

“ANALYSIS OF CARBON BLACK PROPORTION ON DURABILITY AND WEAR BEHAVIOR OF EPDM RUBBER”

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ABSTRACT: The present day scenario in the industrial sector demands high productivity and the life of rubber components. EPDM rubber is widely used in all type of non-tyre applications. Addition of filler materials enhances the properties of an elastomer by improving its mechanical properties such as the hardness, abrasion resistance, durability or the fatigue life. In order to design a product and understand the properties of the filled elastomers used in industry; it is important to understand the effect of a filler on the elastomer.

This work is focused on studying the effect of composition of carbon black type on the mechanical properties of EPDM rubber characterization. Our sponsor's problem was durability of component. It was observed that failure occur due to wear. To increase both durability and wear resistance, different tests were performed on filled EPDM rubber with two different kinds of carbon black N330 and N550. All tests were performed at room temperature. The tests include hardness tests, tensile tests, hot air aging test, wear tests, and durability test.

The findings show that the combination of carbon black improves the wear resistance and durability of EPDM rubber.

Keywords: EPDM rubber, durability, carbon black, wears resistance.

• INTRODUCTION

1.1 GENERAL

Rubber materials are widely used in a wide range of engineering applications. The term rubber does not only describe natural rubber, but is also used to describe any materials exhibiting rubber-like behaviour. The term elastomer is also used to describe materials exhibiting similar properties to rubber. Typical components made from elastomer materials include vehicle tyres, springs, seals, conveyer belts and shoe soles. More than half of the natural rubber and synthetic rubber produced in the world are consumed by the tyre industry

Although elastomeric materials exhibit very high strains for relatively small stresses, making them appropriate for many automotive applications, they still have behaviour in common with other materials. In particular they a limiting strength and tend to fatigue. Mechanical fatigue of elastomers is manifested in a progressive reduction of the physical properties as a result of crack propagation during continuous dynamic excitation. Well founded research into metals has shown that fatigue results from atomic and molecular processes. Similar research for elastomers is at a very early stage, because of the complex interaction between polymers, fillers, softeners and other additives [1].

Understanding the factors that affect the fatigue life of rubber is helpful in at least three ways. First, at least a qualitative understanding of these factors is required in order to develop durable rubber compounds, rubber components and systems that rely upon rubber components. Second, the analysis and prediction of fatigue failure in rubber components depends on a quantitative understanding of these factors. Finally, it should be acknowledged that many aspects of the fatigue failure process remain incompletely understood. Prior reviews identify many factors that affect the fatigue crack nucleation and growth process in rubber [3].

Ethylene-propylene rubber continues to be one of the most widely used and fastest growing synthetic rubber having both specialty and general purpose applications. Polymerization and catalyst technologies in use today provide the ability to design polymers to meet specific and demanding application and processing needs. Versatility in polymer design and performance has resulted in broad usage in automotive weather-stripping and seals, glass run channel, radiator, garden and appliance hose, tubing, belts, electrical insulation, roofing membrane, rubber mechanical goods, plastic impact modification, thermoplastic vulcanizates and motor oil additive applications. Ethylene-propylene rubber are valuable for their excellent resistance to heat, oxidation, ozone and weather aging due to their stable, saturated polymer backbone structure. Properly pigmented black and non-black compounds are color stable. [18]

Typical rubber compounds used to manufacture industrial products contain up to eight classes of chemical additives including curing agents, accelerators, activators, processing aids, and antidegradants. The cure systems in these articles often consists of primary and secondary accelerators, primary and secondary activators, and elemental sulphur. [6]

Filler reinforcement of rubbers has been an area of interest to the rubber industry for more than a century. [14] Reinforcing fillers include carbon blacks, silica and resins, which increase the strength of vulcanized rubber more than tenfold. Filling carbon blacks in elastomers and plastics also reduces the cost of the end product and modifies the electrical and optical properties of the polymer matrix. Reinforcement of rubber by carbon blacks has been intensively studied Carbon black is used as a reinforcement to increase the structural and thermal properties of rubber. When a rubber component filled with carbon black is under loading, the rubber matrix around carbon black is strained more than the rest. It is generally accepted that the reinforcement of elastomers and the improvement of other properties, to a large extent, are associated with the chemical and physical interactions between the polymer matrix and carbon blacks. Thus, researchers try to explain the improved performance of rubber from the microscopic view, relating the observation of cure kinetics and mechanical properties with the interactions between elastomers and carbon blacks. The entanglement of rubber molecules on the carbon black surface is supposed to play an important role for rubber attachment on carbon blacks. Therefore, it is important to figure out the characteristics and effects of carbon black in order to understand the material behavior of rubber. [4, 7]

A detailed understanding of the filler reinforcement should provide an insight into the increase in modulus and strength witnessed with as well as helping to understand the phenomena such as cyclic stress softening, abrasion or changes in the fatigue life resistance. [14]

Accordingly, recent targets of the material development are improved reliability, prolonged lifetime, and maintenance-free service. Needless to say that this demand can only be met when the material development is accompanied by further measures, such as proper construction, design, and suitable manufacturing processes. To be able to fulfill the industrial expectations the processes during dry sliding need to be studied in detail. It is not clear yet what is the effect of temperature (changing during the tribotesting) and how the actual testing parameters affect the material behavior in respect to sliding and wear properties. It is also unknown whether chemical changes take place in the elastomers due to the probably high flash temperatures in the contact area. [11]

Abrasion resistance is the ability of a material to resist mechanical action such as rubbing, scraping, or erosion that tends progressively to remove material from its surface. When a product has abrasion resistance, it will resist erosion caused by scraping, rubbing, and other types of mechanical wear.

