

DEVELOPING OF EPDM COMPOUND HAVING FLAME RESISTANCE

Submitted in partial fulfillment of the requirements for the
award of Bachelor Technology degree in Chemical engineering

By

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**DEPARTMENT OF CHEMICAL ENGINEERING
SCHOOL OF BIO & CHEMICAL ENGINEERING**

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

Accredited with Grade "A" by NAAC

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BONA FIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **SANDEEP P (Reg:39690015)** and **VIGNESH P (Reg:39690021)** who carried out the project entitled "**DEVELOPING OF EPDM COMPOUND HAVING FLAME RESISTANCE**" under our supervision from July 2022 to October 2022

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ABSTRACT

The ethylene–propylene–diene monomer (EPDM) the based compound has been developed to improve the flame resistance by using Expandable Graphite (EG) with different PHR combinations have been explored in this study. The EPDM compound's physical properties, ageing properties, and fire behaviour have been evaluated. The results show that Expandable Graphite (EG) in EPDM rubber has good flame retardant effects and good physical, and mechanical properties like tensile strength, tear strength, heat ageing, compression set, water resistance, low-temperature resistance, ozone resistance, ash content, etc. and also the rate of smoke release of the material decreases significantly. The developed EPDM material has good fire resistance and good mechanical properties.

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CHAPTER 1

1 INTRODUCTION

1.1 POLYMER

Large molecules known as polymers are composed of connected repeating chains or rings of monomeric building blocks. Higher melting and boiling points are typical for polymers. Because there are several molecules in each Polymers typically have higher molecular weights than monomers.

Polymers can be categorized into two groups. Among the natural polymers are silk, keratin, collagen, starch, DNA, cellulose, wool, amber, shellac, DNA, and rubber. Biopolymers provide vital tasks for living things. Synthetic polymer examples include EPDM, polystyrene, synthetic rubber, and PVC (polyvinyl chloride). neoprene, nylon, silicone, and polyethylene. Artificial polymers are employed in create a variety of everyday items, including plastics, paints, adhesives, and mechanical parts.

Two types of synthetic polymers can be distinguished. Plastic thermosets are created from a fluid or squishy material that can be permanently transformed into an insoluble polymer by heating or irradiating it to cure it. Plastic thermosets typically to have a high molecular weight and rigidity. The plastic continues to be deformed. often disintegrate before melting when deformed. Instances of Epoxy, polyester, acrylic resins, polyurethanes, and nylon are examples of thermoset plastics. Thermoset plastics include Bakelite, Kevlar, and vulcanised rubber as well.

The second category is thermoplastic polymers, sometimes known as thermo softening plastics. Artificial polymers. Thermoplastic polymers are flexible in contrast to thermoset plastics. Are malleable and moldable above a particular temperature, but when cool, changes to solid. Temperature. Unlike thermoplastics, which create irreversible chemical connections, the bonding in thermoplastics diminishes with temperature once they are cured. Unlike Thermoplastics melt into a liquid in contrast to

thermosets, which disintegrate instead of melting after heating. Thermoplastic materials include, nylon, Teflon, polyethylene, polypropylene.

1.2 RUBBER

Rubber is an elastomeric substance that has the ability to regain its original shape following deformation. It is produced by the polymerization of isoprene (2-methyl-1,3-butadiene). The bark of rubber trees is used to make natural rubber.

1.2.1 Types of rubber

Based on where they come from, rubbers are divided into two categories: natural rubber and synthetic rubber.

1.2.1.1 Natural rubber

Natural rubber is a polymer that comes from the latex sap of rubber trees. Rubber is a widely used material because of its elasticity. Several products are made from natural rubber that has been vulcanized. The chemical name for natural rubber is polyisoprene. The fact that natural rubber comes from trees, making it renewable and biodegradable, is its finest attribute.

Numerous items, including surgical gloves, medical equipment, tyres for cars and aeroplanes, mattresses, rubber boot bottoms, toys, and more, all use it as its primary component.

1.2.1.2 Synthetic Rubber

Any artificial elastomer is a synthetic elastomer. Unless the synthetic rubber is clearly recognised as a polysulfide rubber, laminates including such a layer will be categorised as extra polymers as synthetic rubber is often formed of additional polymers of polyene monomers. An elastomer is a substance having the mechanical property of being able to bend under tension much more elastically than most substances do while yet regaining its previous size without experiencing permanent deformation. Natural rubber can often be replaced by synthetic rubber, especially when

improved material properties are needed. (Ex: EPDM, CR, NBR, and FKM ext.)

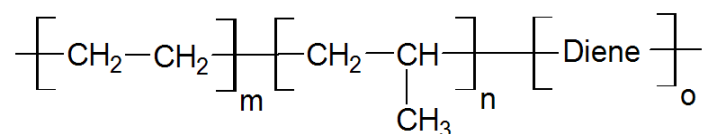
1.3 EPDM (ETHYLENE PROPYLENE DIENE MONOMER)

Ethylene, propylene, and diene monomers are the building blocks of the synthetic rubber known as EPDM (ethylene propylene diene monomer). Its single bond, chemically saturated molecular structure gives it a very strong resistance to the elements. This is due to the fact that, unlike double-bonded rubbers, its molecular structure cannot be disrupted by ozone or UV rays.

The automobile sector is the major market for EPDMs. Weather stripping, window and door seals, O-rings and gaskets, Accumulator bladders, wire and cable connections and insulators, radiator and heater hoses, and diaphragms are a few examples of typical uses.

Waterproofing and roofing, including bitumen modifications, expansion joints, sealants for parapets and facades, and pool and tank liners are further noteworthy applications.

The typical operating temperature range is between -45°C and +150°C, or -50°F and +300°F, with steam reaching +180°C (+355°F).



1.4 EXPANDABLE GRAPHITE

A well-known intumescent flame retardant ingredient is expandable graphite. Its capacity to expand during heating largely determines how well it performs. Additionally, Li et al. have demonstrated that expandable graphite particle size significantly influences flame retardancy qualities.

Expandable graphites (EG) are successfully employed in foams, gaskets, and flame-retardant coatings as flame-retardant additives. Expandable graphite are less expensive than other flame retardant additives and have a good flame retardancy effect that is characterized by little smoke production and an anti-dripping action.

