior to the conventional compound (non-fiber reinforced) in terms of Tear Strength. Further, Tear Strength is superior in transverse direction (direction perpendicular to fiber orientation) than in the longitudinal direction (direction of fiber orientation).

b. Hardness and Specific Gravity remains more or less the same as the conventional compound while increasing the short nylon fiber content. Similar specific gravity means a similar volume at a given weight and is an indication of a more or less similar cost of the final product.

c. The tensile strength and elongation at break properties are negatively influenced by the presence of short nylon fiber in the natural rubber base compound.

6.1.2 Mechanical Properties

- The presence of short nylon fiber in natural rubber base compound is superior to conventional compound in terms of cut & chip resistance and compression test.
- Up to 3 phr loading of short nylon fiber in the natural rubber based compound, abrasion resistance is much better or equal. But, the presence of 5 phr or more of short nylon fiber, the abrasion resistance is negatively influenced.
- The Rebound resilience, an indication of the material elasticity, slightly increases with fiber loading of the composite. The energy absorption of the composite is relatively low in the presence short nylon fibers and it is an indication of low rolling resistance of the compound.

6.1.3 Dynamic Properties

- The presence of short nylon fiber in natural rubber base compound is superior to the conventional compound in terms of rolling resistance (Tangent δ at 600C) up to 5 phr short nylon fiber loading but beyond 5 phr fibers content, the rolling resistance is negatively influenced.

6.1.4 Cure Characteristics

- The values of Mooney viscosity and minimum torque (ML) represent the viscosity values of the compound and the presence of short nylon fiber up to 5 phr in rubber base compound is positively influenced on flow properties of the compound.
- The scorch safety (ts2) has a direct relationship with the fiber contentment and this indicates that nylon fiber has a delaying effect on the cure reaction.

In an overall context, there are many positive advantages in the short nylon fiber reinforced compound but we have to select the most appropriate fiber content for the application. I have used the Hypothesis test for selecting the best combination. According to the hypothesis test, the most suitable short nylon fiber content is 2 phr..

6.2 Recommendation for Future Studies

- The present study was done on a single type of fiber, namely nylon. Many other synthetic and natural fibers are available and the evaluation of other fibers such as Kevlar, polyester, rayon, cotton, etc can be recommended.
- Evaluation of many other rubbers such as SBR, BR or blend would be beneficial for further improvement of composite performance.
- Evaluation of ageing properties of products similar to the tyres studied here, and is exposed to different weather conditions would be an additional advantage.
- Evaluation of different bonding systems on improving the bonding strength of fiber-matrix interface would be beneficial.
- The processing of the fiber added compound is rather challengeable and further investigations on the processability of the composite would be beneficial.

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APPENDIX 1

MATERIAL SPECIFICATION

Natural Rubber

Natural rubber was taken under the following specification

Mooney viscosity [ML(1+4) at 100 C]	80 +/-15
% Dirt content by mass (Max.)	0.05
% of Volatile material by mass (Max.)	0.8
% of Nitrogen by mass (Max.)	0.6
% of Ash by mass (Max.)	0.5
Initial Wallace Plasticity (Po) * Range (Min.)	30
Plastic Retention Index (PRL) (Min.)	70

Short Nylon fiber

Materiel is nylon 6. It was obtained from the approved supplier. The material was cut to approximately 6 mm. Specification of nylon material is as follows.

Breaking Strength – 1260D2 (Min.)	20.5 kg
Elongation at Break	+/-25
Twist Cable S	370 +/- 25 T/m
Twist Ply Z	375 +/- 25 T/m
Breaking Strength – 1890D2 (Min.)	30.5 kkg
Elongation at Break	+/-25
Twist Cable S	300 +/- 25 T/m
Twist Ply Z	305 +/- 25 T/m

1st Stage Chemicals

i. Carbon Black

Carbon black was taken from our approved supplier and it has the following specification:

DPB absorption (cm^3/100g)	115-125
Loading adsorption number (mg/g)	85-89
Pour Density (Kg/m^3)	315-370
Fines Content (%)	10 max.
Ash (Max.) (%)	0.5

ii. Zinc Oxide

Zinc oxide used in the study was procured from approved supplier with the ~~has~~ following specification

% of Moisture (Max)	0.5
% of Pbo (Max)	0.15
% of Purity (Min)	99.5
% of Sieve Residue (max) 45 Micron (325 mesh)	0.25
% of Loss on Ignition (Max)	0.6

iii. Stearic Acid

Steric Acid was obtained from approved supplier with the following specification:

Acid Value (mg,KOH/g)	190-215
Iodine Value (Max) (g,100g)	5
% of Fatty acid C18 (Min)	32

iv. 6PPD

6PPD was taken from approved supplier with the following specification:

% of Ash Content (max)	0.2
Melting point (min) °C	44
% of Active Ingredient (min)	97
% of Heat loss (max)	0.5

v. HS

% of Ash Content (Max.)	0.3
Softening point °C	83-93
% of Active ingredient (Min)	88

vi. OIL (Low PAHs Rubber Processing oil)

Kinematic viscosity @ 100° C cSt	20-32
Aniline point °C	86-94
Density @ 15.0c g/ml	0.930-0.950
Benzo (a) pyrene (BaP) (Max. ppm)	1
Sum 8 PAH's (Max. ppm)	10
Flash Point (Min.) °C	210

vii. RESORCINE 80 SBR

% of Resorcinol Content	77-81
Density (g/cm3)	1.17-1.25

viii. SILICA

BET Surface Area (m^2 / g)	160-195
% of Moisture	4 - 7
% of SiO2, on anhydrous basis (Min.)	98
% of SiO2 (bsed on dry basis, 105 C for 1.5 hrs) (Min.)	93
% of Salt as Na2SO4	< 1.5
% of 20 mesh sieve residue (Min.)	65
% of 80 mesh sieve residue (Min.)	75

2^{nd} Stage Chemicals

i. SULPHUR

% of Sieve Residue (325mesh), (Max.)	10
% of Moisture (Max.)	0.5
% of Purity (Min.)	99.5

ii. TBBS

% of Purity (Min.)	95
% of Ash Content (Max.)	0.5
Melting point (Min.) °C	103
% of Loss of heating (Max.)	1
% of Free amine (Max.)	0.5

iii. PVI

% of Active ingredient content (Min)	95
% of Ash Content (Max.)	0.2
% Drying loss (Heat loss)	0.4
Melting point °C	88
% of Volatile matter (Max.)	0.5

iv. HMT

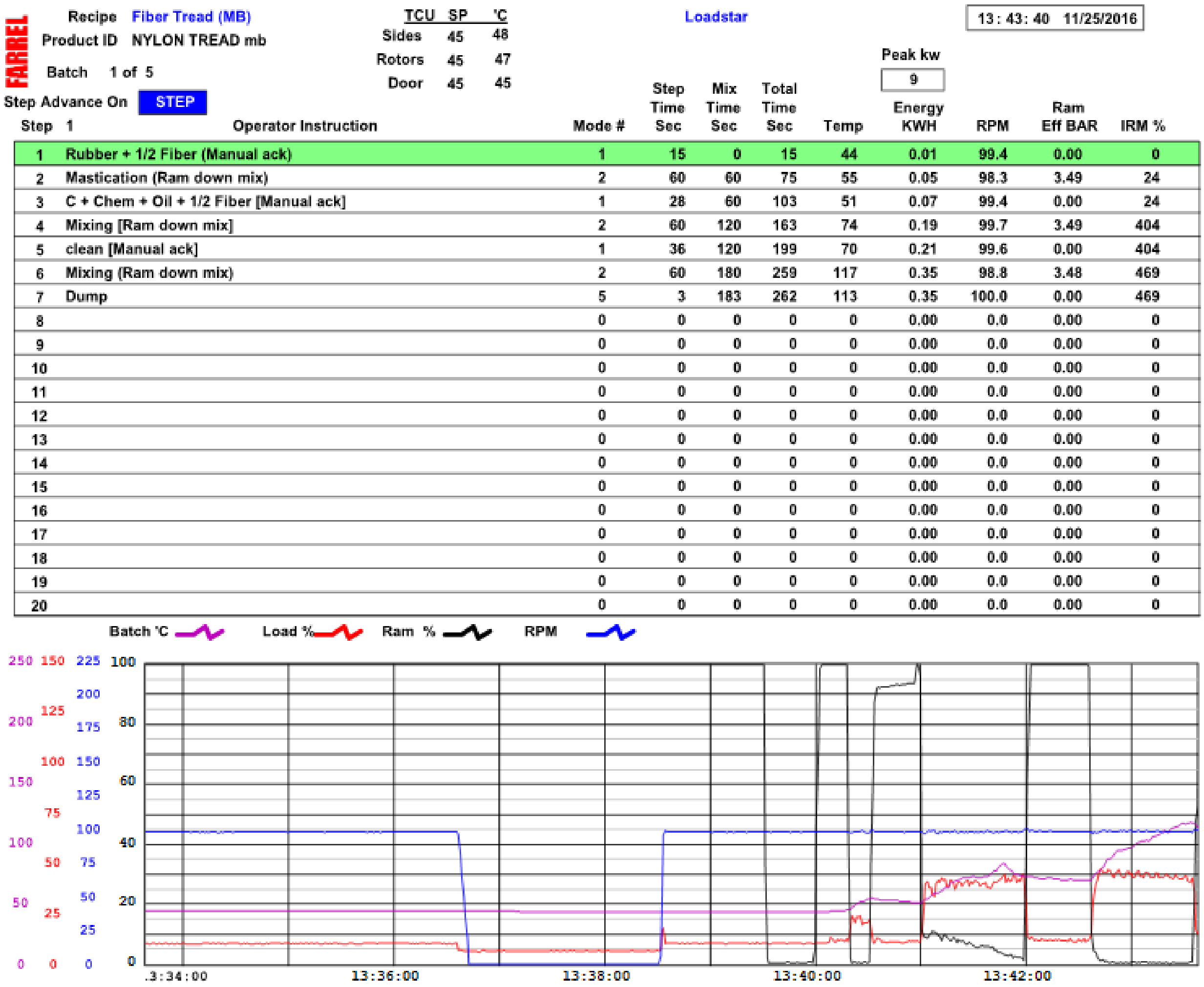
% of Hexamethylene tetramine	Min. 96
% Water content	Max. 0.5
% of Ash content	2.0 - 3.0
% of particles > 63 micro meter	Max. 1.0

APPENDIX 2

Mixing cycle of Compounding trials

First Stage Mixing

Figure A 2.1: 0phr Fiber content



Recipe Fiber Tread (MB)
Product ID NYLON TREAD mb
Batch 1 of 5
Step Advance On STEP

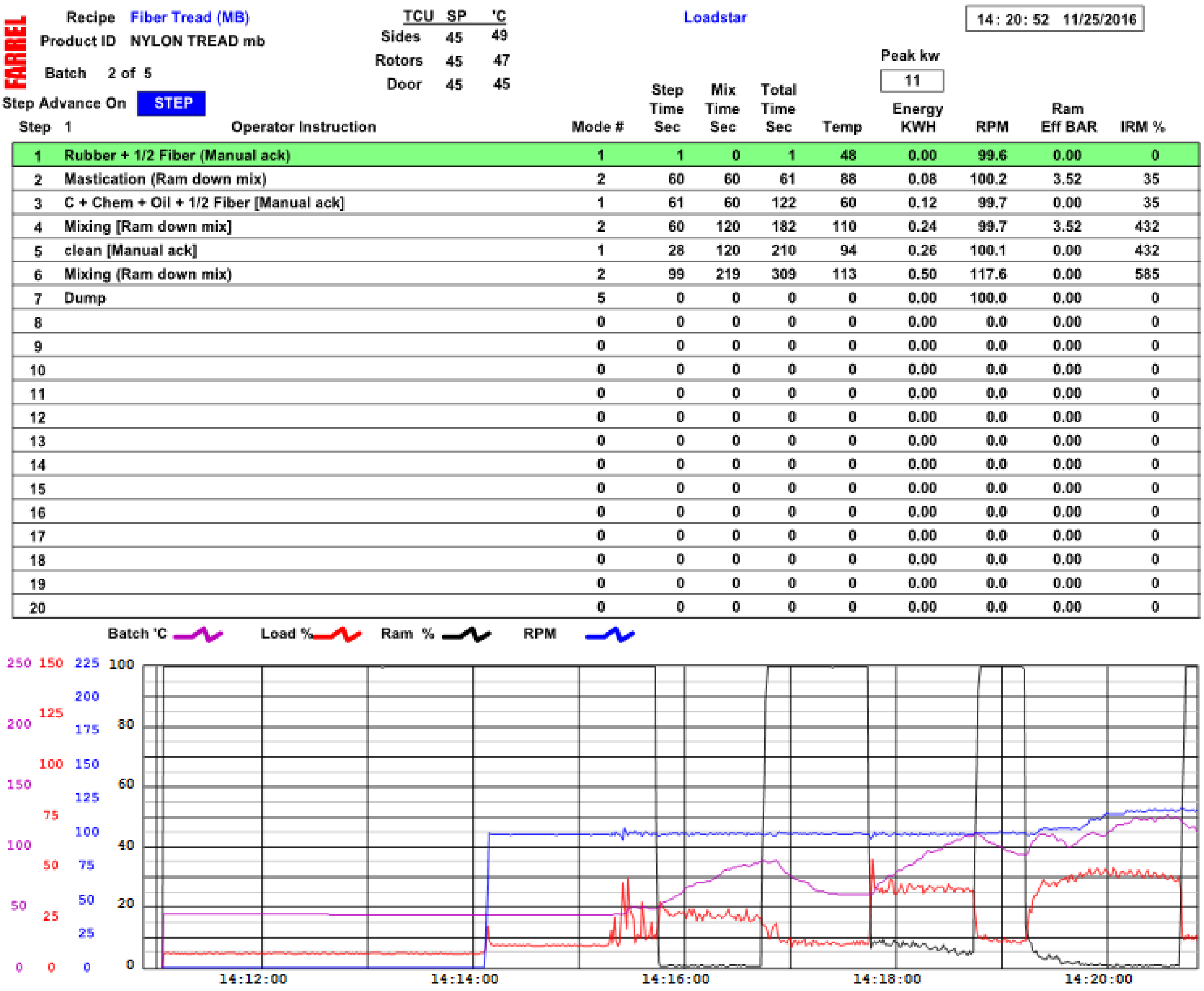
TCU	SP	'C
Sides	45	48
Rotors	45	47
Door	45	45

13 : 43 : 40 11/25/2016

Peak kw 9

Step 1	Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	Rubber + 1/2 Fiber (Manual ack)	1	15	0	15	44	0.01	99.4	0.00	0
2	Mastication (Ram down mix)	2	60	60	75	55	0.05	98.3	3.49	24
3	C + Chem + Oil + 1/2 Fiber [Manual ack]	1	28	60	103	51	0.07	99.4	0.00	24
4	Mixing [Ram down mix]	2	60	120	163	74	0.19	99.7	3.49	404
5	clean [Manual ack]	1	36	120	199	70	0.21	99.6	0.00	404
6	Mixing (Ram down mix)	2	60	180	259	117	0.35	98.8	3.48	469
7	Dump	5	3	183	262	113	0.35	100.0	0.00	469
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
15		0	0	0	0	0	0.00	0.0	0.00	0
16		0	0	0	0	0	0.00	0.0	0.00	0
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