In most elastomeric materials, when the surfaces are abraded by harder material such as a razor blade, sand paper or a road surface without changing the direction, a set of periodic parallel ridged pattern are formed on the surface. These patterns are called abrasion patterns, and arise consistently in all forms of elastomer abrasion, on the surface of tyres, conveyor belts, printing rolls and shoe soles for example. As a result they are regarded as an essential basic phenomenon of elastomer abrasion. The appearance of an abrasion pattern decreases the abrasion resistance of the rubber. Several factors such as changes to the material properties, the loading conditions, the testing temperature, the geometry of the abrader, the moisture content in the air and the presence of liquids at the surface all alter the rate of abrasion , indicating that the exact mechanism that controls the abrasion process is complex. [7]

The main objectives of the present work were: (1) to quantify the improvement in hardness and the wear resistance (WR) of the carbon black filled EPDM rubber, (2) to find out optimum carbon black material composition for durability of rubber.

- **EPDM RUBBER, PROCESSING AND TRIBOLOGY**
- **ELASTOMERIC MATERIALS**

Elastomers are built up from long hydrocarbon polymer chains, which are connected with each other by chemical bonds. However, due to the steady developments within the class of elastomers is more proper to say that a material can be classified as elastomer which can suffer several hundred percent of strain without significant irreversible (plastic) deformation . This “framing” allow us to consider as elastomers also those thermoplastic or hybrid polymeric materials which fulfill the upper requirement. [11]

Among others the huge deformability and unusual thermal behaviors of the elastomers amaze the scientists and explorers since more than hundred years. However, the large scale technical usage of rubber becomes possible only after the discovery of the vulcanization technique (credited to Goodyear, 1839) The uncured rubber was also used for example as eraser (since the name “rubber” from Priestly) or insulator material

in different medical applications. The first trials with rubber products were short timed because of the fast degradation of the uncured material that became fast sticky and smelly.[11]

The modern life history of natural rubber can be dated back to 11 June 1496, the day on which Christopher Columbus returned from his second voyage, bringing back the first rubber balls from the West Indies. The next landmark was the Spanish discovery of the use of latex for the water proofing of leather and fabrics in 1615. The rubber industry in Europe really started with Charles Macintosh in 1818, when he started exploiting the naphtha-based rubber solution as a water proofing layer between two fabrics. Two years later, in 1820, Thomas Hancock discovered mastication. The contributions of Sir Henry Wickham and Henry Ridley in rubber plantation also added on to the vast development and fast growth of rubber industry. The final landmark in the early history of rubber was the discovery of vulcanisation by Charles Goodyear in 1839 which revolutionized the use and applications of rubber, and changed the face of the industrial world. Dunlop's patenting of the pneumatic tire in England in 1888 led to the age of the bicycle as a prologue to the era of automobiles. [21]

However, synthetic alternatives remained mostly of academic interest till World War I during which Germany instituted programs to develop synthetic alternatives. Rubbers might be general purpose "diene" elastomers like styrenebutadiene rubber (SBR), Butadiene rubber (BR) and isoprene rubber [both natural (NR) and synthetic (IR)] which are hydrocarbons with significant chemical unsaturation in their backbones primarily used in automobile and truck tyres. The incapability of these general purpose rubbers in dealing with the higher demands of chemical, environmental and temperature resistance, led to the development of specialty elastomers such as polychloroprene (CR), acrylonitrile-butadiene rubber (NBR), hydrogenated nitrile rubber (HNBR), butyl rubber (IIR), ethylene-propylene rubber (EPDM), silicon rubber, etc. [21]

2.1.1 Natural Rubber

Natural rubber (NR) consists primarily of polyisoprene, a high-molecular-weight polymer of isoprene (C₅H₈). It is derived from latex, a milky substance produced by various plants, the most important of which is the rubber tree (*Hevea brasiliensis*) that grows in tropical climates. Latex is a water emulsion of polyisoprene (about one-third by weight), plus various other ingredients. Rubber is extracted from the latex by various methods (e.g., coagulation, drying, and spraying) that remove the water. Natural crude rubber (without vulcanization) is sticky in hot weather, but stiff and brittle in cold weather. To form an elastomer with useful properties, natural rubber must be vulcanized. Traditionally, vulcanization has been accomplished by mixing small amounts of sulfur and other chemicals with the crude rubber and heating. The chemical effect of vulcanization is cross-linking; the mechanical result is increased strength and stiffness, yet maintenance of extensibility.

Sulfur alone can cause cross-linking, but the process is slow, taking hours to complete. Other chemicals are added to sulfur during vulcanization to accelerate the process and serve other beneficial functions. Also, rubber can be vulcanized using chemicals other than sulfur.

Today, curing times have been reduced significantly compared to the original sulfur curing of years ago. As an engineering material, vulcanized rubber is noted among elastomers for its high tensile strength, tear strength, resilience (capacity to recover shape after deformation), and resistance to wear and fatigue. Its weaknesses are that it degrades when subjected to heat, sunlight, oxygen, ozone, and oil. Some of these limitations can be reduced through the use of additives.