The physical production of a highly expanded, thermally stable residue is the preliminary basis for the flame retardant effect, which can subsequently be introduced into various polymeric systems.

This effectively slows or prevents the spread of fire by inhibiting thermal heat feedback from the flame to the substrate, oxygen migration to the surface, and mass transfer of low molecular weight breakdown products to the gas phase. Expandable graphite has been shown to be effective as a flame retardant in a number of thermoplastic polymer systems (including PP, PE, PET, PS, and PVC), but its practical application is only found in a few niche industries. Expandable graphite is created by intercalating a blowing agent into the graphene layers.

Additionally, expandable graphite have excellent thermal conductivity characteristics. Expandable graphite behave in a multifunctional way by simultaneously improving thermal conductivity and flame retardancy when added to polymeric moulding materials.

CHAPTER 2

2 LITERATURE SURVEY

2.1 LITERATURE 1

Development of flame retardant rubber compound based on general purpose rubber (Feb 2016: International polymer science and technology 43(2):T15-T18 DOI:10)

- To study the fire behavior of rubber material
- The way of reducing the flammability of rubber compound by introducing fire retardant

2.2 LITERATURE 2

Flame retardancy, Fire behavior and flame retardant mechanism of intumescent flame retardant EPDM containing ammonium polyphosphate / pentaerythritol and Expandable graphite.

- To study of different flame retardant used in EPDM rubber.
- Uses of EPDM rubber in different application.
- Research has been done on the intumescent system employed with flame retardant materials.
- Studied about importance of environmental friendly flame retardant material.

2.3 LITERATURE 3

Study of Elastomeric and plastic Materials .Polymer Science Second edition. (V R Gowariker, N V Viswanathan ,Jayadev Sreedhar) New Age International Publishers (First edition :1986 ,Second edition:2015)

- Brief study on rubber materials and its properties.

(Chapter 14: 334 – 347)

- Brief study on Rheology of Polymeric Material .

(Chapter 14: 334 – 347)

- Study of deformation behavior of polymeric material.

(Chapter 14: 334–347)

- Study on Polymer processing – Compounding

(Chapter -15 : 348-369)

2.4 CONCLUSION OF LITERATURE SURVEY:

From all the above literature we can conclude that EPDM is a synthetic rubber that has good properties and it is a combination of ethylene, propylene, and unsaturated diene .and has better resistance to high temperature, ozone, UV radiation, coolant resistance, etc., and is majorly used in heat resistance convey belt, cable wires, gasket, flore sheet, radiator hose, vacuum hose, bellow and so on. EPDM rubber didn't have flame resistance. EPDM can improve flame resistance by compounding. Decabromodiphenyl oxide and decabromodiphenyl ethane are two flame retardants that have a good flame retardant action on EPDM. where it will increase the EPDM compound's flame resistance.due to toxicity halogenated flame retardants have some effect on the environment .now days the halogen materials are banned .so it is important to have halogen less flame retardants. And there are some halogen-free materials like aluminum hydroxide, expandable graphite, etc.

CHAPTER 3

3. AIM

To Develop a EPDM compound having Flame resistance.

3.1 OBJECTIVE

1.To increase the flame resistance of EPDM material by incorporating expandable graphite

2.The resulting compound should not have any significant changes in the other Physical and mechanical properties

3.To avoid further burning of rubber material while fire accident occurs

4.The EPDM with expandable graphite is mainly developed for Electrical vehicle applications so It should have better flame resistance, Vibration absorption, etc

5.To increase the properties for both static and dynamic applications.

3.2 SCOPE

1. It has good flame resistance and low smoke so it can be used in the electrical vehicle where flame resistance is needed (Mat, bellows, Dampers, gaskets, etc)

2. It used as a flooring material (Roofing)

3. This compound can be used for both static and dynamic application

4. The material has good ozone resistance so it can also be used in the outdoor application and it has Good low and high-temperature resistance

CHAPTER 4

4 EXPERIMENTAL PROCEDURE

4.1 PROCESS OVERVIEW

Initially the master batch will be mixed and, after mooney viscosity and specific gravity will be checked in compound to determine the viscosity and density. The obtained results were appropriate it was taken for final batch mixing. After final batch prepared the compound will be tested in ODR (Oscillating disc Rheometer) to check the rheological properties, After testing final compound is cured in laboratory scale(Test slabs).with cured samples testings' which includes tensile strength, elongation at break ,hardness, Ageing properties, Flame test are tested .and to be better than the base compound. This became the basis for the further work that is done on the project .

Table 4.1 Compound formulation

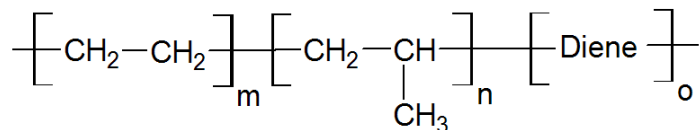
Sl.No	Ingredient	EG With 5 PHR	EG With 10 PHR	EG With 15 PHR
Carbon Master Batch Formulation				
1	EPDM	150.00	150.00	150.00
2	Zinc oxide	5.00	5.00	5.00
3	Stearic Acid	1.50	1.50	1.50
4	TDQ	1.00	1.00	1.00
5	MBI	1.00	1.00	1.00
6	FEF Carbon Black	50.00	50.00	50.00
7	HAF Carbon Black	75.00	75.00	75.00
8	P-25 Oil	40.00	40.00	40.00
9	Polykim –TR Resin	3.00	3.00	3.00
Master Batch Total Weight		326.50	326.50	326.50
Final Batch Formulation				
1	Master Batch	326.50	326.50	326.50
2	Expandable graphite	5.00	10.00	15.00
3	CBS	1.30	1.30	1.30
4	TMTD	0.50	0.50	0.50
5	ZDBC	2.00	2.00	2.00
6	Sulphur	1.00	1.00	1.00
7	PVI	0.30	0.30	0.30
Final Batch Weight		338.60	341.60	346.60

4.2 RAW MATERIALS:

1. Rubber (EPDM)
2. Activator (Zinc oxide, Stearic Acid)
3. Carbon black (FEF,HAF)
4. Process oil (P-25)
5. Resin (Polykim –TR Resin)
6. Retarder (PVI)
7. Flame Retardant (Expandable graphite)
8. Accelerator (CBS,TMTD,ZDBC)
9. Curing agent (Sulphur)

4.2.1 Rubber (EPDM) :

The EPDM synthetic rubber's building components are ethylene, propylene, and diene monomers (ethylene propylene diene monomer). Its single bond, chemically saturated molecular structure gives it a very strong resistance to the elements. This is due to the fact that, unlike double-bonded rubbers, its molecular structure cannot be disrupted by ozone or UV rays.