Figure A 2.2: 1 phr Fiber content



FARREL

Recipe Fiber Tread (MB)
Product ID NYLON TREAD mb
Batch 2 of 5
Step Advance On STEP

TCU	SP	'C
Sides	45	49
Rotors	45	47
Door	45	45

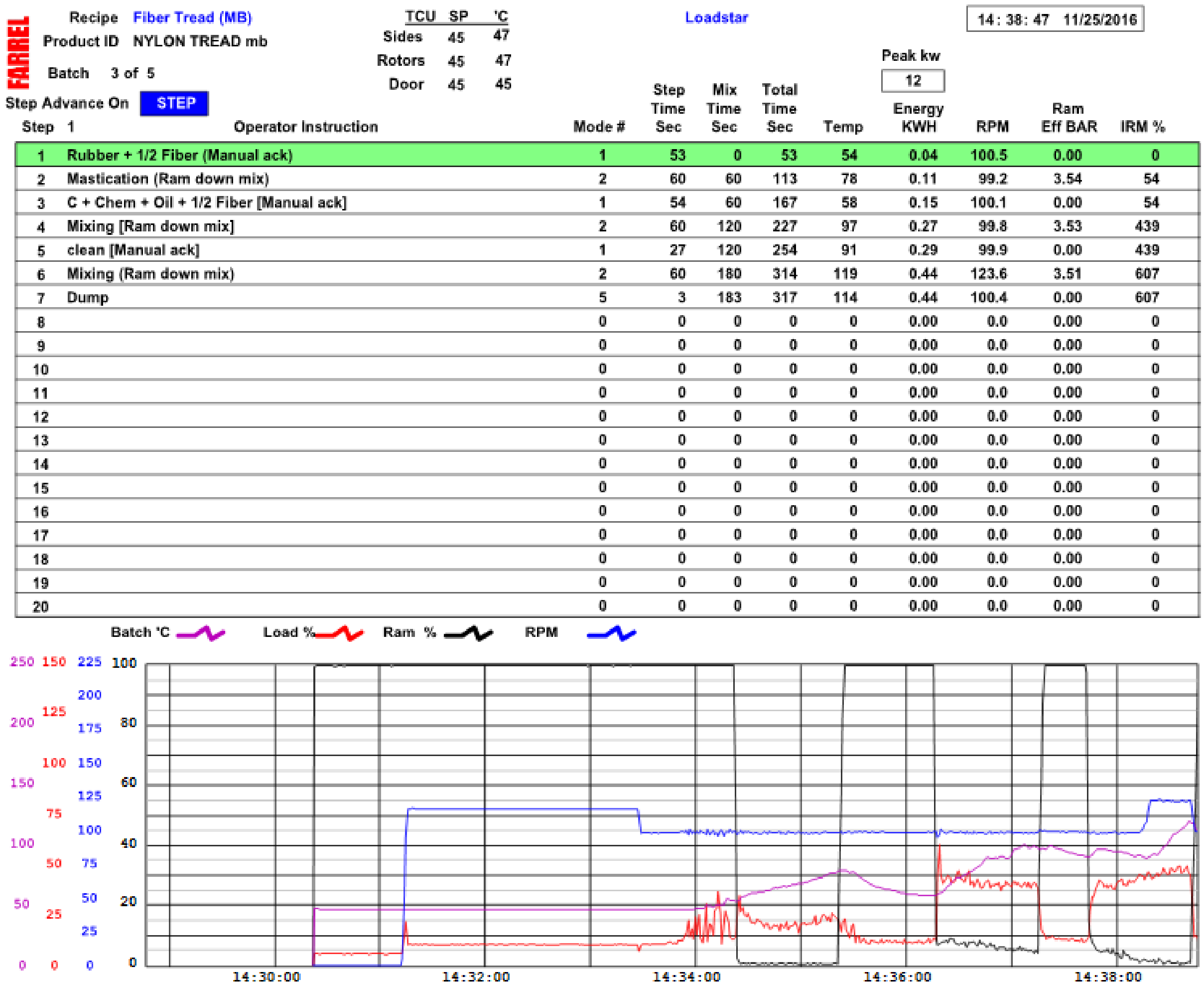
Loadstar

14 : 20: 52 11/25/2016

Peak kw
11

Step 1	Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	Rubber + 1/2 Fiber (Manual ack)	1	1	0	1	48	0.00	99.6	0.00	0
2	Mastication (Ram down mix)	2	60	60	61	88	0.08	100.2	3.52	35
3	C + Chem + Oil + 1/2 Fiber [Manual ack]	1	61	60	122	60	0.12	99.7	0.00	35
4	Mixing [Ram down mix]	2	60	120	182	110	0.24	99.7	3.52	432
5	clean [Manual ack]	1	28	120	210	94	0.26	100.1	0.00	432
6	Mixing (Ram down mix)	2	99	219	309	113	0.50	117.6	0.00	585
7	Dump	5	0	0	0	0	0.00	100.0	0.00	0
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
15		0	0	0	0	0	0.00	0.0	0.00	0
16		0	0	0	0	0	0.00	0.0	0.00	0
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

Figure A 2.3: 1.5 phr Fiber content



FARREL

Recipe Fiber Tread (MB)
Product ID NYLON TREAD mb
Batch 3 of 5
Step Advance On STEP
Step 1

TCU	SP	'C
Sides	45	47
Rotors	45	47
Door	45	45

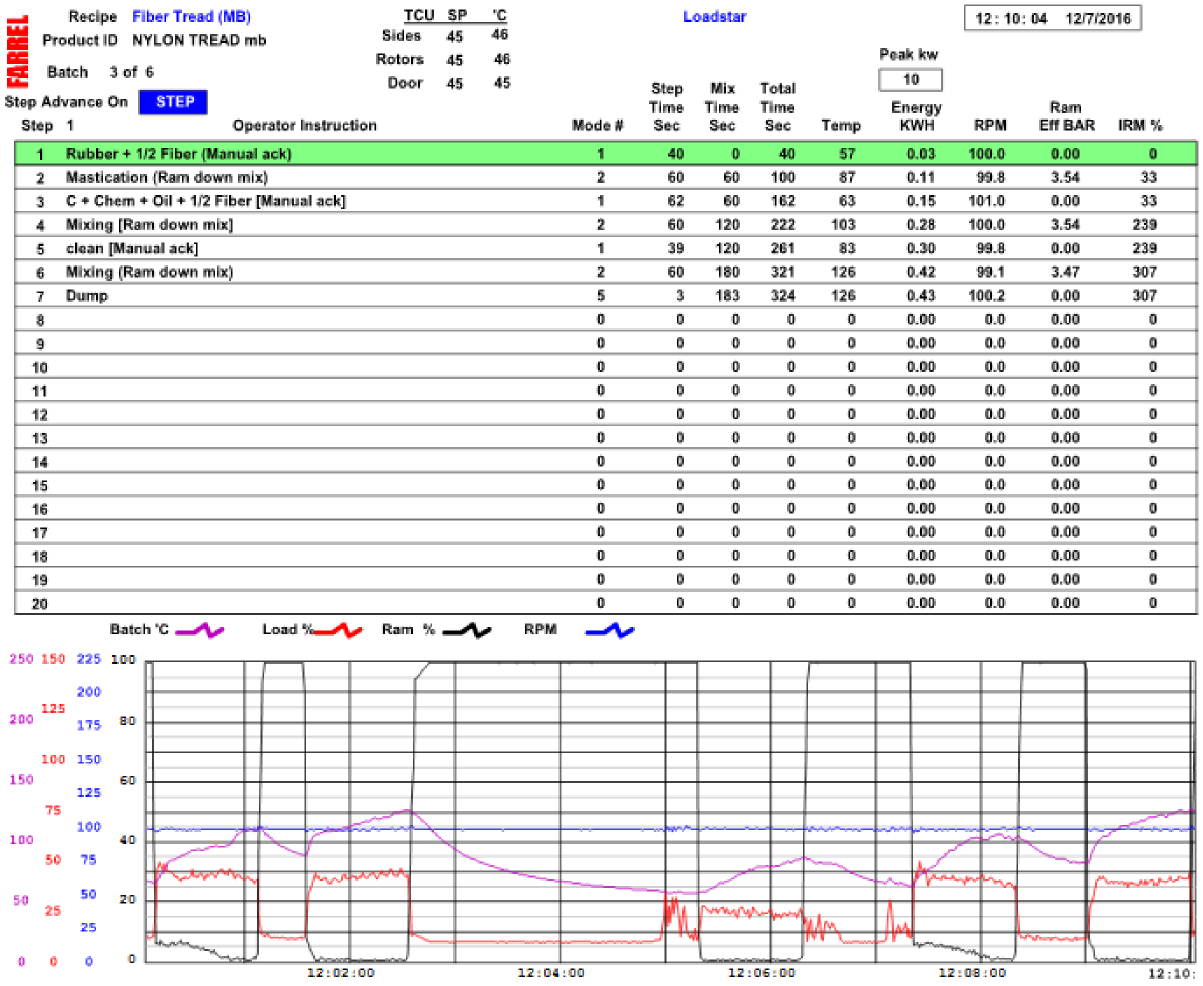
Loadstar

14: 38: 47 11/25/2016

Peak kw 12

Step	Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	Rubber + 1/2 Fiber (Manual ack)	1	53	0	53	54	0.04	100.5	0.00	0
2	Mastication (Ram down mix)	2	60	60	113	78	0.11	99.2	3.54	54
3	C + Chem + Oil + 1/2 Fiber [Manual ack]	1	54	60	167	58	0.15	100.1	0.00	54
4	Mixing [Ram down mix]	2	60	120	227	97	0.27	99.8	3.53	439
5	clean [Manual ack]	1	27	120	254	91	0.29	99.9	0.00	439
6	Mixing (Ram down mix)	2	60	180	314	119	0.44	123.6	3.51	607
7	Dump	5	3	183	317	114	0.44	100.4	0.00	607
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
15		0	0	0	0	0	0.00	0.0	0.00	0
16		0	0	0	0	0	0.00	0.0	0.00	0
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

Figure A 2.4 : 2 phr Fiber content



FARREL

Recipe **Fiber Tread (MB)**
Product ID NYLON TREAD mb
Batch 3 of 6
Step Advance On **STEP**

Loadstar

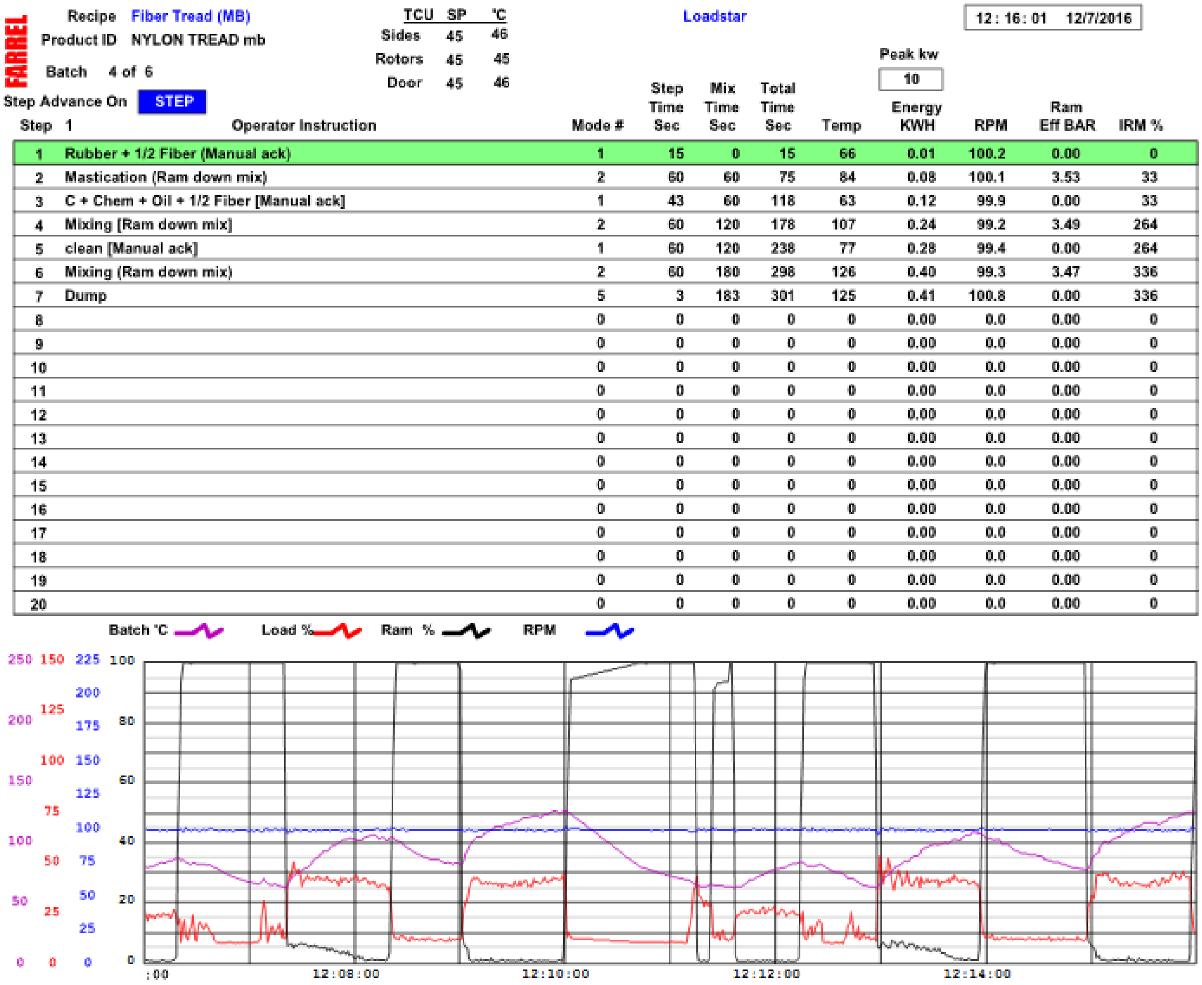
12 : 10: 04 12/7/2016

TCU	SP	'C
Sides	45	46
Rotors	45	46
Door	45	45

Peak kw: 10

Step 1	Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	Rubber + 1/2 Fiber (Manual ack)	1	40	0	40	57	0.03	100.0	0.00	0
2	Mastication (Ram down mix)	2	60	60	100	87	0.11	99.8	3.54	33
3	C + Chem + Oil + 1/2 Fiber [Manual ack]	1	62	60	162	63	0.15	101.0	0.00	33
4	Mixing [Ram down mix]	2	60	120	222	103	0.28	100.0	3.54	239
5	clean [Manual ack]	1	39	120	261	83	0.30	99.8	0.00	239
6	Mixing (Ram down mix)	2	60	180	321	126	0.42	99.1	3.47	307
7	Dump	5	3	183	324	126	0.43	100.2	0.00	307
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
15		0	0	0	0	0	0.00	0.0	0.00	0
16		0	0	0	0	0	0.00	0.0	0.00	0
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