2.1.2 Synthetic Rubbers

Today, the tonnage of synthetic rubbers is more than three times that of natural rubber. Development of these synthetic materials was motivated largely by the world wars when NR was difficult to obtain. The most important of the synthetics is styrene-butadiene rubber (SBR), a copolymer of butadiene (C₄H₆) and styrene (C₈H₈). As with most other polymers, the predominant raw material for the synthetic rubbers is petroleum.

Butadiene Rubber Polybutadiene (BR) is important mainly in combination with other rubbers. It is compounded with natural rubber and with styrene (styrene-butadiene rubber is discussed later) in the production of automotive tires. Without compounding, the tear resistance, tensile strength, and ease of processing of polybutadiene are less than desirable. Butyl Rubber Butyl rubber is a copolymer of polyisobutylene (98%–99%) and polyisoprene (1%–2%). It can be vulcanized to provide a rubber with very low air permeability, which has led to applications in inflatable products such as inner tubes, liners in tubeless tires, and sporting goods.

Chloroprene Rubber Polychloroprene was one of the first synthetic rubbers to be developed (early 1930s). Commonly known today as Neoprene, it is an important special purpose rubber. It crystallizes when strained to provide good mechanical properties. Chloroprene rubber (CR) is more resistant to oils, weather, ozone, heat, and flame (chlorine makes this rubber self-extinguishing) than NR, but somewhat more expensive. Its applications include fuel hoses (and other automotive parts), conveyor belts, and gaskets, but not tires.

Ethylene-Propylene Rubber Polymerization of ethylene and propylene with small

proportions (3%–8%) of a diene monomer produces the terpolymer ethylene–propylene diene (EPDM), a useful synthetic rubber. Applications are for parts mostly in the automotive industry other than tires. Other uses are wire and cable insulation.

Isoprene Rubber Isoprene can be polymerized to synthesize a chemical equivalent of natural rubber. Synthetic (unvulcanized) polyisoprene is softer and more easily molded than raw natural rubber. Applications of the synthetic material are similar to those of its natural counterpart, car tires being the largest single market. It is also used for footwear, conveyor belts, and caulking compound. Cost per unit weight is about 35% higher than for NR.

Nitrile Rubber This is a vulcanizable copolymer of butadiene (50%–75%) and acrylonitrile (25%–50%). Its more technical name is butadiene–acrylonitrile rubber. It has good strength and resistance to abrasion, oil, gasoline, and water. These properties make it ideal for applications such as gasoline hoses and seals, and also for footwear.

Polyurethanes Thermosetting polyurethanes with minimum cross-linking are elastomers, most commonly produced as flexible foams. In this form, they are widely used as cushion materials for furniture and automobile seats. Unfoamed polyurethane can be molded into products ranging from shoe soles to car bumpers, with cross-linking adjusted to achieve the desired properties for the application. With no cross-linking, the material is a thermoplastic elastomer that can be injection molded. As an elastomer or thermoset, reaction injection molding and other shaping methods are used.

Silicones Like the polyurethanes, silicones can be elastomeric or thermosetting, depending on the degree of cross-linking. Silicone elastomers are noted for the wide temperature range over which they can be used. Their resistance to oils is poor. The silicones possess various chemistries, the most common being polydimethylsiloxane. To obtain acceptable mechanical properties, silicone elastomers must be reinforced, usually with fine silica powders. Owing to their high cost, they are considered special-purpose rubbers for applications such as gaskets, seals, wire and cable insulation, prosthetic devices, and bases for caulking materials.

Styrene–Butadiene Rubber SBR is a random copolymer of styrene (about 25%) and butadiene (about 75%). It was originally developed in Germany as Buna-S rubber before World War II. Today, it is the largest tonnage elastomer, totaling about 40% of all rubbers produced (natural rubber is second in tonnage). Its attractive features are low cost, resistance to abrasion, and better uniformity than NR. When reinforced with carbon black and vulcanized, its characteristics and applications are very similar to those of natural rubber. Cost is also similar. A close comparison of properties reveals that most of its mechanical properties except wear resistance are inferior to NR, but its resistance to heat aging, ozone, weather, and oils is superior. Applications include automotive tires, footwear, and wire and cable insulation. A material chemically related to SBR is styrene–butadiene–styrene block copolymer, a thermoplastic elastomer discussed below.

Thermoplastic Elastomers As previously described, a thermoplastic elastomer (TPE) is a thermoplastic that behaves like an elastomer. It constitutes a family of polymers that is a fast-growing segment of the elastomer market. TPEs derive their elastomeric properties not from chemical cross-links, but from physical connections between soft and hard phases that make up the material. Thermoplastic elastomers include styrene–butadiene–styrene (SBS), a block copolymer as opposed to styrene–butadiene rubber (SBR) which is a random copolymer; thermoplastic polyurethane; thermo plastic polyester copolymers; and other copolymers and polymer blends. The chemistry and structure of these materials are generally complex, involving two materials that are incompatible so that they form distinct phases whose room temperature properties are different. Owing to their thermoplasticity, the TPEs cannot match conventional cross-linked elastomers in elevated temperature strength and creep resistance. Typical applications include footwear, rubber bands, extruded tubing, wire coating, and molded parts for automotive and other uses in which elastomeric properties are required. TPEs are not suitable for tires.