4.2.2 Carbon black (FEF,HAF)

The commonly used reinforcing agents are furnace blacks and precipitated silica. There are three kinds of carbon blacks available in the market based on the manufacturing process of blacks, They are channel black, thermal black and furnace blacks.

Carbon black improves the following compound properties:

- Mechanical Properties
- Tear strength
- Abrasion resistance
- Cut resistance

Based on measurements of the surface area and structural complexity, carbon blacks are categorised and given a grade number. resulting in decreased permeability, less edoema in challenging settings, and decreased cost.

4.2.3 Process oil

These materials can improve processability and reduce cost without affecting physical properties. Different types of rubber process oils are utilised, depending on the type of rubber and the applications for the finished product. For this compound paraffin oils were chosen as the process oils.

4.2.3.1 Paraffinic oil

Branched chain or straight chain connected hydrocarbon molecules of varying viscosities make up this family of hydrocarbons. The viscosity of the rubber fluid medium grow more viscous as the chain length rises.

4.2.4 Activator

4.2.4.1 Stearic acid

The fatty acid stearic acid has lengthy hydrocarbon chain molecules with a carboxylic acid end group. Stearic acid is primarily employed in the rubber compounding industry's activator system, and specifically in sulfur-based curing systems, in the activator-accelerator network. Stearic acid acquires a dual hydrophobic/hydrophilic character that makes it more rubber soluble. This property

will be highly beneficial in the rubber compounding activator system because it will aid Zinc Oxide in effectively dispersing into the rubber matrix by converting into a rubber-soluble state.

4.2.4.2 Zinc Oxide

Zinc oxide is a white or slightly yellowish powder micro strip that is simple to mix with latex and rubber in natural rubber. It is also a white plastic material used as a colouring and filling ingredient for synthetic rubber. Rubber compound can have strong abrasion resistance, tear resistance, and flexibility after adding active zinc oxide. Apart from activating sulphur cures, ZnO itself acts as cross linking agent for rubbers containing halogen or carboxyl groups, such as chloroprene rubber, bromo butyl rubber (BIIR), carboxylated nitrile rubber (NBR)etc.

4.2.5 Curing agent

4.2.5.1 Sulphur

The first vulcanization agent for unsaturated rubbers still in use today is sulphur. Only in the presence of accelerators and activators do cross-links to rubber macromolecules, which are an effective and quick link, develop. Unsaturated rubbers are first vulcanised with sulphur in three phases. The transitional complex is produced in the first step by the vulcanization system's element interaction. This compound produces an active curing agent when combined with rubber. The principal network of vulcanizate is constructed in the second stage, mostly using polysulfide cross-links. In the third step, rubber macromolecules and modified cross-links (sulphidity decrease) remodel it (isomerization, dehydrogenation, and cyclization). The vulcanizate network reaches its completion.

4.3 MIXING METHODOLOGY

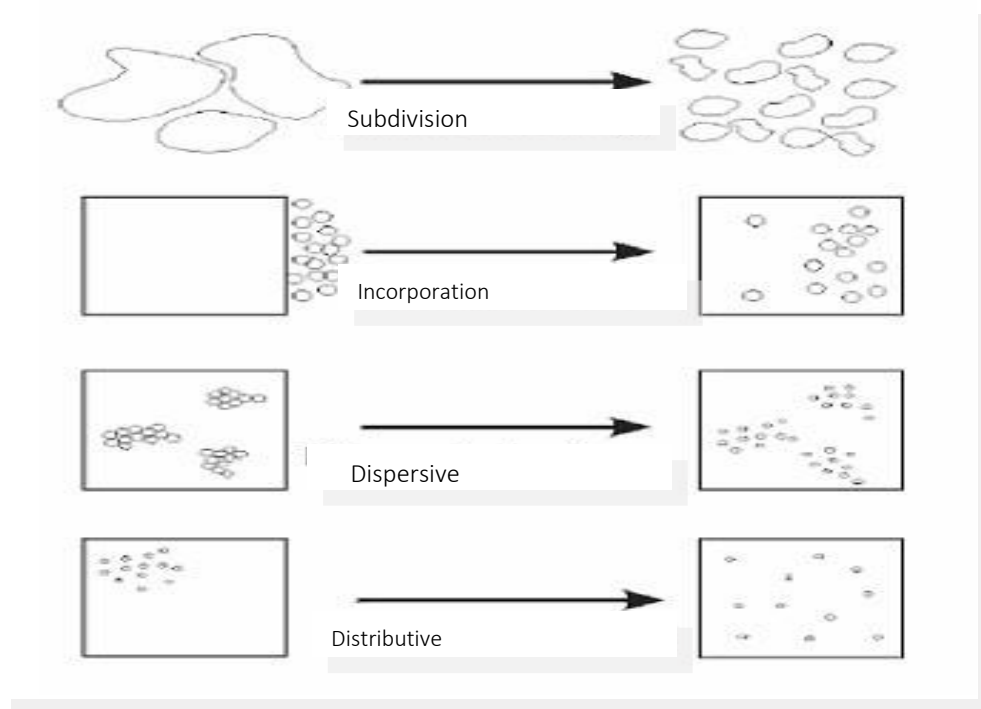


Fig 4.1 Principle of mixing

Four basis steps are involved in the mixing operation:

1. Incorporation: This is the initial stage of mixing, when the previously dispersed elements come together to form a cohesive mass.
2. Dispersion is the procedure used to shrink carbon black agglomerates to their final size.
3. Distribution: is a straightforward homogenization process in which the constituent components are dispersed at random throughout the mass of the mixture.
4. Plasticization: During this process, plasticizers effectively lubricate the mixture as it achieves its final viscosity.

Mixing is a process by which two or more components are converted to a homogenous dispersion. Rubber has to be mixed with compounding ingredients like carbon black, sulphur, accelerators etc.