Figure A 2.5: 2.5 phr Fiber content



FARREL

Recipe **Fiber Tread (MB)**
Product ID NYLON TREAD mb
Batch 4 of 6
Step Advance On STEP

TCU	SP	'C
Sides	45	46
Rotors	45	45
Door	45	46

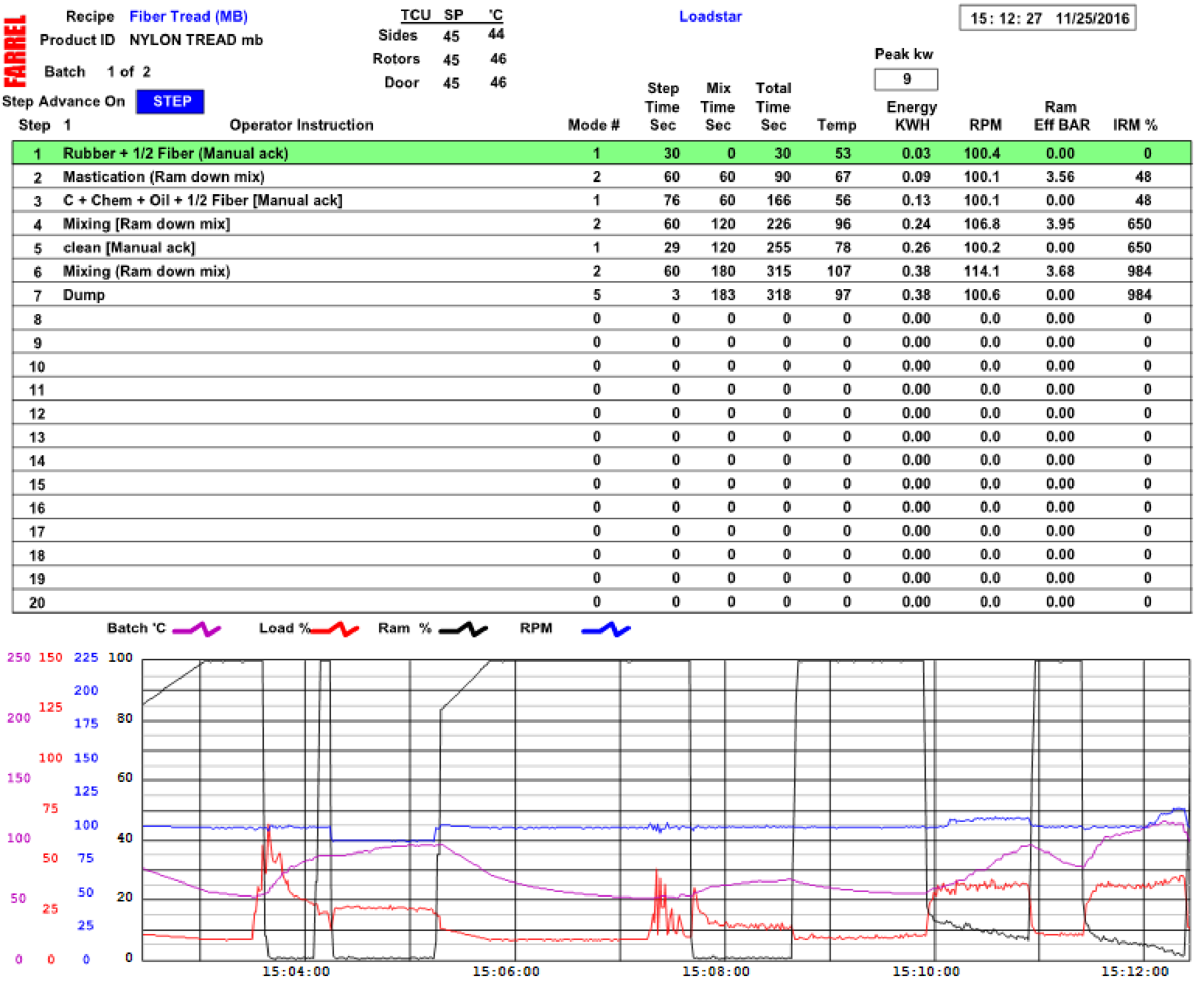
Loadstar

12 : 16 : 01 12/7/2016

Peak kw 10

Step 1	Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	Rubber + 1/2 Fiber (Manual ack)	1	15	0	15	66	0.01	100.2	0.00	0
2	Mastication (Ram down mix)	2	60	60	75	84	0.08	100.1	3.53	33
3	C + Chem + Oil + 1/2 Fiber [Manual ack]	1	43	60	118	63	0.12	99.9	0.00	33
4	Mixing [Ram down mix]	2	60	120	178	107	0.24	99.2	3.49	264
5	clean [Manual ack]	1	60	120	238	77	0.28	99.4	0.00	264
6	Mixing (Ram down mix)	2	60	180	298	126	0.40	99.3	3.47	336
7	Dump	5	3	183	301	125	0.41	100.8	0.00	336
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
15		0	0	0	0	0	0.00	0.0	0.00	0
16		0	0	0	0	0	0.00	0.0	0.00	0
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

Figure A 2.6: 3 phr Fiber content



FARREL

Recipe Fiber Tread (MB)

Product ID NYLON TREAD mb

Batch 1 of 2

Step Advance On STEP

Step 1

TCU	SP	'C
Sides	45	44
Rotors	45	46
Door	45	46

Loadstar

15: 12: 27 11/25/2016

Peak kw: 9

Step	Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	Rubber + 1/2 Fiber (Manual ack)	1	30	0	30	53	0.03	100.4	0.00	0
2	Mastication (Ram down mix)	2	60	60	90	67	0.09	100.1	3.56	48
3	C + Chem + Oil + 1/2 Fiber [Manual ack]	1	76	60	166	56	0.13	100.1	0.00	48
4	Mixing [Ram down mix]	2	60	120	226	96	0.24	106.8	3.95	650
5	clean [Manual ack]	1	29	120	255	78	0.26	100.2	0.00	650
6	Mixing (Ram down mix)	2	60	180	315	107	0.38	114.1	3.68	984
7	Dump	5	3	183	318	97	0.38	100.6	0.00	984
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
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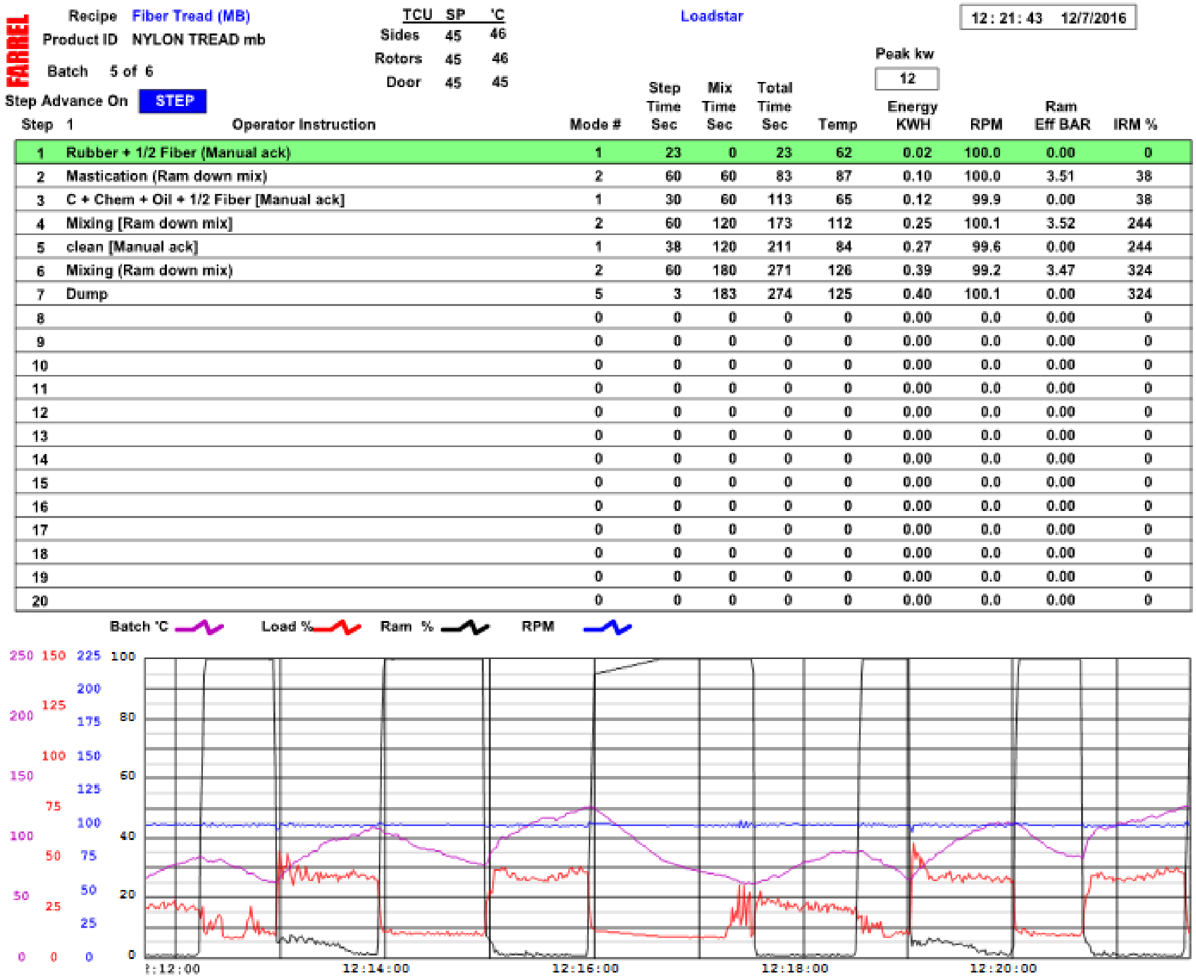
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

Figure A 2.7: 5 phr Fiber content



FARREL

Recipe **Fiber Tread (MB)**
Product ID **NYLON TREAD mb**
Batch 5 of 6
Step Advance On **STEP**

TCU	SP	'C
Sides	45	46
Rotors	45	46
Door	45	45

Loadstar

12 : 21 : 43 12/7/2016

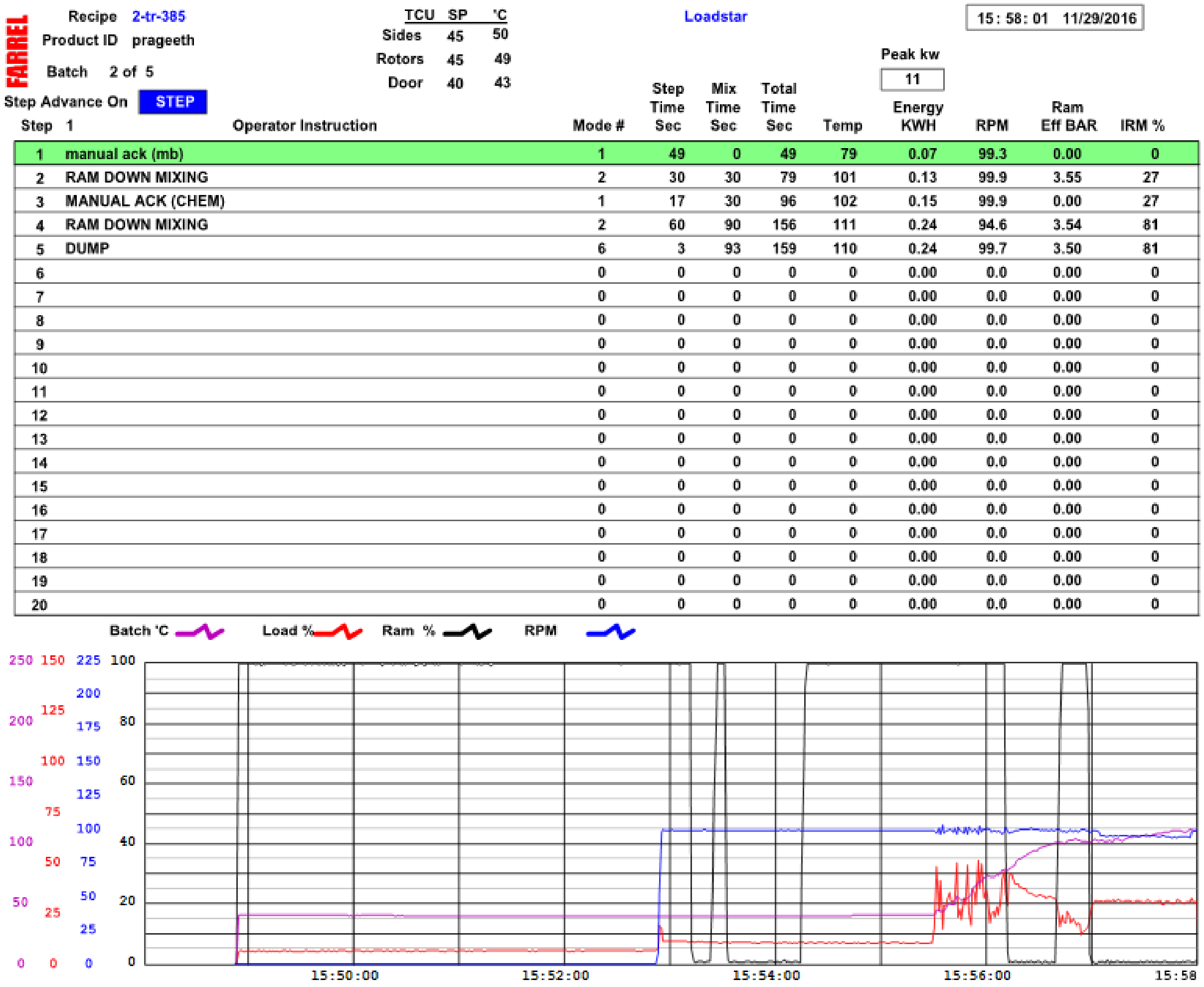
Peak kw 12

Step 1

Step	Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	Rubber + 1/2 Fiber (Manual ack)	1	23	0	23	62	0.02	100.0	0.00	0
2	Mastication (Ram down mix)	2	60	60	83	87	0.10	100.0	3.51	38
3	C + Chem + Oil + 1/2 Fiber [Manual ack]	1	30	60	113	65	0.12	99.9	0.00	38
4	Mixing [Ram down mix]	2	60	120	173	112	0.25	100.1	3.52	244
5	clean [Manual ack]	1	38	120	211	84	0.27	99.6	0.00	244
6	Mixing (Ram down mix)	2	60	180	271	126	0.39	99.2	3.47	324
7	Dump	5	3	183	274	125	0.40	100.1	0.00	324
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
15		0	0	0	0	0	0.00	0.0	0.00	0
16		0	0	0	0	0	0.00	0.0	0.00	0
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