2.1.3 EPDM rubber

The ethylene–propylene–diene rubber (EPDM) was chosen for investigation, [11] because of its advantageous chemical and mechanical properties. The EPDM rubber is a synthetic elastomer whereby the M- character means that the backbone of the polymer chain is built up only from C-atoms (ISO 1692 or ASTM 1418). The EPDMs are terpolymers of ethylene, propylene and a small amount of a nonconjugated diene, which provides unsaturation in side chains, pendent from the fully saturated backbone

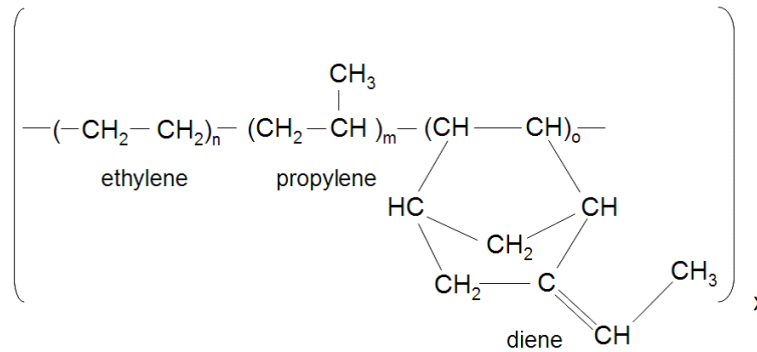


Figure 1.1: The molecular structure of the EPDM.

The EPDMs are produced by solution polymerization using Ziegler-Natta type catalysts. That way, a moderate wide molecular mass distribution and a fairly regular molecular structure can be created [14]. Recently EPDM rubbers of improved properties are produced by metallocen catalysis as well. The EPDMs have a fair tensile strength over a wide hardness range and excellent resistance to ozone, weathering and chemical attack. Furthermore EPDM rubbers exhibit very good electrical insulation properties. Sulphure cured EPDM compounds have high compression set and are less resistant to high temperatures than peroxide cured ones.

The EPDM vulcanizates are resistant to:

- Non-mineral oil based brake fluids
- Hot water and steam
- Silicon oils and greases
- Aqueous solutions of inorganic acids, alkalis and salts
- Alkalis, glycols, ketones...

The EPDM vulcanizates are not resistant to:

- Mineral oil-based fluids and greases
- Synthetic hydrocarbon lubricants
- Organic ester-based lubricants
- Hydrocarbon fuels.

EPDM rubbers are used for a wide range of products, including sealings, o-rings, gaskets, window and door seals, wire and cable insulations, roller covers, conveyor belts, hoses and water proofing sheets.[11]

2.2 INGREDIENTS

There are many kinds of ingredients that differ in their task. The aim of the ingredients is to optimize the properties of the compound. The amount of ingredients in the compound is given by the phr unit (number of parts per hundred pieces of rubber)

2.2.1 Vulcanisation

None of the elastomers could have been made useful without proper compounding with various ingredients. Vulcanisation, named after Vulcan -Roman god of fire- is the chemical process of interlinking of rubber molecules to change the predominantly plastic/viscous properties to elastic properties. The vulcanisation system is mainly a collection of additives required to transform the essentially linear polymer molecules into a three dimensional network by the insertion of crosslinks. Though, various curing systems are used for different rubbers, sulphur vulcanisation remains most favoured vulcanisation route for general purpose rubbers.[21]

2.2.2 Vulcanizing agents

Vulcanization is a chemical process for converting rubber or related polymers into more durable materials via the addition of sulfur or other equivalent vulcanizing agent. The function of vulcanizing agent is to modify the polymer by forming crosslinks (bridges) between individual polymer chains; the most common ones are the sulfur type for unsaturated rubber and peroxides for saturated polymers. Uncured natural rubber is sticky, deforms easily when warm, and is brittle when cold. In this state, it is a poor material when a high level of elasticity is required. Vulcanized material is less sticky and has superior mechanical properties.

A vast array of products is made with vulcanized rubber including tires, shoe soles, hoses, and hockey pucks. The main polymers subjected to vulcanization are polyisoprene (natural rubber) and styrene-butadiene rubber, which are used for most passenger tires. Chemicals called accelerators may be added to control the cure rate in the sulfur system; these materials generally are complex organic chemicals containing sulfur and nitrogen atoms. Stearic acid and zinc oxide usually are added to activate these accelerators. Metal oxides are used to cure halogenated polymers such as polychloroprene or chlorosulfonated polyethylene.

2.2.3 Sulphur Vulcanisation of Natural Rubber

Sulphur vulcanisation is usually carried out industrially by heating the mechanically plasticized rubber with sulphur and auxiliary vulcanizing agents such as organic accelerators, ZnO, long chain fatty acids or the zinc soaps of these acids (activators) along with additional ingredients like antioxidants, anti-ozonants, fillers and reinforcing agents such as carbon black. Major effects of vulcanisation on use related properties include increased elastic recovery, tear strength, tensile strength, fatigue life, toughness, low hysteresis and compression set, etc.[21]

2.2.4 Plasticizers

These materials are added to reduce the hardness of the compound and can reduce the viscosity of the uncured compound to facilitate processes such as mixing and extruding. The most common materials are petroleum-based oils, esters, and fatty acids. Critical properties of these materials are their compatibility with the rubber and their viscosity. Failure to obtain sufficient compatibility will cause the plasticizer to diffuse out of the compound. The oils are classified as aromatic, naphthenic, or paraffinic according to their components. Aromatic oils will be more compatible with styrene-butadiene rubber than paraffinic oils, whereas the inverse will be true for butyl rubber. The aromatic oils are dark colored and thus cannot be used where color is critical, as in the white sidewall of a tire. The naphthenic and paraffinic oils can be colorless and are referred to as nonstaining.