First we have to calculate the weight of every ingredient

$$\text{Weight of an ingredient} = \frac{\text{Weight of the ingredient in formulation} \times \text{batch weight}}{\text{Total weight of ingredients in formulation}}$$

$$\text{Batch weight} = \text{chamber volume} \times \text{s.g} \times \text{fill factor}$$

Then we have to design the mixing cycle. Mixing mainly done in two stages.

- First stage master batch
- Second stage final batch.

1. Master batch mixing (First stage)

The operation cycle of master batch is as follows : (Open Mill)

- Start Mix
- Add polymer + Chemical
- Add carbon
- Add oil
- Activator
- Compound Discharge

1. Final batch mixing (Second stage)

The operation cycle of final batch is as follows (Open Mill)

- Start Mix
- Add master batch + Chemical
- Compound Discharge



Fig 4.2 Open Rubber Mixer (Two-Roll Mill)

4.4 TESTINGS

4.4.1 Specific gravity

4.4.1.1 Scope:

This procedure applies to the determination of specific gravity of rubber compounds using Densimeter.



Fig 4.3 Densimeter

4.4.1.2 Purpose:

To describe the test method for determining compound specific gravity of rubber compounds.

4.4.1.3 Procedure:

- Place the specimen on the Densimeter scale (tower), which will indicate the weight of the specimen.
- Wait till to get stable mark "O"
- Press "Enter" key to memorise the data in Densimeter.
- Take out the sample with stainless steel Tweezers and dip out in the beaker which containing 50 % diluted IPA for 1 times and shank the sample gently.
- Place the specimen in to the liquid scale (tank) of the

Densimeter and press “Enter” key after it displays the Stable mark “O”.

- Now weight of the sample in water getting memorised in the Densimeter and S.G of the sample displayed.

4.4.2 Rheometer (ASTM D5289)



Fig.4.4 Rheometer

4.4.2.1 Scope

This test method is used to determine the vulcanization characteristics of finalbatch compounds for quality control of mixed rubber stock.

4.4.2.2 Purpose

To define a method for the measurement of selected curing (vulcanization) characteristics of rubber compounds using ODR Rheometer.

4.4.2.3 Procedure

- Switch on the instrument and press the "PLATEN" button, which will close the thermal shield and the platens of the instrument. Once the thermal shield is down and the platens are closed, press the "STANDBY" button, to turn the standby indicator off.

- From the main menu select "Run Rheo Tests" by pressing . Enter the compound code, batch number, date & shift of production in respective fields on the screen.
- A test screen will appear on the monitor. The compound code, test date & the batch number will appear on the top of the test screen. A message "The program will start when the cavity is closed" appears on the bottom of the test screen indicating that the system is ready for data acquisition.
- Once the test cavity temperature reaches and stabilizes at the set temperature a green "Ready" light on the panel illuminates. The platens of the instrument will not close if the temperature is not stabilized. And both die seal condition to be visually verified before loading the specimen.
- Arrange the test specimens on the instrument table. Cover the top & bottom of the specimen with heat resistant film.
- Press the "Platen" button on the ODR panel to open the thermal shield and the platens, place the test sample in the centre of the lower die and close the platens. Make sure that nothing is in the path of the thermal shield or the shield will not close. When the platens are closed a red "Testing" light will illuminate indicating the test is in progress. The prompt on the monitor disappears and a graph of Rheometer cure curve appears on the monitor. The cavity temperature during the test and the current torque values will be displayed on the right side of the cure curve on the monitor.
- After the set time has elapsed, the instrument emits an audible beep signal, indicating the end of the test. The red "Testing" light will go off and "ready" light will illuminate on the panel. An OK message will appear on the right side of the monitor if the test results pass the set limits. A red visual warning will appear for a few seconds on centre of the monitor if the test fails and the parameters that are out of limits will

be displayed in the message. A prompt on the monitor will be displayed to continue with the testing.

- Press the "Platen" button on the ODR panel to open the thermal shield and platens of the instrument. Remove the cured specimen from the test cavity using tweezers and keep aside for specific gravity testing. Place the next specimen in the test cavity and close the platens immediately. Take proper care while loading and removing of test specimens. If the platens kept open for a long time and the dies are exposed to the ambient condition, the temperature of the test cavity may fall and the Ready light will not appear. In such cases put the instrument in standby mode and close the platens until the temperature of the cavity reaches the set point.
- After testing all the specimens put the instrument in standby mode. Press escape button on the computer keyboard to exit the test screen and return to the main menu. From the main menu press F5 - Redraw curves option to open the data recall module. Specify the compound code date and shift of mixing, batch numbers in the respective fields
- Summary report to redraw the test curves of the specified range. The curves will appear on the monitor and the data will be sent to the printer. If a printout of test graphs is required press F1 to send the graph to the printer. After completion of printing press F8 or Escape to return to the main menu. File the printouts in respective compound files.
- Test Control compound daily once to check the correct operation of the instrument. Store control compound in a refrigerator and the storage life must not exceed three months. The test specimens of refrigerated control compound must be allowed to condition to a minimum of 30 minutes at room temperature before testing. Clean the dies thoroughly to remove any compound residue before testing control compound.

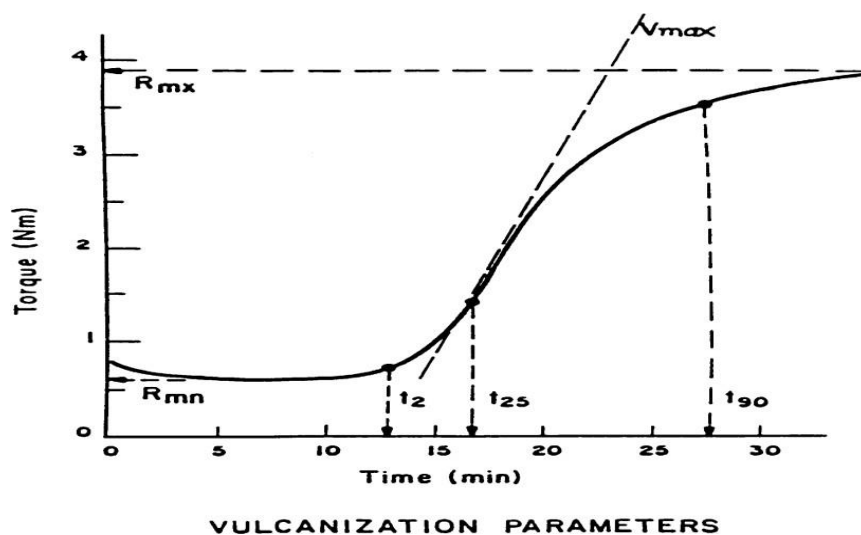


Fig.4.5 Rheological curve

Following parameters shall be reported for the control compound.