Second Stage Mixing

Figure A 2.8: 0 phr Fiber content



Recipe 2-tr-385
Product ID prageeth
Batch 2 of 5
Step Advance On STEP

TCU	SP	'C
Sides	45	50
Rotors	45	49
Door	40	43

Loadstar

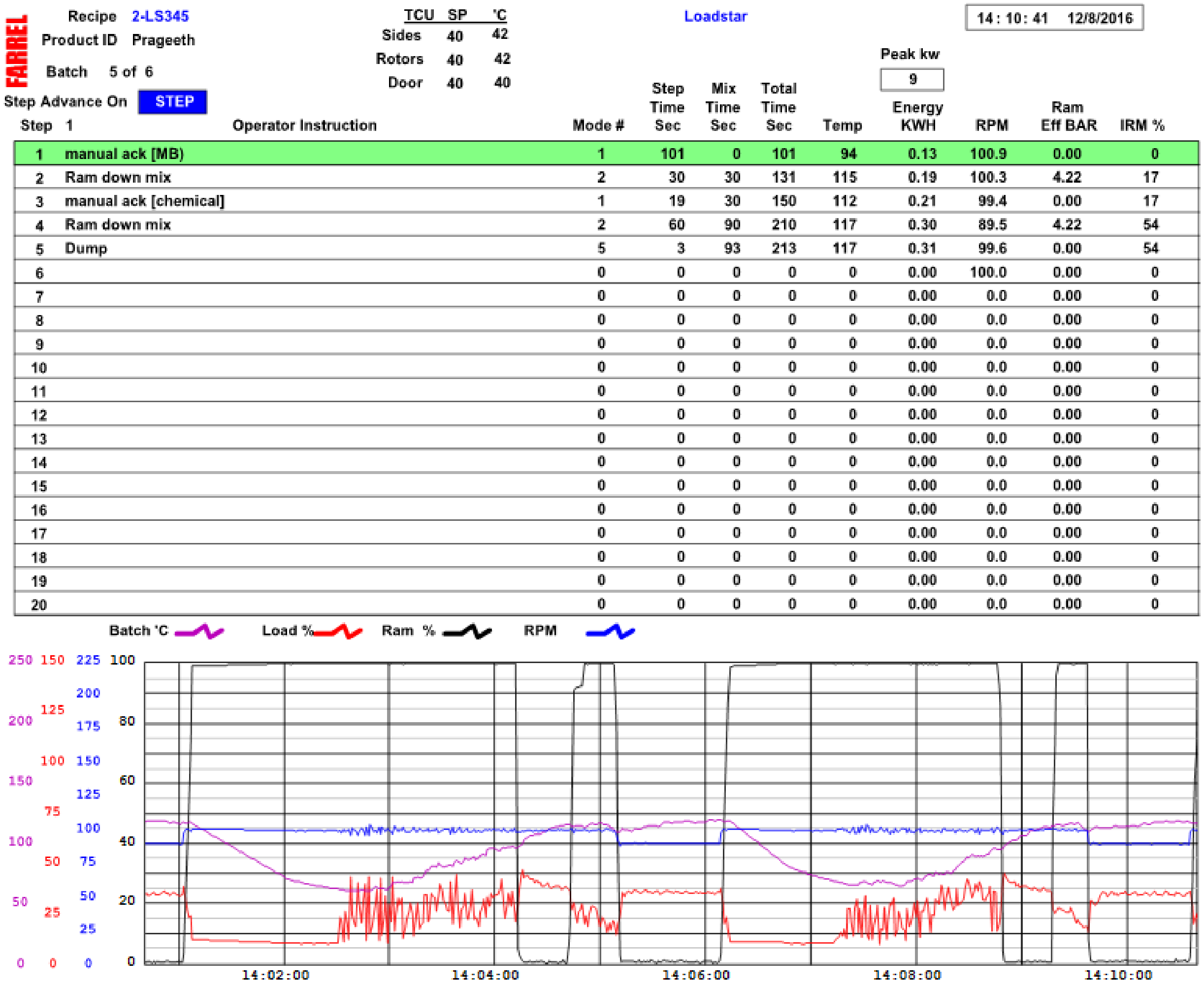
15: 58: 01 11/29/2016

Peak kw 11

Step 1

Step	Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	manual ack (mb)	1	49	0	49	79	0.07	99.3	0.00	0
2	RAM DOWN MIXING	2	30	30	79	101	0.13	99.9	3.55	27
3	MANUAL ACK (CHEM)	1	17	30	96	102	0.15	99.9	0.00	27
4	RAM DOWN MIXING	2	60	90	156	111	0.24	94.6	3.54	81
5	DUMP	6	3	93	159	110	0.24	99.7	3.50	81
6		0	0	0	0	0	0.00	0.0	0.00	0
7		0	0	0	0	0	0.00	0.0	0.00	0
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
15		0	0	0	0	0	0.00	0.0	0.00	0
16		0	0	0	0	0	0.00	0.0	0.00	0
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

Figure A 2.9: 1 phr Fiber content



Recipe 2-LS345
Product ID Prageeth
Batch 5 of 6
Step Advance On STEP
Step 1

TCU	SP	'C
Sides	40	42
Rotors	40	42
Door	40	40

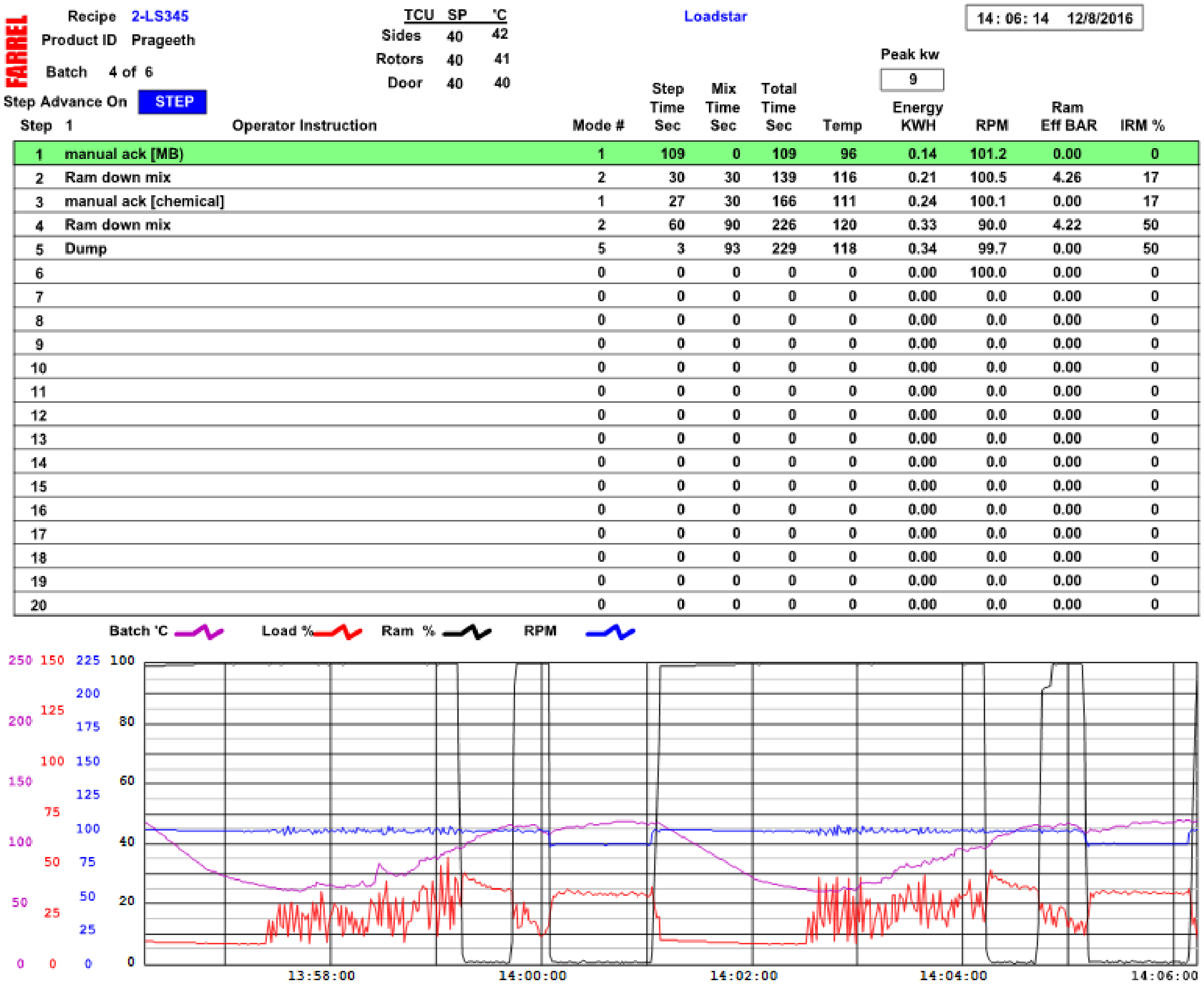
Loadstar

14 : 10 : 41 12/8/2016

Peak kw 9

Step	Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	manual ack [MB)	1	101	0	101	94	0.13	100.9	0.00	0
2	Ram down mix	2	30	30	131	115	0.19	100.3	4.22	17
3	manual ack [chemical]	1	19	30	150	112	0.21	99.4	0.00	17
4	Ram down mix	2	60	90	210	117	0.30	89.5	4.22	54
5	Dump	5	3	93	213	117	0.31	99.6	0.00	54
6		0	0	0	0	0	0.00	100.0	0.00	0
7		0	0	0	0	0	0.00	0.0	0.00	0
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
15		0	0	0	0	0	0.00	0.0	0.00	0
16		0	0	0	0	0	0.00	0.0	0.00	0
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

Figure A 2,10: 1.5 phr Fiber content



FARREL

Recipe 2-LS345
Product ID Prageeth
Batch 4 of 6
Step Advance On **STEP**

TCU	SP	'C
Sides	40	42
Rotors	40	41
Door	40	40

Loadstar

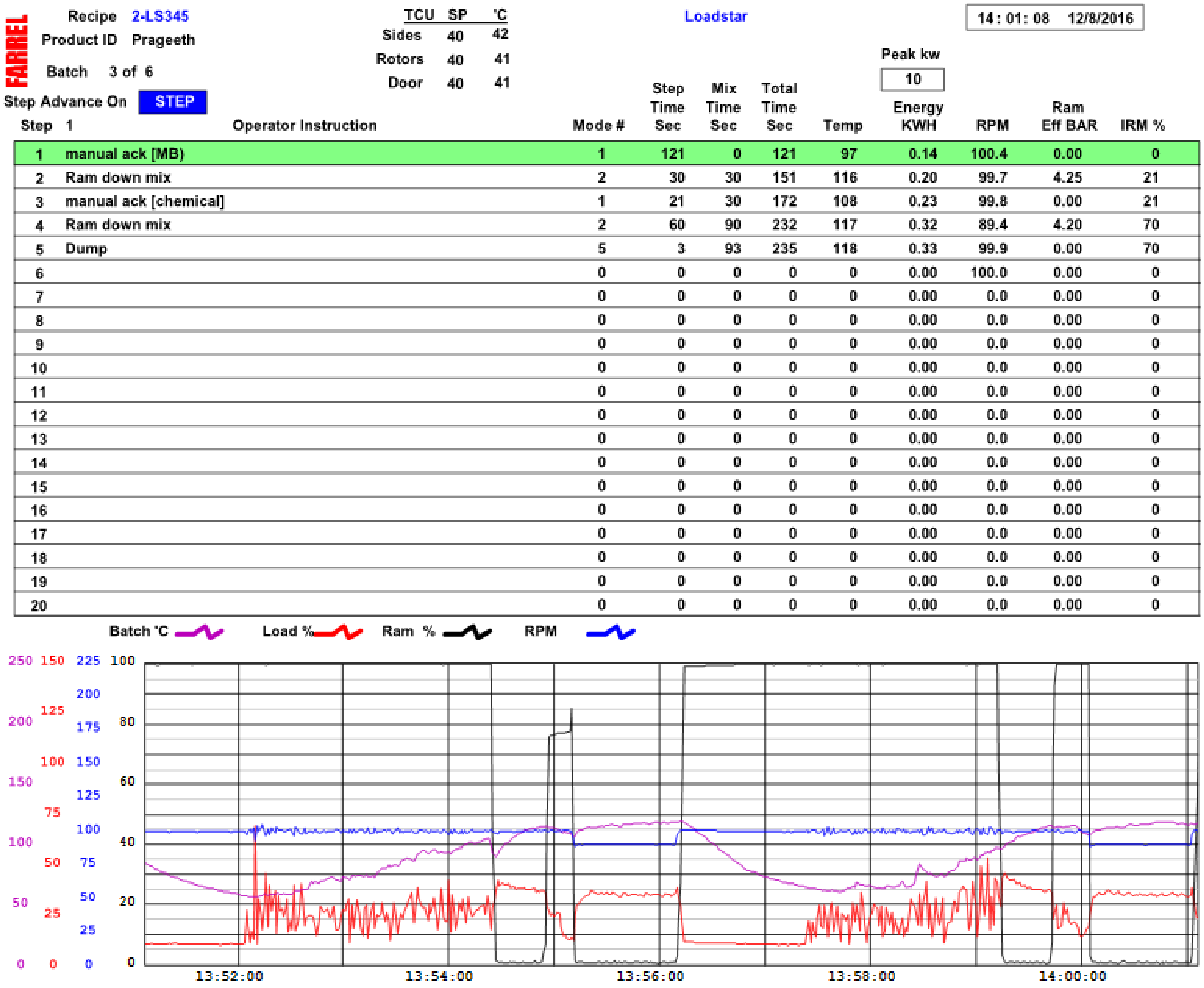
14: 06: 14 12/8/2016

Peak kw 9

Step 1

Step	Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	manual ack [MB)	1	109	0	109	96	0.14	101.2	0.00	0
2	Ram down mix	2	30	30	139	116	0.21	100.5	4.26	17
3	manual ack [chemical]	1	27	30	166	111	0.24	100.1	0.00	17
4	Ram down mix	2	60	90	226	120	0.33	90.0	4.22	50
5	Dump	5	3	93	229	118	0.34	99.7	0.00	50
6		0	0	0	0	0	0.00	100.0	0.00	0
7		0	0	0	0	0	0.00	0.0	0.00	0
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
15		0	0	0	0	0	0.00	0.0	0.00	0
16		0	0	0	0	0	0.00	0.0	0.00	0
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