2.2.5 Antidegradents

An antidegradant, this group of chemicals is an ingredient in rubber compounds to deter the aging of rubber products. The most important are the antioxidants, which trap free radicals and prevent chain scission and crosslinking. Antiozonants are added to prevent ozone attack on the rubber, which can lead to the formation and growth of cracks. Antiozonants function by diffusion of the material to the surface of the rubber, thereby providing a protective film. Certain antioxidants have this characteristic, and waxes also are used for this purpose. [18]

4.1.1 PROBLEM BACKGROUND

Viscon Rubber Industries, Pune is a well-known name in the field of elastomer filed. Company manufactures extensive range of elastomeric products for the esteemed customers like Tata Motors Ltd., Bajaj Auto Ltd., Mahindra and Mahindra, Endurance Technologies, Varroc Elastomers etc.

Company was facing continuous customer complaints about the failure of EPDM component- Bump stopper. Component is used in rear fork assembly of two wheeler. Customer was expecting life of component minimum 3, 00,000 cycles while failure occurs much earlier due to wear.

Customer complaint frequency was 10 complaints in year 2015, 8 complaints in 2016, 14 complaints in 2017. This was creating very adverse effect on brand name and the free warranty cost was increasing day by day. Also the increasing cost of quality was the main reason to undertake this project.

Thorough analysis was made by quality and design & development team to minimize the issue. Various trials were taken to formulate possible material composition. Process parameters were altered but can't get the expected results.

Analysis was done to improve both durability and wear resistance of rubber component. For this formulation history, testing reports, failure history, customer complaints record, research papers and literature were analyzed. [2, 3, 6, 14, 16, 18]

Although other filler material like silica was available but research by Ramin zafarmehrabian et.al shows that the hardness of the samples decreases gradually as the percentage of the silica was increased. The decrease of hardness was mainly because of the decrease in crosslink density in the samples which happens as the active surface of silica in contact with the factors creating crosslink is increased.

Company data and all the reference literature show use of carbon black; Carbon FEF N550 or Carbon HAF N330 separately. We decide to combine both N330 and N550 in the four possible formulations as shown in table 5.1. N-330 is a fine active carbon black. It provides high tensile strength and good wear resistance. While N550 is a Medium-dispersion, medium-active carbon black. It imparts high extrudability, relatively high tear resistance.

4.2 OBJECTIVES OF DISSERTATION

The purpose of this work is to study the effect of carbon black on mechanical properties of EPDM rubber. In this work the optimum combination of carbon black is investigated to enhance wear performance and durability of EPDM rubber.

Following are the objectives of the project work,

- To quantify the improvement in hardness, durability and the wear resistance (WR) of EPDM rubber with different carbon black percentage.
- To understand the probable mechanism that is responsible for the improvement experienced, and
- To confirm whether to use single or combination of different carbon black types to achieve desired properties.

TABLE 5.1 COMPOUND FORMULATIONS USED IN THIS STUDY

INGREDIENT/ PHR	A	B	C	D
EPDM	100	100	100	100
Zinc Oxide	5	5	5	5
Stearic Acid	2	2	2	2
Stearic Acid 6 PPD	1.5	1.5	1.5	1.5
Carbon FEF N550	70	00	30	40
Carbon HAF N330	00	70	60	70
Silica	20	20	20	20
Oil	70	70	70	70
Processing Aid	2	2	2	2
Resin	3	3	3	3
Sulphur	1.2	1.2	1.2	1.2
MBTS	2	2	2	2
TMTD	0.5	0.5	0.5	0.5
ZDBC	2	2	2	2

4.3 METHODOLOGY

The methodology adopted to achieve the objectives is given in the flowchart figure 4.1. The optimum combination carbon black proportion is investigated to enhance wear performance and durability of EPDM rubber. For study EPDM rubber is selected. This carbon black combination will be processes to achieve the different mechanical properties.

The abrasive wear is a complex function of load and speed. Hence these two variables were considered for the study of wear behaviour. Wear test were performed using Plate-on-Roller Wear and Friction monitor. Testing conditions were used as follows,

- Normal Load, F_N : - 25N
- Sliding speed, N =50,75,100,125,150 rpm
- Test Duration, T =900 seconds.

Hardness of specimens was measured by using Shore A method. Tensile tests were carried out on UTM machine .Durability test was carried out on Durability Test rig with the help of actual manufactured component that is bump stop.

5. EXPERIMENTAL METHODS

5.1 MATERIAL PREPERATION

The EPDM rubber specimens for the tests have been prepared indigenously in the premises of Viscon Rubber Pvt.Ltd. Chakan, Pune. It is needless to mention that the actual proportions of various ingredients are trade secret and strictly a 'not disclosed grade. The following table indicates a very close approximation of various chemical constituents of EPDM as prepared in the mixing section of company.

The compounding formulations for the EPDM blend with its various ingredients were mixed in a two roll mill at a friction ratio of 1:2 following standard mixing sequence. Compounding formulations based on changing of the Carbon FEF N550, Carbon HAF N330, and Silica contents are shown in Table 1. Two types of carbon blacks N550 and N330 used. Sulphur was added as curing agent. Accelerators like MBTS, TMTD, ZDBC were based on 100 phr of rubber and the samples have the code name A, B, C, D. 6 PPD was used as crack resistance additive.