1. ML - Minimum Torque
2. MH - Maximum Torque
3. TS2-Time to 2 units rise above ML
4. TC90-Time to 90 % cure

4.4.3 Mooney viscometer (astm d1646)



Fig.4.6 Mooney viscometer

4.4.3.1 Scope

This method applies to the measurement of Mooney Viscosity of raw or compounded Natural Rubbers, Synthetic rubbers & other elastomeric materials. This method is also used for determination of Mooney scorch properties of Vulcanisable rubber compounds.

4.4.3.2 Purpose

To define a method for the measurement of Mooney Viscosity of rubbers & Pre vulcanization characteristics of rubber compounds using Mooney Viscometer.

4.4.3.3 Procedure

- Switch the instrument on by pressing the RED power switch located on the lower right rear side of the instrument frame. All indicators on the front panel of the instrument will light for a few seconds & then go off. After a few seconds, the LED s will illuminate and the "STANDBY" light will be on indicating the machine is on and in standby mode.
- Press the "PLATEN" button, which will close the thermal shield and the platens of the instrument. Once the thermal shield is down and the platens are closed, press the "STANDBY" button, to turn the standby indicator off.
- Press 'Temp' button on the panel of the instrument. The display panel shows the current temperature of the dies. Press 'Set' and using 'Increment + ' or 'Decrement –' keys, set the required temperature. Press 'Enter' to store the temperature value in the memory of the instrument
- Press the 'Viscosity' Button twice. The display shows the current test type. The test type toggles between two settings (viscosity or scorch). Using 'Increment + or 'Decrement – ' buttons select the required test type and press enter.
- Press 'Test time' button, the display shows the current set time. Press 'Set' button. Adjust the time using 'Increment + ' or 'Decrement –' keys until the display reads the required test time. Press Enter to select. For viscosity tests set the time according to above table. For scorch tests set the test time to 25 minutes. For

compounds having scorch time above 25 minutes increase the time accordingly.

- Press 'Configure' and 'Select'. The display shows the output device 'Printer' and output type. 'Scroll through the options to select 'Data points' for Mooney Viscosity and 'Viscosity and Temperature' for Mooney scorch test. When 'Data points' is selected the data points from the test will be printed at the end of a test. When the output option 'Viscosity & Temperature' is selected a real time test curve is produced on the printer. Data points are printed at the end of the plot.
- Check the 'Ready' light is illuminated on the panel of the instrument. Press 'Platens' button to open the platens of the instrument. Insert a brass screwdriver below the rotor and push upward to extract the rotor from the shaft. Insert the stem of the rotor through the hole cut into one of the test specimens. With the test specimen mounted pass the rotor shaft through a piece of cellophane or Mylar sheet and replace the rotor in the lower die. Using the brass screwdriver push the rotor firmly into place. Place the second piece of the test specimen on the top of the rotor and cover with a piece of Mylar. Close the platens of the instrument. A 'Testing' light will illuminate on the panel indicating the test is in progress
- After the set time has elapsed, the instrument emits an audible beep signal, indicating the end of the test. The red "Testing" light will go off and "ready light will illuminate on the MV panel. Results will be printed on the printer.

- Open the platens and remove the sample using the brass screwdriver. If the rotor is kept out of the test cavity or more than 60 seconds put the rotor back and wait for at least 5 minutes until the temperature stabilizes before loading a sample. When idle put the instrument in 'Standby' mode
- Report 'Viscosity' value as Mooney viscosity to the nearest whole value for Raw & compounded Rubber samples. Report T5' Value as Mooney Scorch to the nearest 0.01 minutes for compounded rubber final batches.

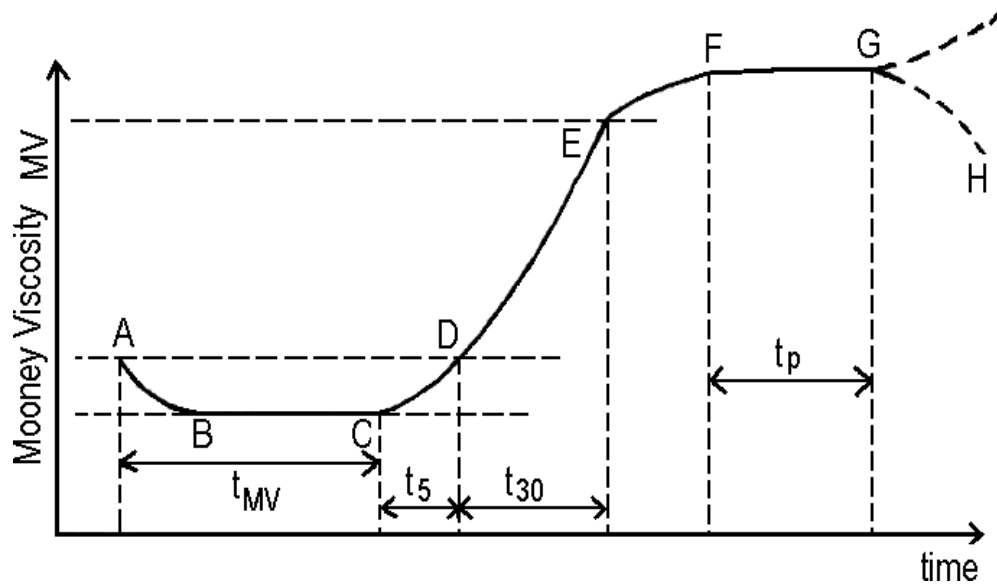


Fig.4.7 Mooney curve

4.4.4 Mechanical property.

4.4.4.1 Tensile properties (ASTM D412)

4.4.4.1.1 Scope

This test method covers procedure used to evaluate the tensile properties of vulcanized rubber.