Figure A 2.11: 2 phr Fiber content



FARREL

Recipe 2-LS345
Product ID Prageeth
Batch 3 of 6
Step Advance On STEP

TCU	SP	'C
Sides	40	42
Rotors	40	41
Door	40	41

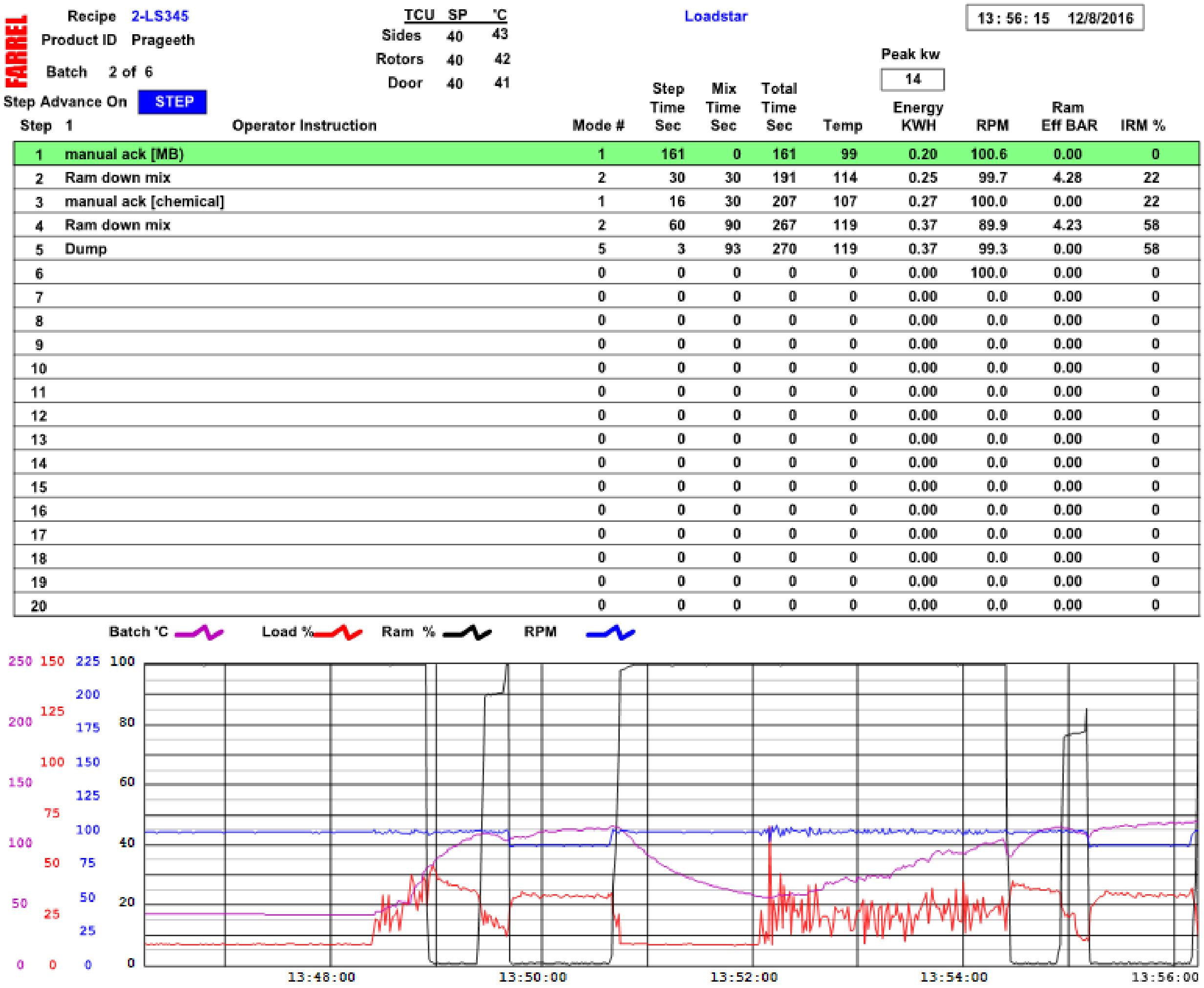
Loadstar

14 : 01 : 08 12/8/2016

Peak kw 10

Step	1 Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	manual ack [MB)	1	121	0	121	97	0.14	100.4	0.00	0
2	Ram down mix	2	30	30	151	116	0.20	99.7	4.25	21
3	manual ack [chemical]	1	21	30	172	108	0.23	99.8	0.00	21
4	Ram down mix	2	60	90	232	117	0.32	89.4	4.20	70
5	Dump	5	3	93	235	118	0.33	99.9	0.00	70
6		0	0	0	0	0	0.00	100.0	0.00	0
7		0	0	0	0	0	0.00	0.0	0.00	0
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
15		0	0	0	0	0	0.00	0.0	0.00	0
16		0	0	0	0	0	0.00	0.0	0.00	0
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

Figure A 2.12: 3 phr Fiber content



FARREL

Recipe 2-LS345
Product ID Prageeth
Batch 2 of 6
Step Advance On STEP

TCU	SP	'C
Sides	40	43
Rotors	40	42
Door	40	41

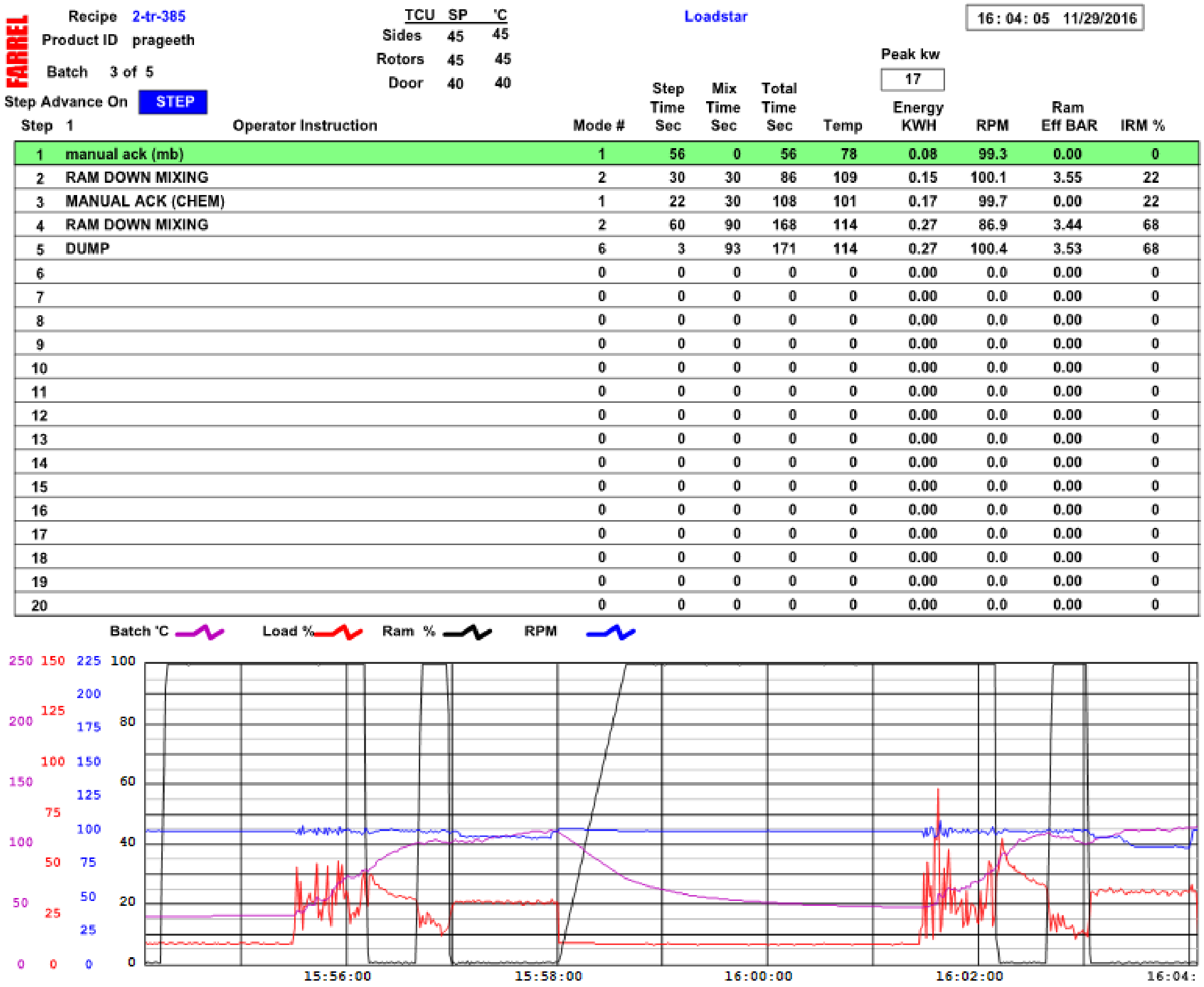
Loadstar

13 : 56 : 15 12/8/2016

Peak kw 14

Step 1	Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	manual ack [MB)	1	161	0	161	99	0.20	100.6	0.00	0
2	Ram down mix	2	30	30	191	114	0.25	99.7	4.28	22
3	manual ack [chemical]	1	16	30	207	107	0.27	100.0	0.00	22
4	Ram down mix	2	60	90	267	119	0.37	89.9	4.23	58
5	Dump	5	3	93	270	119	0.37	99.3	0.00	58
6		0	0	0	0	0	0.00	100.0	0.00	0
7		0	0	0	0	0	0.00	0.0	0.00	0
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
15		0	0	0	0	0	0.00	0.0	0.00	0
16		0	0	0	0	0	0.00	0.0	0.00	0
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

Figure A 2.13: 10 phr Fiber content



FARREL

Recipe 2-tr-385
Product ID prageeth
Batch 3 of 5
Step Advance On STEP

TCU	SP	'C
Sides	45	45
Rotors	45	45
Door	40	40

Loadstar

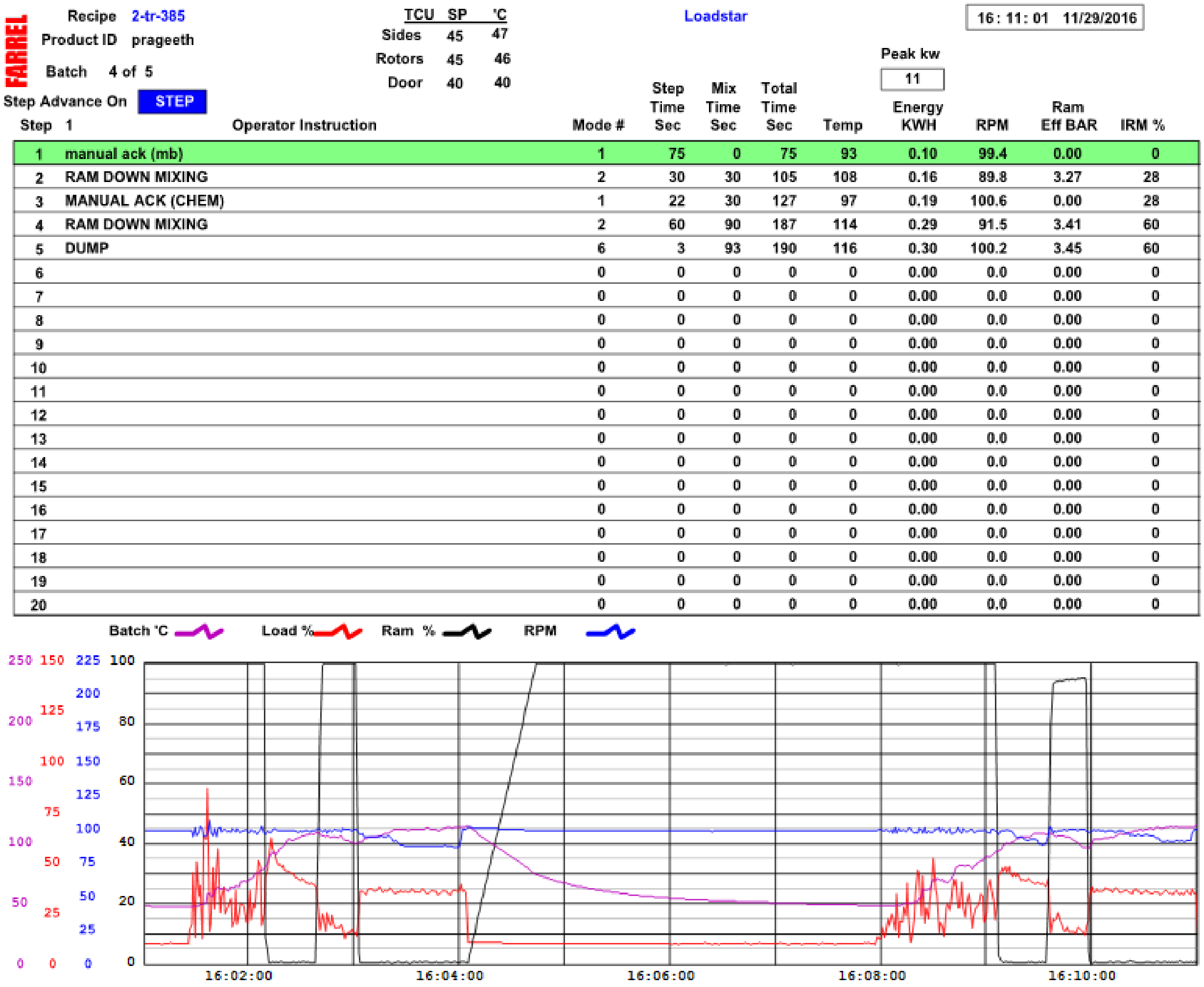
16: 04: 05 11/29/2016

Peak kw 17

Step 1

Step	Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	manual ack (mb)	1	56	0	56	78	0.08	99.3	0.00	0
2	RAM DOWN MIXING	2	30	30	86	109	0.15	100.1	3.55	22
3	MANUAL ACK (CHEM)	1	22	30	108	101	0.17	99.7	0.00	22
4	RAM DOWN MIXING	2	60	90	168	114	0.27	86.9	3.44	68
5	DUMP	6	3	93	171	114	0.27	100.4	3.53	68
6		0	0	0	0	0	0.00	0.0	0.00	0
7		0	0	0	0	0	0.00	0.0	0.00	0
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
15		0	0	0	0	0	0.00	0.0	0.00	0
16		0	0	0	0	0	0.00	0.0	0.00	0
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