Vulcanized slabs were prepared by compression molding. Warm up of rubber done by passing it through mill. It gives good bonding on mill. Then curing agents and accelerator were added. Curing was done for cycle time 30 minutes at temperature 90°C.

The material is compressed in the press for approximately 10 minutes. The molding operation has been carried out as per IS: 3400 1977 specification. The extra projection of materials, if any, has been trimmed by scissor after molding to give the specimen proper shape. The dimension, specific gravity and shore hardness values of all the samples have been measured accordingly using appropriate measuring tools and instruments for the respective parameters.

5.2 ESTIMATION OF VULCANISATE PROPERTIES

The vulcanisate properties of the original samples and the revulcanisate properties of the devulcanised samples were determined by different tests and the per cent retention of the properties were estimated. The details of the physical testing are described below.

5.2.1 Hardness Test

Shore hardness, using either the Shore A or Shore D scale, is the preferred method for determining the hardness of rubbers. The Shore A scale is used for 'softer' rubbers while the Shore D scale is used for 'harder' ones. The Shore hardness is measured with an apparatus known as a Durometer and consequently is also known as 'Durometer hardness'. The hardness value is determined by the penetration of the Durometer indenter foot into the sample. The indentation hardness is inversely related to the penetration and is dependent on the elastic modulus and visco-elastic behavior of the material. The hardness of the specimens have been measured as per IS:3400 (Part II) -1980 standard using a Shore (A) Durometer. Indentations have been made at several points on the upper surface of each specimen and the average value has been taken as a measure of hardness. Standard size test buttons (6mm thickness) were tested for hardness.

5.2.2 Tensile strength Test

Tensile Strength is defined as a measure of the ability of a material to withstand a longitudinal stress, expressed as the greatest stress that the material can stand without breaking. Tensile properties are key indicators of the strength of a material. ASTM D412 covers the tensile properties of thermoset rubbers. Stress-strain measurements were carried out using dumbbell shaped specimens. Stress-strain curves represent the response of materials subjected to mechanical load. Stress is the force per unit area from the initial specimen dimensions while strain denotes the ratio of the extension divided by the initial specimen length. Young's modulus is the ratio of stress to strain

Tensile strength was measured with a Microvision testing machine (UTM) at a test speed of 500 mm/min. Dumb-bell shaped test samples were punched out from the moulded sheets along the grain direction. The thickness of the specimen was recorded before testing. The tensile strength, elongation at break, stress at 100%, 200% and 300% of elongation are recorded by software 'Star Testing 2012' software. At least five specimens were tested for each sample and the average value was taken.



Fig. 5.1: Universal Testing Machine

5.2.3 Specific Gravity Test

This property is fully defined by the composition of the material. The specific gravity is the mass per unit volume and is measured by weighing the sample in air and in water.

5.2.4 Hot air aging Test

Hot air aging test was carried out in oven with 100 degree Celsius for 70hours.Both cut samples and actual work piece kept in same oven for processing. Parameters were recorded by software attached to the system.