4.4.4.1.2 Significance and use

- The materials used in this test method For certain applications to be successful, materials must be able to bear tensile forces. These test procedures make it possible to measure these tensile qualities.
- Materials should only be compared when tested under identical conditions because tensile qualities depend on both the material and the test conditions. Temperature and rate of extension should be regulated because they may have a significant impact on tensile characteristics. Depending on the kind of substance being examined, these impacts will change.

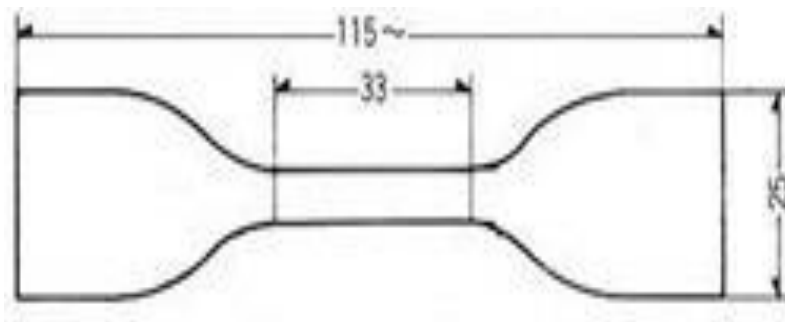


Fig 4.8 tensile test specimen

$$\textit{Tensile strength} = \frac{F}{A}$$

Where,

F- Force required to break the specimen

A- Cross sectional area of the un stretched specimen

It can be expressed in mega pascal (MPa) kilogram force/ square inch or Poundforce/ square inch.

Percentage elongation at break is calculated as follows:

$$\textit{Elongation at break} = \frac{(L - L_0)}{L_0} \times 100$$

Where,

L – Observed distance between the benchmarks on the stretched specimen

L₀ - Original distance between the benchmarks before stretching.



Fig 4.9 UTM

4.4.4.2 Hardness (Shore A) (ASTM D2240)

4.4.4.2.1 Scope

This test procedure is applicable to 12 different Durometer models that assess rubber hardness. A, B, C, D, DO, E, M, O, OO, OOO, OOO-S, and R are the types. how to determine a substance's indentation hardness using vulcanised rubber, elastomeric materials, or TPE.

4.4.4.2.2 Purpose

To describe the procedure for determining the hardness of vulcanisable rubber compounds using hardness button.

4.4.4.2.3 Specimen

The test specimen must have a thickness of at least 6mm. The specimen's lateral dimensions must be adequate to allow measurements to be taken at least 12mm from any edge. It is impossible to determine the hardness of a surface that is spherical, uneven, or rough.



Fig 4.10 Durometer (Shore A)

4.4.4.2.4 Procedure

- Vulcanisable rubber compound in square shape identified with compound code and batch number is put into the cavity of cleaned Hardness button curing mould maintained at 170 +/- 5 Deg.C in button curing hydraulic press. Moulding is done at 170 +/-5 Deg.C for 20 Min. Moulded piece is to be cooled in water to room temperature.
- Set the specimen down on a smooth, hard surface. Apply the tester to the specimen while placing the presser foot parallel to the specimen's surface and holding the tester vertically with the Indentor point at the center of the specimen.
- Apply pressure with hand without shock until the pressure foot just makes a firm contact with the specimen surface. Note the reading from the indicating dial & Report.

4.4.4.3 Tear Strength (ASTM D 624 Die – C)

4.4.4.3.1 Scope

The tear strength of vulcanised rubber material is tested using this approach.

4.4.4.3.2 Test Specimen

A razor - nicked crescent specimen with tab ends and thickness of 2.0 ± 0.2 mm.

4.4.4.3.3 Procedure

- Cut out the test specimen using suitable die (ASTM D 624 DIE - B)
- Carefully cut a single nick using a razor - blade to the nick depth of 0.5 ± 0.05 mm.
- Measure the thickness of the test specimen at three places across the width of the specimen in the region of the test area.
- Carefully align the test specimen with the direction of grip travel before clamping it into the tensile testing machine's grips.
- Record the highest load and the thickness of the specimen.

$$\text{Tear Strength (TR)} = \frac{F}{t}$$

Where

TR = Tear strength in required unit

F = Load in required unit

t = Thickness of the specimen in required unit

4.4.4.4 Heat Ageing Properties (ASTM D 573)

4.4.4.4.1 Scope

To determine the retained physical properties of vulcanized rubber material after aging at the elevated temperature.

4.4.4.4.2 Test Apparatus:

Hot air oven, Tensile Testing Machine, Dumbbell Cutting Die (ASTM D 412 Die-C), Shore-A Hardness meter, Weighing balance

4.4.4.4.3 Test Specimen:

Dumbbell specimen cut from vulcanized test slab of thickness 2.0 ± 0.2 mm.

4.4.4.4.4 Procedure:

- Check the original Hardness and Tensile properties of the specimen
- Ensure the oven temperature for the specified temperature.
- Hang the test specimens in the oven.
- During the aging period the test specimens should not come under contact with each specimen or with the side wall of the oven.
- After completion of the aging period, take out the samples and cool the samples at room temperature for 30 minutes.

4.4.4.4.5 CALCULATIONS

Express the results of the aging tests as a percentage change in physical property

(Tensile Strength, Elongation At Break).

$$P = \frac{A-O}{O} * 100$$

Where P – Percentage of Change in Property

O - Original Value

A – Value after aging.

This results also can be expressed as Co-efficient of retention as: $\frac{A}{O} * 100$

Express the result of hardness as difference between original And aged specimen

Change in Hardness = (Aged specimen Hardness (°A) – Original specimen Hardness (°A).

4.4.4.5 Low temperature brittleness (astm d 2137 method a)

4.4.4.5.1 Scope

This test method is used to determine the lowest temperature at which the rubber vulcanized will not exhibit the brittleness when subjected impact / bending conditions.

4.4.4.5.2 Test Apparatus

Low temperature test chamber

4.4.4.5.3 Test Specimen

Test slab: 2.0 ± 0.2 mm Length : 100 mm

4.4.4.5.4 Procedure

- Switch on the test chamber and set the required test temperature(- 0 to – 55°C)
- Prepare the test specimen as mentioned above.
- Open the door and place the test specimen in to the chamber after the temperature attained to the required °C.
- Close chamber door for the require time period as specified.

- Open the door and immediately bend the specimen in the chamber itself to 180°C for the brittleness verification.
- Take out the sample and close the door.
- Record the observation with respect to the brittleness or cracks.