Figure A 2.14: 15 phr Fiber content



FARREL

Recipe 2-tr-385
Product ID prageeth
Batch 4 of 5
Step Advance On **STEP**

TCU	SP	'C
Sides	45	47
Rotors	45	46
Door	40	40

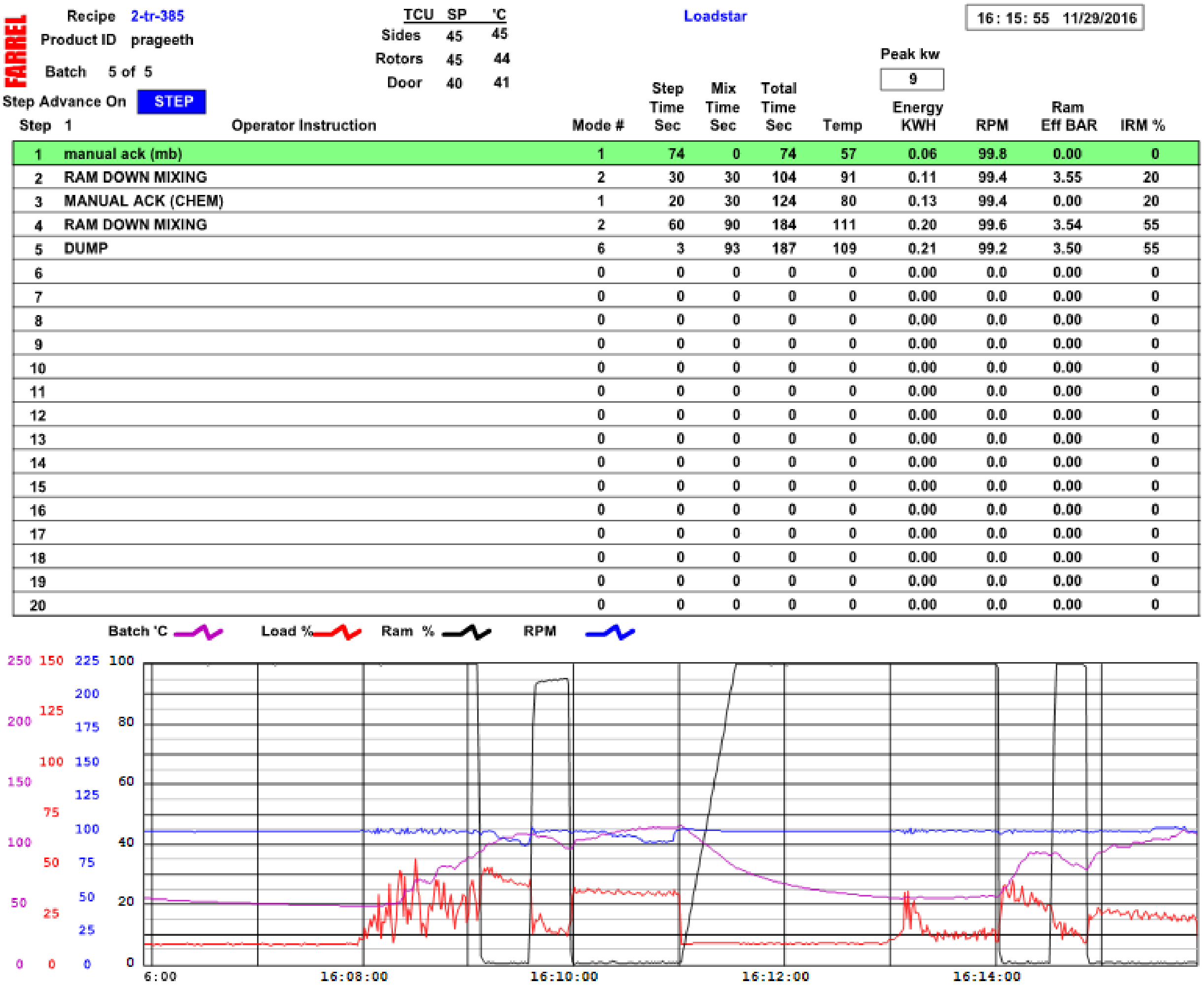
Loadstar

16: 11: 01 11/29/2016

Peak kw
11

Step	1 Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	manual ack (mb)	1	75	0	75	93	0.10	99.4	0.00	0
2	RAM DOWN MIXING	2	30	30	105	108	0.16	89.8	3.27	28
3	MANUAL ACK (CHEM)	1	22	30	127	97	0.19	100.6	0.00	28
4	RAM DOWN MIXING	2	60	90	187	114	0.29	91.5	3.41	60
5	DUMP	6	3	93	190	116	0.30	100.2	3.45	60
6		0	0	0	0	0	0.00	0.0	0.00	0
7		0	0	0	0	0	0.00	0.0	0.00	0
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
15		0	0	0	0	0	0.00	0.0	0.00	0
16		0	0	0	0	0	0.00	0.0	0.00	0
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

Figure A 2.15: 20 phr Fiber content



FARREL

Recipe 2-tr-385
Product ID prageeth
Batch 5 of 5
Step Advance On **STEP**

TCU	SP	'C
Sides	45	45
Rotors	45	44
Door	40	41

Loadstar

16 : 15 : 55 11/29/2016

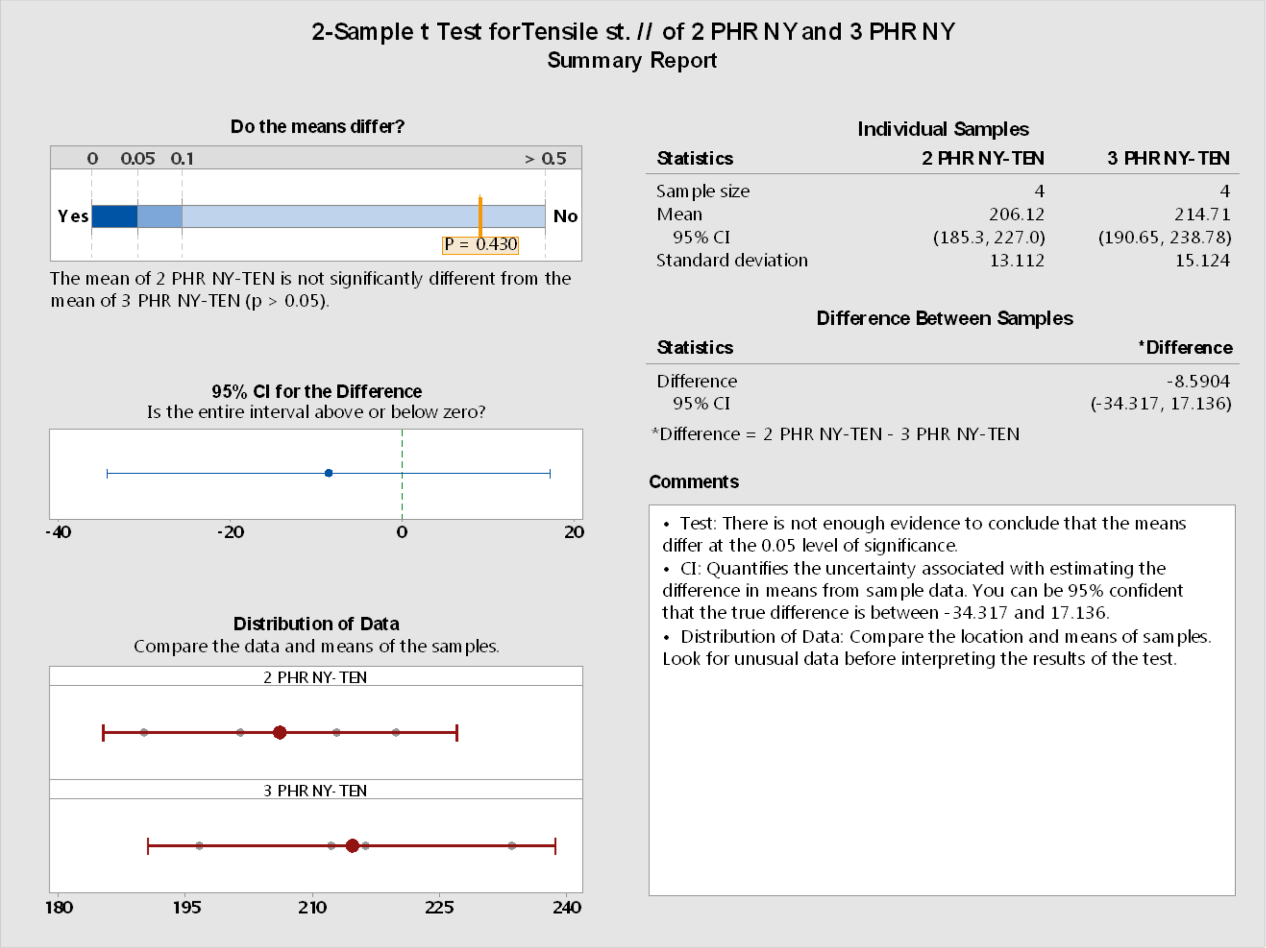
Peak kw 9

Step 1	Operator Instruction	Mode #	Step Time Sec	Mix Time Sec	Total Time Sec	Temp	Energy KWH	RPM	Ram Eff BAR	IRM %
1	manual ack (mb)	1	74	0	74	57	0.06	99.8	0.00	0
2	RAM DOWN MIXING	2	30	30	104	91	0.11	99.4	3.55	20
3	MANUAL ACK (CHEM)	1	20	30	124	80	0.13	99.4	0.00	20
4	RAM DOWN MIXING	2	60	90	184	111	0.20	99.6	3.54	55
5	DUMP	6	3	93	187	109	0.21	99.2	3.50	55
6		0	0	0	0	0	0.00	0.0	0.00	0
7		0	0	0	0	0	0.00	0.0	0.00	0
8		0	0	0	0	0	0.00	0.0	0.00	0
9		0	0	0	0	0	0.00	0.0	0.00	0
10		0	0	0	0	0	0.00	0.0	0.00	0
11		0	0	0	0	0	0.00	0.0	0.00	0
12		0	0	0	0	0	0.00	0.0	0.00	0
13		0	0	0	0	0	0.00	0.0	0.00	0
14		0	0	0	0	0	0.00	0.0	0.00	0
15		0	0	0	0	0	0.00	0.0	0.00	0
16		0	0	0	0	0	0.00	0.0	0.00	0
17		0	0	0	0	0	0.00	0.0	0.00	0
18		0	0	0	0	0	0.00	0.0	0.00	0
19		0	0	0	0	0	0.00	0.0	0.00	0
20		0	0	0	0	0	0.00	0.0	0.00	0

APPENDIX 3

MINITAB RESULT SHEET

Figure A 3.1: Tensile test Results (Longitudinal or parallel to fiber oriented direction)



2-Sample t Test for Tensile st. // of 2 PHR NY and 3 PHR NY
Summary Report

Do the means differ?

The mean of 2 PHR NY-TEN is not significantly different from the mean of 3 PHR NY-TEN (p > 0.05).

95% CI for the Difference
Is the entire interval above or below zero?

Distribution of Data
Compare the data and means of the samples.

Individual Samples

Statistics	**2 PHR NY-TEN**	**3 PHR NY-TEN**
Sample size	4	4
Mean	206.12	214.71
95% CI	(185.3, 227.0)	(190.65, 238.78)
Standard deviation	13.112	15.124

Difference Between Samples

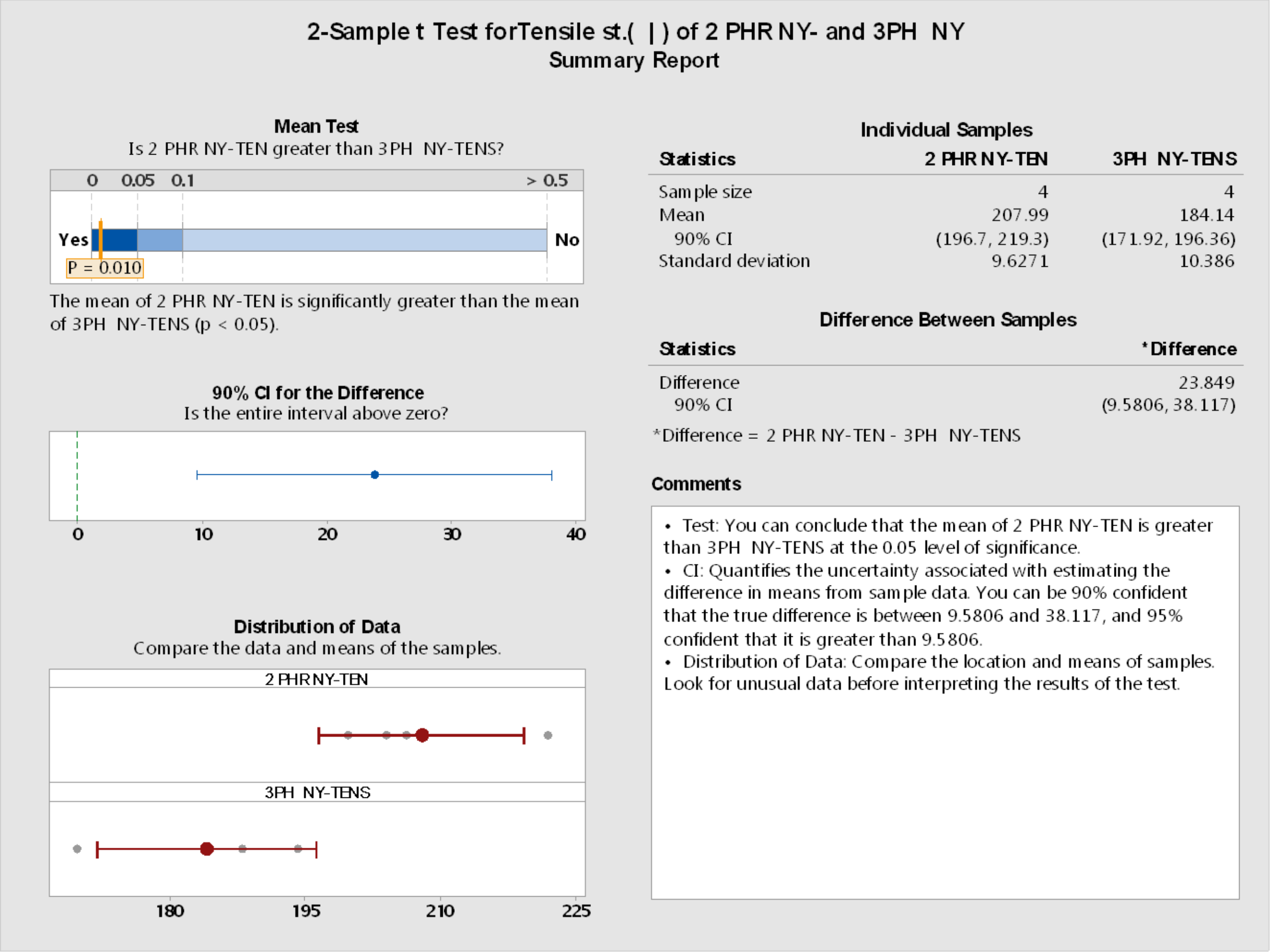
Statistics	***Difference**
Difference	-8.5904
95% CI	(-34.317, 17.136)

*Difference = 2 PHR NY-TEN - 3 PHR NY-TEN

Comments

- Test: There is not enough evidence to conclude that the means differ at the 0.05 level of significance.
- CI: Quantifies the uncertainty associated with estimating the difference in means from sample data. You can be 95% confident that the true difference is between -34.317 and 17.136.
- Distribution of Data: Compare the location and means of samples. Look for unusual data before interpreting the results of the test.

Figure A 3.2: Tensile test Results (Transvers or perpendicular to fiber oriented direction)



2-Sample t Test forTensile st.(|) of 2 PHR NY- and 3PH NY
Summary Report

Mean Test
Is 2 PHR NY-TEN greater than 3PH NY-TENS?

The mean of 2 PHR NY-TEN is significantly greater than the mean of 3PH NY-TENS (p < 0.05).

90% CI for the Difference
Is the entire interval above zero?

Distribution of Data
Compare the data and means of the samples.

