PREPARATION AND CHARACTERIZATION OF EPDM NANOCOMPOSITES FOR HIGH VOLTAGE INSULATION

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POWER SYSTEMS

Submitted by

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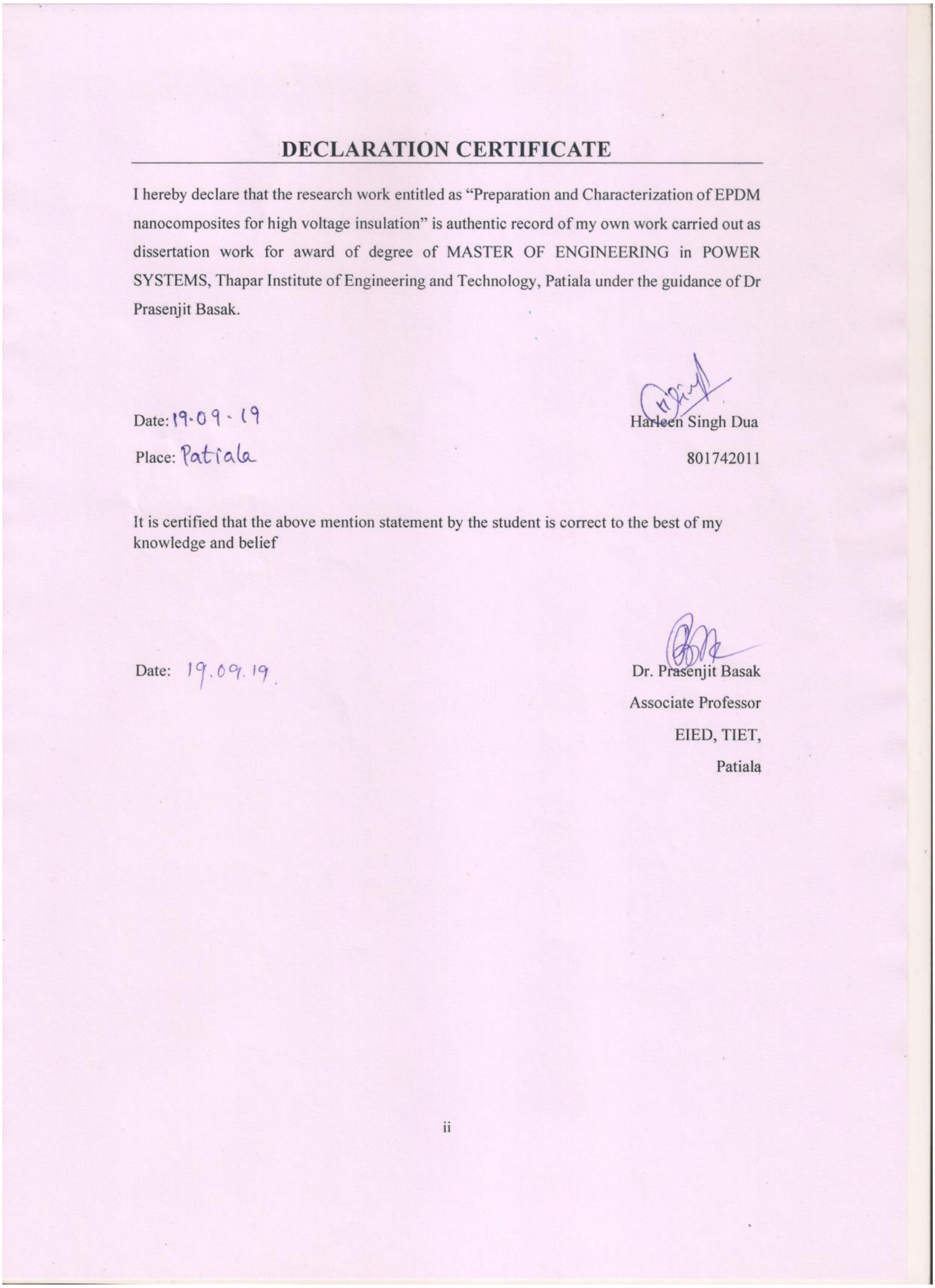

DECLARATION CERTIFICATE

I hereby declare that the research work entitled as “Preparation and Characterization of EPDM nanocomposites for high voltage insulation” is authentic record of my own work carried out as dissertation work for award of degree of MASTER OF ENGINEERING in POWER SYSTEMS, Thapar Institute of Engineering and Technology, Patiala under the guidance of Dr Prasenjit Basak.

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Place: Patiala

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It is certified that the above mention statement by the student is correct to the best of my knowledge and belief

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TABLE OF CONTENT

LIST OF FIGURES

LIST OF TABLES

ABBREVIATIONS

EPDM	Ethylene-Propylene-Diene Monomer
MBT	Mercapto-Benzothiazole
TMTD	Thiuram Disulphide
DOP	Di-octyl Phthalate
PHR	Parts per Hundred Rubber
PD	Partial Discharge
ENB	Ethylidene Norbornene
DCDP	Dicyclopentadiene
HD	1,4-Hexadiene
ASTM	American Society for Testing and Materials
IEC	International Electrotechnical Commission
RPM	Revolutions per minute

ABSTRACT

Polymers, in recent years, have been extensively studied and researched due to their impact on modern technologies because they exhibit improved properties with respect to their counterparts. Addition of nanoparticles, i.e. clay, silica, zeolite and mwcnt produce polymer nano-composites with the interlayering of polymer with the nanoparticle. The compound formed as the polymer nanocomposite has shown improved performance which depends basically on the preparation methods and the fillers used in preparation methods.