4.4.4.6 *Flame Resistance (UL-94)*

4.4.4.6.1 *SCOPE :*

This test method is used to measure the flammability of polymeric materials (Rubber Sample)

4.4.4.6.2 *Test Apparatus*

Bunsen Burner, Scale, Cotton, Tray and stand.

4.4.4.6.3 *Test Specimen Preparation*

- Each and every specimen must be sliced from sheet material, cast, or molded using pultrusion, injection moulding, compression moulding, or transfer moulding to get the required shape. Care must be taken to thoroughly clean the surface after any cutting operation, and the cut edges must have a smooth finish. The creation of test specimens must follow current ASTM Practices.
- Standard bar specimens must be at least 3.0 (-0.0 + 0.2) mm thick and must measure 125± 5 mm long by 13.0± 0.5 mm wide. If the minimum thickness is greater than 3.0 mm or the maximum thickness is less than 3.0 mm, the 3.0 mm thick specimens are not required. The thickness cannot be greater than 13 mm. The radius of the corners must not be greater than 1.3 mm, and the edges must be smooth.
- Specimens must be preconditioned for a minimum of 48 hours at 23 ± 2 °C

and 50 ± 10 % relative humidity in accordance with standard

- Specimens for certain tests are to be preconditioned in an air-circulating oven for 168 ± 2 hours at $70^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and then cooled in the desiccator for at least 4 hours at room temperature, prior to testing.
- Within 30 minutes of being taken out of the pre-conditioning environment, specimens must be tested.
- Cotton must be desiccated for at least 24 hours before being used.
- Once removed from the desiccator, the cotton shall be used within 30 minutes.

4.4.4.6.4 Procedure

- Three samples will be examined. On each specimen, two lines perpendicular to the longitudinal axis of the bar must be drawn at intervals of 25 ± 1 mm and 100 ± 1 mm from the lit end.
- Clamp the specimen so that the end that is furthest from the 25 mm mark has its longitudinal axis horizontal and transverse axis 45 degrees angled. The wire gauze must be secured horizontally beneath the sample with 10 mm separating its lowest edge from the gauze and its free end parallel to its edge
- To produce the nominal 50 W test flame, the methane gas supply to the burner must be adjusted to provide a gas flow rate of 105 ± 5 ml/min with a back pressure less than 10 mm water.
- After lighting it, the burner must be moved away from the specimen. Set the burner so that it produces a blue flame that is 20 ± 1 mm high. It is

necessary to regulate the burner's gas supply and air ports to produce a flame with a yellow tip that is around 20 mm in diameter. until the yellow tip disappears, increase airflow. Reevaluate the flame's height and modify as required.

- Light a match and touch the specimen's at the bottom border, a free end.. The longitudinal bottom edge of the specimen should be parallel to the central axis of the burner tube, which should be $45^{\circ} \pm 2^{\circ}$ angled toward the end of the specimen.
- Place the burner in such a way that the flame impinges on the specimen's free end to a depth of 6 ± 1 mm. Remove the burner after 30 ± 1 seconds or when the combustion front of the specimen reaches 25 millimetres while maintaining the test flame's position (if less than 30 seconds). When the combustion front measures 25 mm, start the timing device.
- If the specimen continues to burn after the test flame has been extinguished, note the damaged length, L ($L = 75$), as well as the time it takes the combustion front to move from the 25 mm mark up to the 100 mm mark in seconds. If the combustion front crosses the 25 mm barrier but does not reach the 100 mm threshold, note the passing time in seconds and the damaged length, L. The distance between the combustion front's stopping point and the 25 mm mark, L, is used to compute the damaged length (maximum distance = 75).
- At least three specimens must be tested using the specified protocol. After each specimen, the fume hood in the lab needs to be removed.

4.4.4.6.5 Calculations

For each specimen, use the equation to calculate the linear burning rate, V , in millimetres per minute.

$$V = 60 L/t$$

Where:

V is the linear burning rate in mm/minute

L is the damaged length, in millimeters

t is time, in seconds

4.4.4.7 Ash content

4.4.4.7.1 Purpose

To establish a standard procedure for testing of ash content of a rubber material Which indicates the percentage of inorganic materials (impurities) present in the sample. The impurities present in the sample are known to have deteriorated effect during the service life of the products.

4.4.4.7.2 Apparatus

Silica crucible, weighing machine ,desiccator with desiccant. Muffle furnace.

4.4.4.7.3 Procedure

- Clean the crucible, cool and weigh it. Weigh approx. 1 - 2 gm sample into crucible.
- Transfer crucible into the muffle furnace at the temperature and time and ignite.
- After ignition carefully transfer the crucible containing the ash to the desiccator. For rubber compounds crucible with sample should be kept in the muffle furnace for 1 hour with closed door.

- Cool it half an hour and weigh to the nearest 0.1mg.
- Express the ash content of the sample as a percentage of the mass of test portion.

4.4.4.7.4 Calculation

Weight of the empty crucible = W1

Weight of the crucible with sample = W2

Weight of the crucible with ash = W3

$$\text{Percentage ash content of the sample} = \frac{(W3 - W1) * 100}{(W2 - W1)}$$

4.4.4.8 Compression Set

4.4.4.8.1 Scope

This test method is to determine the permanent set property of a vulcanized rubber under constant deflection in air and for a specified period.

4.4.4.8.2 Test specimen

The standard test specimen shall be cylindrical disc, prepared by direct molding of rubber compound in circular disc mould with the following dimensions. 12.5±0.5 mm in Thickness and 29.0±0.5 mm in Diameter

4.4.4.8.3 Test apparatus:

Compression Set device, Hot air oven, Low force Venire, Dial gauge, Spacer.

4.4.4.8.4 Procedure

- Measure the original thickness (BT) of test specimens to the nearest 0.2 mm.
- To achieve the desired compression, sandwich the specimens between the plates with the necessary spacer.
- The percentage of compression shall be given as 25 %.
- Give sufficient space from the spacers to the specimen for the bulging of the rubber specimen when compressed.
- Tighten the bolts so that the plates are drawn together uniformly until they are in contact with the appropriate spacers. Then without delay place compression set apparatus into the oven, which is operating at the test temperature for the specified period.
- At the end of the specified compressive aging period, loosen the bolts of the compression set device and remove the test specimen and allow cooling at room temperature.
- Measure the test specimen's final thickness (AT) to the nearest 0.2 mm after it has cooled for 30 minutes.