Individual Samples

Statistics	**2 PHR NY-TEN**	**3PH NY-TENS**
Sample size	4	4
Mean	207.99	184.14
90% CI	(196.7, 219.3)	(171.92, 196.36)
Standard deviation	9.6271	10.386

Difference Between Samples

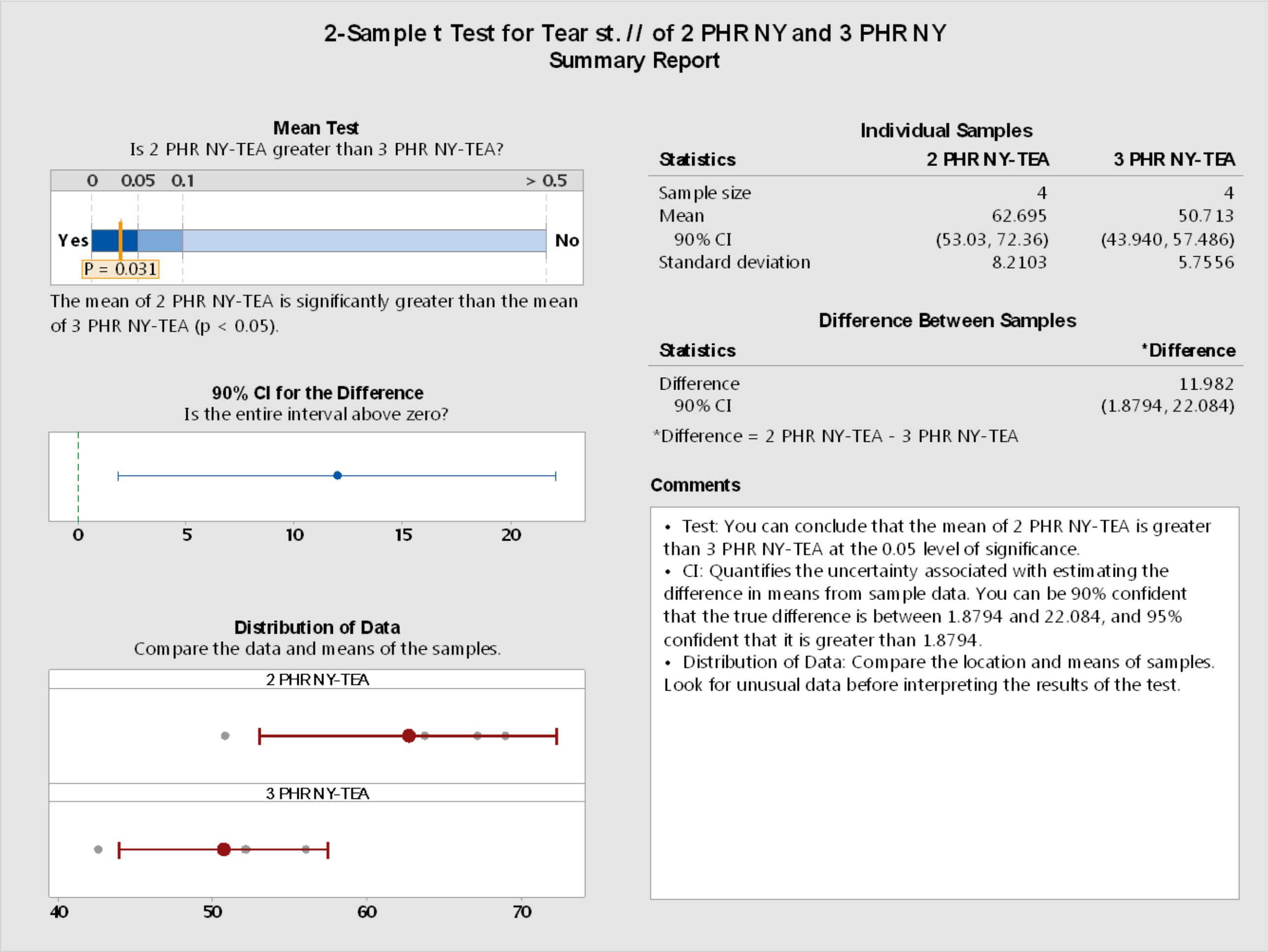
Statistics	***Difference**
Difference	23.849
90% CI	(9.5806, 38.117)

*Difference = 2 PHR NY-TEN - 3PH NY-TENS

Comments

- Test: You can conclude that the mean of 2 PHR NY-TEN is greater than 3PH NY-TENS at the 0.05 level of significance.
- CI: Quantifies the uncertainty associated with estimating the difference in means from sample data. You can be 90% confident that the true difference is between 9.5806 and 38.117, and 95% confident that it is greater than 9.5806.
- Distribution of Data: Compare the location and means of samples. Look for unusual data before interpreting the results of the test.

Figure A 3.3: Tearing test Results (Longitudinal or parallel to fiber oriented direction)



2-Sample t Test for Tear st. // of 2 PHR NY and 3 PHR NY
Summary Report

Mean Test
Is 2 PHR NY-TEA greater than 3 PHR NY-TEA?

The mean of 2 PHR NY-TEA is significantly greater than the mean of 3 PHR NY-TEA (p < 0.05).

90% CI for the Difference
Is the entire interval above zero?

Distribution of Data
Compare the data and means of the samples.

Individual Samples

Statistics	**2 PHR NY-TEA**	**3 PHR NY-TEA**
Sample size	4	4
Mean	62.695	50.713
90% CI	(53.03, 72.36)	(43.940, 57.486)
Standard deviation	8.2103	5.7556

Difference Between Samples

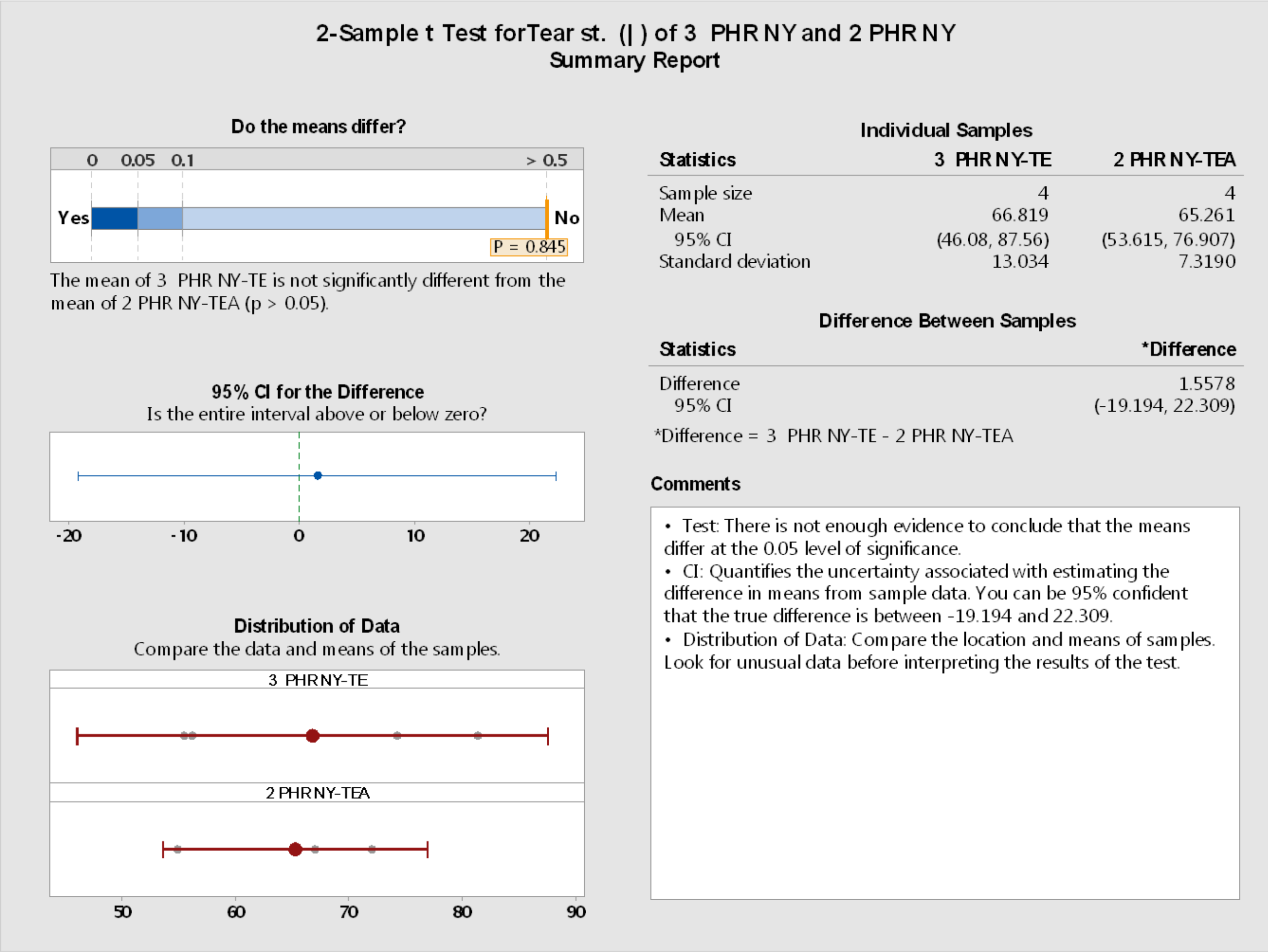
Statistics	***Difference**
Difference	11.982
90% CI	(1.8794, 22.084)

*Difference = 2 PHR NY-TEA - 3 PHR NY-TEA

Comments

- Test: You can conclude that the mean of 2 PHR NY-TEA is greater than 3 PHR NY-TEA at the 0.05 level of significance.
- CI: Quantifies the uncertainty associated with estimating the difference in means from sample data. You can be 90% confident that the true difference is between 1.8794 and 22.084, and 95% confident that it is greater than 1.8794.
- Distribution of Data: Compare the location and means of samples. Look for unusual data before interpreting the results of the test.

Figure A 3.4: Tearing test Results (Transvers or perpendicular to fiber oriented direction)



2-Sample t Test for Tear st. (|) of 3 PHR NY and 2 PHR NY
Summary Report

Do the means differ?

The mean of 3 PHR NY-TE is not significantly different from the mean of 2 PHR NY-TEA (p > 0.05).

95% CI for the Difference
Is the entire interval above or below zero?

Distribution of Data
Compare the data and means of the samples.

Individual Samples

Statistics	3 PHR NY-TE	2 PHR NY-TEA
Sample size	4	4
Mean	66.819	65.261
95% CI	(46.08, 87.56)	(53.615, 76.907)
Standard deviation	13.034	7.3190

Difference Between Samples

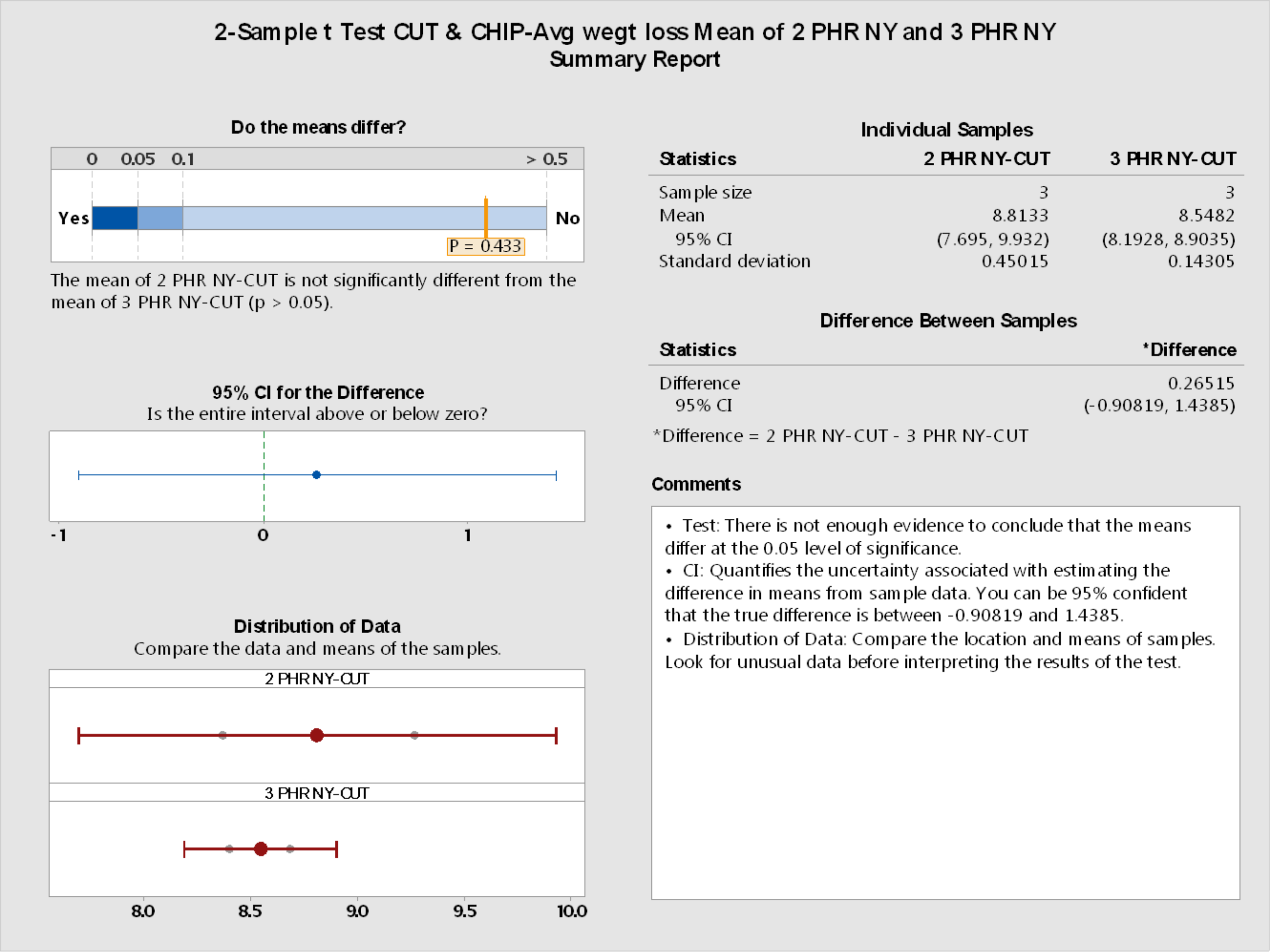
Statistics	*Difference
Difference	1.5578
95% CI	(-19.194, 22.309)

*Difference = 3 PHR NY-TE - 2 PHR NY-TEA

Comments

- Test: There is not enough evidence to conclude that the means differ at the 0.05 level of significance.
- CI: Quantifies the uncertainty associated with estimating the difference in means from sample data. You can be 95% confident that the true difference is between -19.194 and 22.309.
- Distribution of Data: Compare the location and means of samples. Look for unusual data before interpreting the results of the test.

Figure A 3.5: Cut & Chip test Results (Average Weight loss)



2-Sample t Test CUT & CHIP-Avg wegt loss Mean of 2 PHR NY and 3 PHR NY
Summary Report

Do the means differ?

The mean of 2 PHR NY-CUT is not significantly different from the mean of 3 PHR NY-CUT (p > 0.05).

95% CI for the Difference

Is the entire interval above or below zero?

Distribution of Data

Compare the data and means of the samples.