Fig. 5.2: Hot air aging Test setup

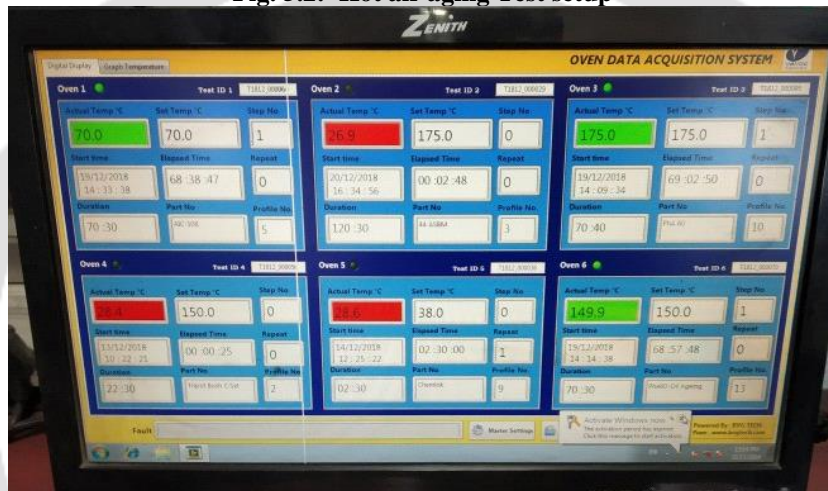


Fig. 5.3: Hot air aging Test screen

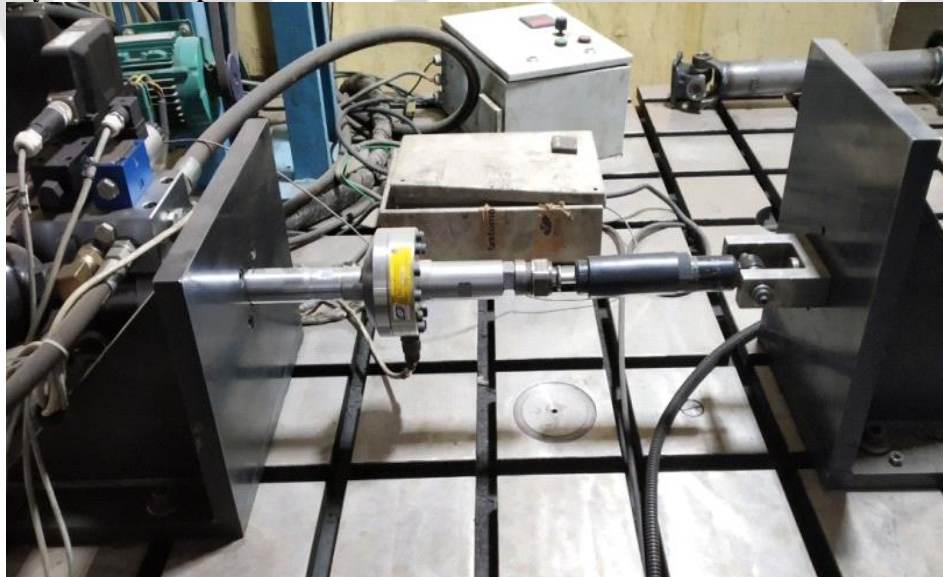
5.2.5 Durability Test for material

Durability test for material was carried out on Flex Testing Machine as shown in figure No. .Machine was Presto India make. Rubber samples were cut as per ASTM D813standard and were tested for life. A crack propagation and total failure of component was observed. Total number of cycles was set 3,00,000 with frequency of 1-3 Hz.



Fig. 5.4: Flex testing machine**Fig. 5.5: Sample in loading condition**

5.2.6 Durability Test for component

**Fig. 5.6: Durability Tester for component**

Durability test was performed on Durability Tester with following test conditions. Temperature was maintained $30\pm 10^{\circ}\text{C}$. The number of cycles was 3,00,000 with frequency of 1-3 Hz. The stroke length was 11 mm. Required specification by customer was,

- There should not crack or breakage of part
- Maximum change in length 10% max.
- Change in load 15% maximum to initial value.

5.2.7 Sample preparation for wear test

The samples have been prepared for the wear test as per the requirement of the sample older of multi tribo tester 'TR- 25'. 20mm × 20mm × 6mm mild steel blocks have been prepared following various machining operations. Blocks of 20mm × 20mm × 2mm EPDM rubber have been cut from the larger sheets of EPDM. The smaller sheets have been pasted on the top of the mild steel blocks using Fevistick adhesive. Thus, samples of size 20mm × 20mm × 8mm have been prepared to be accommodated in the holder of the multi tribo tester. The substrate of each sample is 6mm thick mild steel and the abrading surface is a 2m thick EPDM rubber.

5.2.8 Wear Test

Wear test has been carried out in a multi tribo tester 'TR- 25' of DUCOM, India. Plate-on-Roller configuration has been utilized for this purpose. The rubber surfaces of the samples have been slid against an EN-8 steel roller of diameter 50mm and hardness of 55 HRC. The test parameters have been selected judiciously considering the data base as found in research papers. In the present study, five rotational speeds of the wheel namely 50 rpm, 75 rpm, 100 rpm, 125 rpm and 150 rpm have been selected against a constant load and time of 25 N and 900 sec respectively. The coefficient of friction, wear and mass loss of the samples have been recorded for each test. The samples have been cleaned thoroughly with acetone before and after the tests to remove any trace of dirt, oil or grease, wear debris etc. The initial and final weights have been measured in an electronic analytical and precession balance

The schematic arrangement of the testing procedure has been indicated by line diagram in Figure

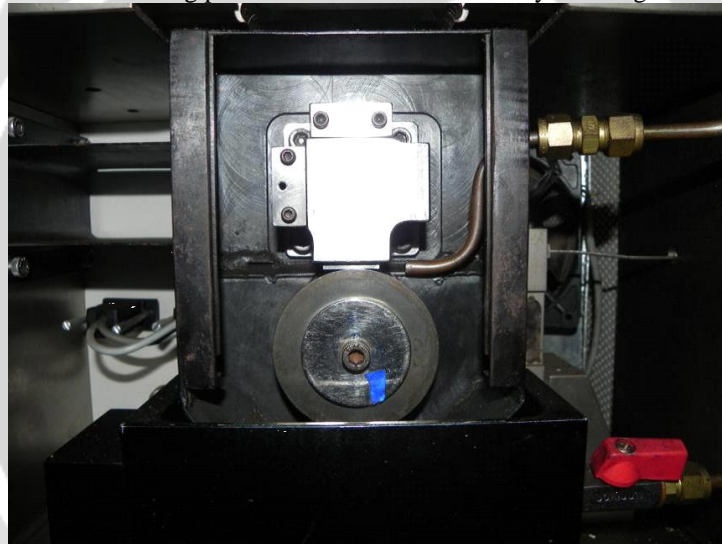


Fig. 5.7: Schematic arrangement and Photograph of experimental set up (Tribometer TR-25)

a) Test Conditions for Wear test

From the work of previous researchers following specification were finalized. Wear test were performed using Plate-on-Roller Wear and Friction monitor. Testing conditions were used as follows,

Normal Load, FN:- 25N

Sliding speed, N=50, 75, 100, 125, 150 rpm

Test Duration, T=900 seconds.

b) Test Conditions for Durability test

As per requirements of customer for final product and parameters as per standard ASTM D813 specimens were prepared and tested. Test conditions were as follows.

Temperature = 30±10°C.

Stroke length = 11 mm

Frequency =1-3 Hz

5.3 TEST DATA

5.3.1 Sample Observation Table

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Following tables shows the sample test data recorded.