4.4.4.8.5 Calculation

The compression set shall be calculated by following formula:

$$\text{COMPRESSION SET (CS) \%} = \frac{\text{BT} - \text{AT}}{\text{BT} - \text{ST}} * 100$$

Where BT = The original thickness of test specimen,

AT = The final thickness of specimen after recovery,

ST = Spacer thickness.

CHAPTER 5

5 RESULTS AND DISCUSSION

5.1 SPECIFIC GRAVITY

Table 5.1 Specific gravity

Si No	EPDM samples (PHR)	Specific Gravity
1	5 (PHR)	1.128
2	10 (PHR)	1.130
3	15 (PHR)	1.137

The table above has been given to compare the specific gravity values between three different PHR combinations.

5.2 RHEOLOGICAL PROPERTIES

Test Condition: Rheometer 6 min @180°C

Table 5.2 Rheo properties

Si No	EPDM samples (PHR)	Parameter			
		ML lb.in	MH lb.in	TS 2 Min	TC 90 Min
1	5 (PHR)	4.84	23.88	1.25	3.38
2	10 (PHR)	5.24	24.05	1.16	3.13
3	15 (PHR)	5.49	25.13	1.19	3.18

Rheological properties of compounds have better safety time and normal Tc 90 values

5.3 PHYSICAL PROPERTIES

Physical properties are Hardness, Tensile strength, Elongation, Tear strength and Ash content

Table 5.3 Physical properties

SI NO	EPDM Samples (PHR)	Hardness ° A	Tensile strength Kg/cm ²	EAB %	Tear strength Kg/cm	Ash content %
1	5 (PHR)	65	148	522	46	2.88
2	10 (PHR)	65	145	511	44	2.97
3	15 (PHR)	66	138	498	42	4.76

Hardness:

There is no much variation in hardness

Tensile strength and EAB:

Both 5 PHR compound and 10 PHR compound looks same but due to high loading of Expandable graphite (15 PHR) tensile strength and EAB has slightly reduced

Tear strength:

All 5 PHR, 10 PHR, 15 PHR compound's tear strength is better

Ash content:

Comparing of 5 and 10 PHR compound 15 PHR compound has high ash content because high loading weight of flame retardant .

All the three compounds have better physical properties.

5.4 AGEING TESTS

Ageing tests are Compression set, Heat Resistance, Water Resistance, Low Temperature Resistance, and Ozone Resistance

Table 5.4 Compression set

Si No	EPDM samples (PHR)	Compression set at 70° C / 22 Hrs %	Compression set at 100° C / 22 Hrs %
1	5 (PHR)	13.67	22.20
2	10 (PHR)	14.63	23.58
3	15 (PHR)	15.51	24.01

Table 5.5 Heat Resistance

Si no	EPDM samples (PHR)	Heat Resistance 70° C / 70 Hrs			Heat Resistance 100° C / 70 Hrs		
		Change in HS	Change in T/S	Change in EAB	Change in HS	Change in T/S	Change in EAB
1	5 (PHR)	1	-1.39 %	-8.92%	2	-5.69%	-23.6%
2	10 (PHR)	2	-3.57%	-9.89%	3	-2.67%	-21.87%
3	15 (PHR)	2	2.27%	-3.33%	3	4.39%	-19.13%

Table 5.6 Water Resistance

Si no	EPDM samples (PHR)	Water resistance 100° C / 70 Hrs	
		Change in HS	Change in volume
1	5 (PHR)	-1	1.72%
2	10 (PHR)	-1	1.69%
3	15 (PHR)	-1	2.34%

Table 5.7 Low temperature Resistance

Sample	EPDM samples (PHR)	Low temperature resistance(-40°C/5Hrs)
1	5 (PHR)	Pass the test
2	10 (PHR)	Pass the test
3	15 (PHR)	Pass the test

Table 5.8 Ozone Resistance

Si no	EPDM samples (PHR)	Ozone Resistance, 50±5pphm 20% elongation , 40 °C/ 72 hours (D 1149-16 Method B1)		
		Ozone Crack Rating in 24 Hrs	Ozone Crack Rating in 48 Hrs	Ozone Crack Rating in 72 Hrs
1	5 (PHR)	No Crack	No Crack	No Crack
2	10 (PHR)	No Crack	No Crack	No Crack
3	15 (PHR)	No Crack	No Crack	No Crack

Ageing tests of all the three compounds gave good results. Ozone test passes 72 hrs. And there is no crack in -40 °C / 5hrs. So all compounds gives better results in both low temperature and high temperatures.

5.5 Flame Test (UL – 94)

Table 5.9 UL – 94 (Horizontal Burning)

Sample	EPDM (PHR)	Expandable graphite (PHR)	UL 94 (Values)
1	100	0	NR
2	100	5	Linear Burning Rate V = 0
3	100	10	Linear Burning Rate V = 0
4	100	15	Linear Burning Rate V = 0

V = 0 (51 -55) Sec V = 1 and V = 2 (251 -255) Sec

As per UL-94 standard all three compounds meet the specification .comparatively 15 PHR compound has better flame resistance than 5 and 10 PHR compounds.

CHAPTER 6

6 SUMMARY AND CONCLUSIONS

6.1 SUMMARY

- All the three compound has better Rheological properties.
- All the three compound has comparatively good physical properties.
- Developed EPDM compound gives good ageing properties.
- Developed Fire resistance EPDM material passes UL-94 (HB Test)

6.2 CONCLUSIONS

Expandable graphite was used to create flame-resistant EPDM materials, and this process was shown. The EG had an impressive synergistic flame-retardant impact on the EPDM materials. The resultant EPDM materials exhibit good flame retardant properties with the addition of various EG phr combinations and have a V-0 rating in the UL-94 HB combustion test. Meanwhile, the EPDM component has strong physical characteristics, including heat resistance, ozone resistance, and smoke release. Systematically investigating EG The findings demonstrate that EPDM with EG has improved mechanical and physical characteristics, and that EG's complement causes an enlarged and compact carbon layer with a continuous dense microstructure to emerge during combustion. The outcome also demonstrates that EPDM and expandable a greater quality of graphite is its ability to withstand burning

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