Individual Samples

Statistics	**2 PHR NY-CUT**	**3 PHR NY-CUT**
Sample size	3	3
Mean	8.8133	8.5482
95% CI	(7.695, 9.932)	(8.1928, 8.9035)
Standard deviation	0.45015	0.14305

Difference Between Samples

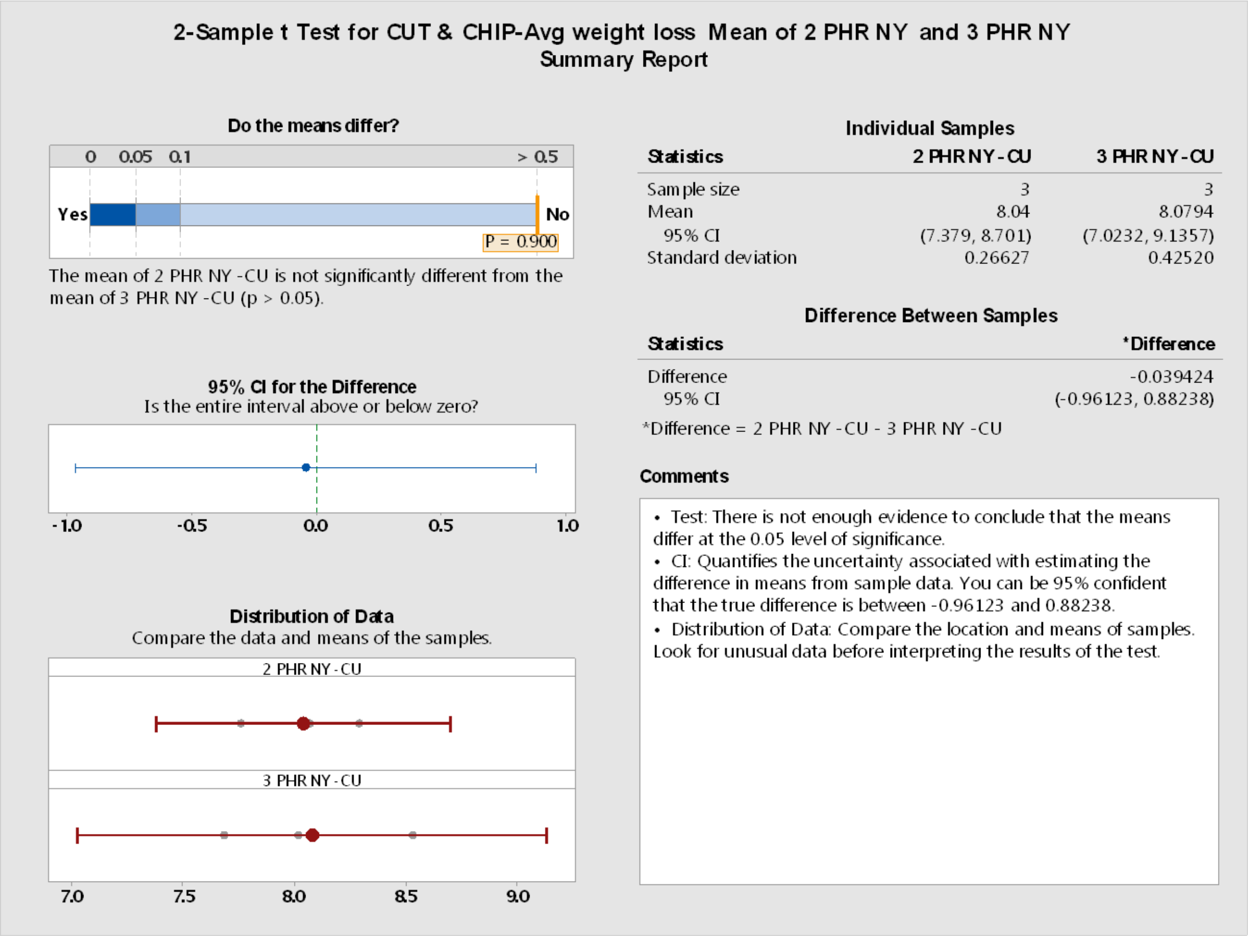
Statistics	***Difference**
Difference	0.26515
95% CI	(-0.90819, 1.4385)

*Difference = 2 PHR NY-CUT - 3 PHR NY-CUT

Comments

- Test: There is not enough evidence to conclude that the means differ at the 0.05 level of significance.
- CI: Quantifies the uncertainty associated with estimating the difference in means from sample data. You can be 95% confident that the true difference is between -0.90819 and 1.4385.
- Distribution of Data: Compare the location and means of samples. Look for unusual data before interpreting the results of the test.

Figure A 3.6: Cut & Chip test Results (Average Diameter loss)



2-Sample t Test for CUT & CHIP-Avg weight loss Mean of 2 PHR NY and 3 PHR NY
Summary Report

Do the means differ?

The mean of 2 PHR NY -CU is not significantly different from the mean of 3 PHR NY -CU (p > 0.05).

95% CI for the Difference
Is the entire interval above or below zero?

Distribution of Data
Compare the data and means of the samples.

Individual Samples

Statistics	**2 PHR NY - CU**	**3 PHR NY - CU**
Sample size	3	3
Mean	8.04	8.0794
95% CI	(7.379, 8.701)	(7.0232, 9.1357)
Standard deviation	0.26627	0.42520

Difference Between Samples

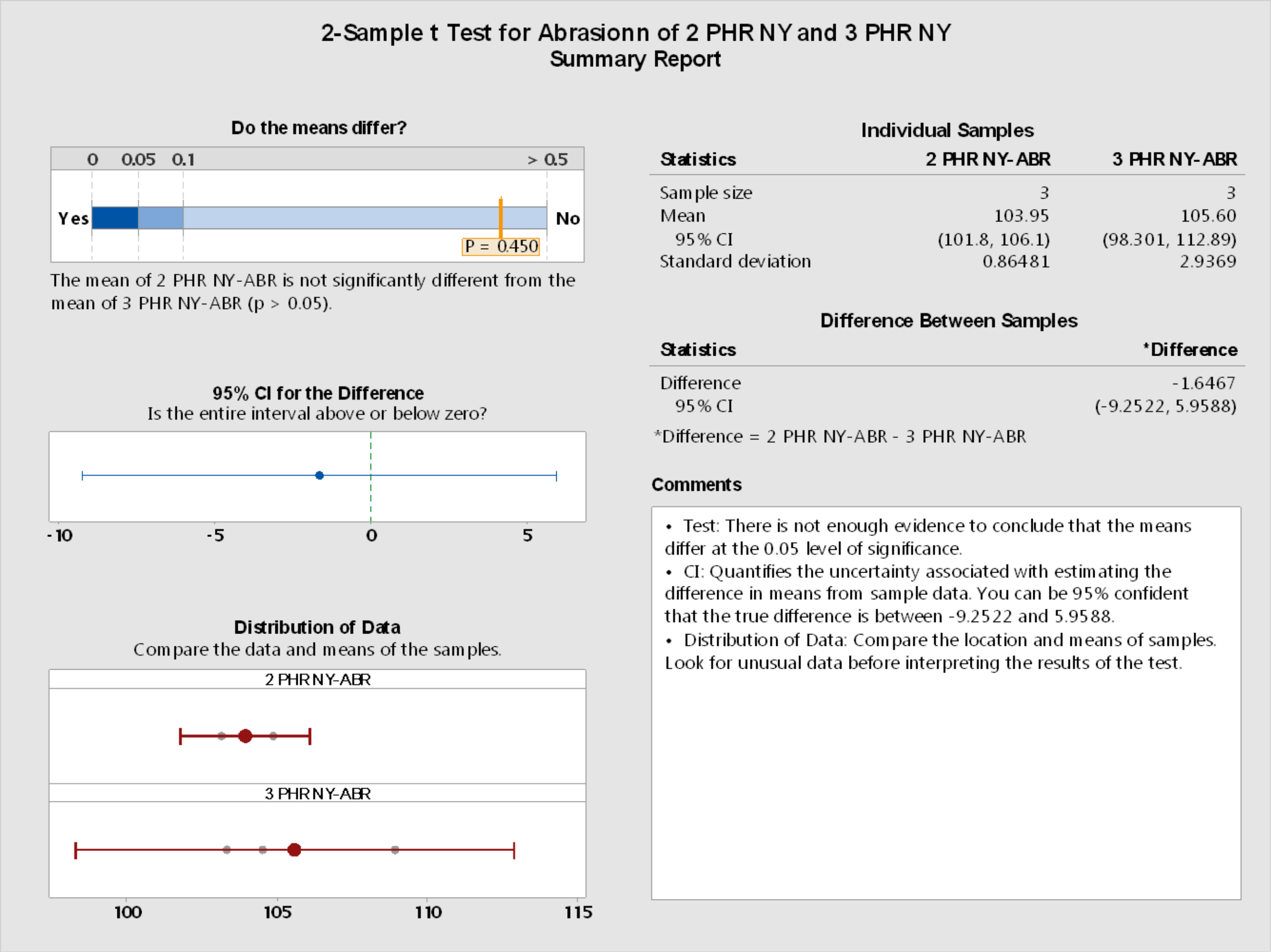
Statistics	***Difference**
Difference	-0.039424
95% CI	(-0.96123, 0.88238)

*Difference = 2 PHR NY -CU - 3 PHR NY -CU

Comments

- Test: There is not enough evidence to conclude that the means differ at the 0.05 level of significance.
- CI: Quantifies the uncertainty associated with estimating the difference in means from sample data. You can be 95% confident that the true difference is between -0.96123 and 0.88238.
- Distribution of Data: Compare the location and means of samples. Look for unusual data before interpreting the results of the test.

Figure A 3.7: Abrasion test Results



2-Sample t Test for Abrasionn of 2 PHR NY and 3 PHR NY
Summary Report

Do the means differ?

The mean of 2 PHR NY-ABR is not significantly different from the mean of 3 PHR NY-ABR (p > 0.05).

95% CI for the Difference
Is the entire interval above or below zero?

Distribution of Data
Compare the data and means of the samples.

Individual Samples

Statistics	2 PHR NY-ABR	3 PHR NY-ABR
Sample size	3	3
Mean	103.95	105.60
95% CI	(101.8, 106.1)	(98.301, 112.89)
Standard deviation	0.86481	2.9369

Difference Between Samples

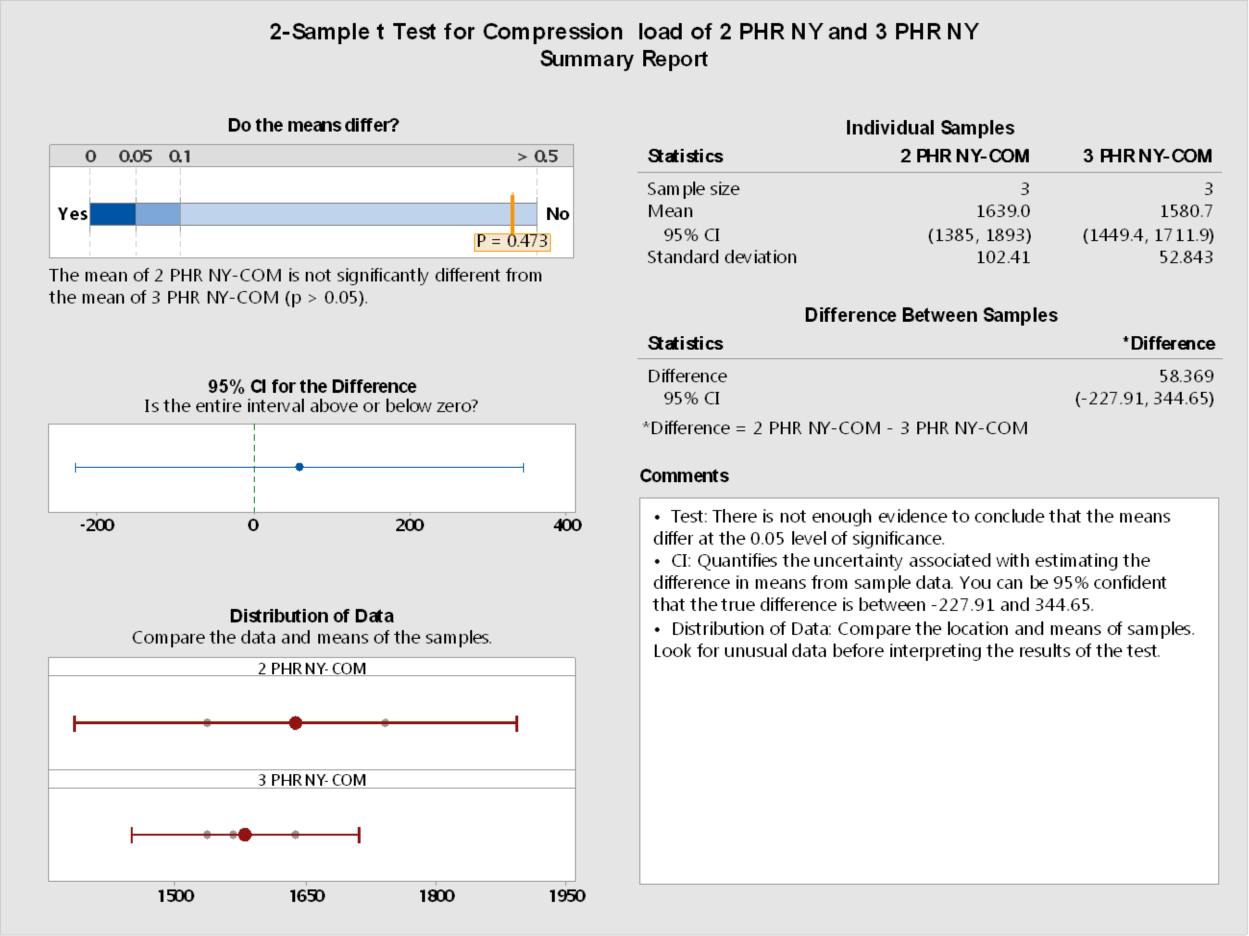
Statistics	*Difference
Difference	-1.6467
95% CI	(-9.2522, 5.9588)

*Difference = 2 PHR NY-ABR - 3 PHR NY-ABR

Comments

- Test: There is not enough evidence to conclude that the means differ at the 0.05 level of significance.
- CI: Quantifies the uncertainty associated with estimating the difference in means from sample data. You can be 95% confident that the true difference is between -9.2522 and 5.9588.
- Distribution of Data: Compare the location and means of samples. Look for unusual data before interpreting the results of the test.

Figure A 3.8: Compression test Results



2-Sample t Test for Compression load of 2 PHR NY and 3 PHR NY
Summary Report

Do the means differ?

The mean of 2 PHR NY-COM is not significantly different from the mean of 3 PHR NY-COM (p > 0.05).

95% CI for the Difference
Is the entire interval above or below zero?

Distribution of Data
Compare the data and means of the samples.

Individual Samples

Statistics	2 PHR NY-COM	3 PHR NY-COM
Sample size	3	3
Mean	1639.0	1580.7
95% CI	(1385, 1893)	(1449.4, 1711.9)
Standard deviation	102.41	52.843

Difference Between Samples

Statistics	*Difference
Difference	58.369
95% CI	(-227.91, 344.65)

*Difference = 2 PHR NY-COM - 3 PHR NY-COM

Comments

- Test: There is not enough evidence to conclude that the means differ at the 0.05 level of significance.
- CI: Quantifies the uncertainty associated with estimating the difference in means from sample data. You can be 95% confident that the true difference is between -227.91 and 344.65.
- Distribution of Data: Compare the location and means of samples. Look for unusual data before interpreting the results of the test.