Table 5.2. Tensile Strength & % Elongation

Sample	Tensile Strength in Mpa	% elongation
A	10.5	442
B	11.3	407
C	12	382
D	14.3	335

Table 5.3. Hardness in Shore A

Sample	Hardness in Shore A
A	56
B	60
C	67
D	72

Table 5.4 wear Data at 50 Rpm

Time (seconds)	Wear in micron			
	A	B	C	D
180	77.4	78.4	29.4	8.6
360	76.5	77.7	19.4	8.5
540	72.3	73.1	13.1	5.9
720	67.5	68.8	9.8	2.5
900	65.1	65.9	5.6	6.4

Table 5.5 COF Data at 50 Rpm

Time (seconds)	COF OF EPDM SAMPLES			
	A	B	C	D
180	2.66	2.87	2.91	1.85
360	2.45	2.43	2.86	1.65
540	2.33	2.49	2.51	1.58
720	2.27	2.39	2.66	1.54
900	2.19	2.11	2.46	1.56

Table 5.6 Hardness in Shore A

Sample	Hardness in Shore A
A	56
B	60
C	67
D	72

The mass loss data of the samples have also been recorded before and after the tribo testing using a precision balance as mentioned earlier. The weight loss data after 900 sec of sliding have been furnished in Table 2.

Table 5.7 Mass loss Data

Speed(rpm)	Weight loss (gm)			
	A	B	C	D
50	0.004	0.003	0.001	0.003
75	0.006	0.003	0.006	0.003
100	0.006	0.003	0.004	0.002
125	0.007	0.004	0.003	0.002
150	0.007	0.007	0.004	0.002

6. RESULT AND DISCUSSION

6.1 HARDNESS STUDY

Three types of EPDM rubber have been selected for the wear test based on the proportions of carbon black as filler material. Hardness of the material depends on the carbon black content (parts per hundred). The Shore A hardness of the rubbers was determined according to the ISO 868 standard. The hardness was measured by Shore A hardness tester. (BSE, India). Hardness tester with probe 1.25 mm, truncated cone type. Measuring time was 3 seconds and minimum sheet thickness of specimen was kept 6mm.

Graph 6.1 Hardness in Shore A

From graph it is clear that hardness increases with increase in carbon black percentage.

6.2 Mechanical Characterization (Tensile Strength And % Elongation)

The dumb-bell shaped specimens were punched out from a molded sheet by using ASTM Die C. The tests were done by means of a universal tensile testing machine (Hounsfield H10KS) under ambient condition (25 ± 2 °C), following the ASTM D 412-07 and ASTM D 624-07. Tensile strength and elongation at break (%) were measured at room temperature. The initial length of the specimens was 25 mm and the speed of the jaw separation was 500 mm per min. Five samples were tested for each set of conditions, at the same elongation rate. The relative error was below 5%.

From readings it is observed that tensile strength decreased with increase in carbon black while elongation at break increased with increase in carbon black percentage.

Graph 6.2 Tensile strength and % elongation

6.3 Effect of Hot Air Aging

Air aging test was conducted at 100 degree Celsius temperature for 70 hours at laboratory. This test was carried out as per standard procedure. Hot air Aging oven (Bio Technics India) Results shows the increase in percentage elongation and hardness while decrease in tensile strength. Following results were found

TABLE 6.1 Hot Air Aging results

Sample	A	B	C	D
Tensile Strength in Mpa	10.1	11.0	11.8	13.4
% elongation	477	435	400	381
Hardness in Shore A	60	63	80	74

Graph 6.3 Hot Air Aging results (Tensile and Hardness)

7. CONCLUSION & FUTURE SCOPE

7.1 CONCLUSION

The present experimental work has been intended to study the Durability and Abrasive wear characteristics of EPDM rubber of different hardness achieved by changing rubber composition in dry condition using plate on roller type of tribo tester against EN-8 steel counterpart. It is needless to mention that it is very difficult to harmonize the experimental conditions with real life applications and it is also very difficult to arrive at an unique conclusion in case of a viscoelastic material like rubber. In spite of the limitations as mentioned, the following conclusions have been drawn:

1. Hardness of the rubber has been increased with increasing carbon black (N330) content (pph);
2. Coefficient of friction decreases with increase in hardness;
3. Carbon black N550 plays important role in curing kinematics in combination with N330.
3. Wear resistance of EPDM rubber has been improved by the addition of carbon black with proper composition;
4. Stick-slip effect of friction is prominent in case of two smooth sliding surfaces. In the present study, the friction curves reveal strong stick slip behavior.
6. Weight loss due to wear of the surface layer is minimum in case of harder EPDM compared to that of softer one. The weight loss, in general, is not too high in all the cases.
7. Various other works must be carried out in future to understand more clearly the friction and wear mechanism of EPDM rubber.

7.2. FUTURE SCOPE

There is always some future scope for any research work. Some of the future scopes are given as below.

1. In the present experimental work interpretation of carbon black percentage analysis with improved properties like durability, hardness and wear resistance are done but there is scope to study the effect of other parameter like curing temperature, silica filler percentage on the same properties.
2. In the present experimental work wear test is conducted at room temperature, but wear test can be conducted by maintaining actual working temperatures of specific application.
3. In the present experimental work, parameters like contact area, surface roughness of counter surface were kept constant. The effect of parameters like contact area, surface roughness, sliding distance on wear test result can be studied and compared.
4. The wear test carried out here for dry operating condition i.e. without any lubrication which can be done with any type of lubricant. The effect of lubrication on wear rate and wear resistance can be studied and compared.

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