In this research, ethylene-propylene-diene monomer (EPDM) pure and with nanocomposites clay, silica, zeolite and mwcnt in the ratio of 1%, 2%, 3%, 4%, 5% respectively are processed in two rolling machines after proper mixing of each component. The prepared sample of EPDM pure and with nanoparticles tested mechanically before and after ageing. The mechanical tests performed on the pure and nanocomposite samples were tensile and elongation test. The mechanical tests exhibit that the samples with nanocomposites outperformed than the pure samples. The mechanical properties of both the samples pure as well as with nanoparticles samples are observed as degraded after the ageing process which clearly founded in this research. The electrical testing of the samples with the partial discharge test has been conducted which shows better breakdown strength of the samples with nanoparticles compared to pure samples.

Keywords: EPDM, Nanocomposites, Tensile, Elongation, Partial discharge, Thiuram Disulphide (TMTD), Mercapto-Benzothiazole (MBT), Di-octyl Phthalate (DOP)

CHAPTER 1

INTRODUCTION

In recent times, the material with a high rate of performance under low economics is in great demand by most of the industries along with various high-level research laboratories. With the current developments in the insulation material, as per the present state of the art, the addition of nanocomposites to the existing polymer layered based materials as EPDM has significantly been highlighted in the field of insulating materials with its enhanced properties, particularly the mechanical, chemical and electrical properties [1]. With increased strength levels along with the low-temperature flexibility in comparison to the other material, the various application of the material includes; automobile sector, electrical wire and cable covers, electrical insulation, household-based applications, roofing membrane etc. along with the various general application and numerous sports goods. In addition to these applications, the material is widely used in high heat and weather resistant based applications.

Nanoparticles used in this research such as clay, silica, zeolite and mwcnt made up of layered structure which is having the thickness of 1nm. The nanoparticles have a weaker bond so they have higher ion exchange capabilities. The mixing of nanoparticles with the pure EPDM and other chemicals is very facile till the nanometric levels due to feeble bonds and high ion exchange [2]. The combination of nanoparticles with the polymer structure is feasible so for the homogeneous interaction of both the nanoparticles and polymer matrix we need to add some binding compounds to produce a homogenised layer structure of nanocomposite of EPDM.

The vulcanisation process we also need to add some compounds to enhance the properties of the nanocomposite produced. To complete the process of favourable interaction we need to use good surfactants that are not only compatible but also help in ion cation exchange process, so to do that we need to research a large number of surfactants. Here for the favourable interaction of nanoparticle with the EPDM layer, we are using dioctyl phthalate (DOP) this compound not only provide better dispersion but provide flexibility to the sample. A small quantity of stearic acid is added in the mixture while combining as it provides multifunctional effects on the properties of the nanocomposite and also helps in its processing also. Steric acid acts as a softener, dispersion agent and helps in preventing the clumping of the compounds in the dispersion process [3]. Sulphur is added in the mixing process as an accelerator that helps in fastening the vulcanisation process of the sample and also helps in reducing the free sulphur amount as it exhibits a deterministic effect on the properties of the sample. Zinc oxide in this process also acts as a vulcanising agent but it also provides strength to the sample and helps in making nanocomposite more durable [4]. This strength helps the sample from not degrading from heat and normal wear and tear as it acts as a guard. Tetramethyl thiuram disulphide (TMTD) and 2-Mercaptobenzothiazole (MBT) both acts as an accelerator in the mixing of the sample as TMTD acts as ultra-fast accelerator while MBT acts as a semi-fast accelerator.

EPDM is basically processed in two ways direct melt bending method and indirect melt bending method [6]. In the indirect melt bending method first, the EPDM is melted and mixed with some chemicals to form the layering and on another side melted mixed compound is again melted and mixed with nanoparticles to produce EPDM nanocomposite. In the direct method, we used to melt mixed all the compounds such as EPDM, nanoparticles and all chemicals in the single step in the two rolling pin machines. The direct method is much simpler and reasonable in price and less time-consuming. While the properties of nanocomposite formed by both methods were similar to each other.

In this research effort has been made to quantify the effect of SILICA, CLAY, MWCNT and ZEOLITE loading in Ethylene-Propylene-Diene-Monomer (EPDM) based high voltage insulation. EPDM was rolled with different nanocomposites in two-roll mills. In this study, EPDM and its nanocomposite including samples preparation have been well discussed along with its performance analysis. Several tests performed over the sample includes tensile strength and elongation-based test for the mechanical properties and partial discharge-based test to analyse the electrical property of the prepared samples.

CHAPTER 2

LITERATURE SURVEY

Nanocomposites are the product formed by the spreading of nanoparticles into the minute ingredients. The materials that are having better performance in terms of mechanical, electrical and chemical properties are in great demand by the industrial sector such as automobile sector for tyre manufacturing, construction sector, household material, agriculture, space sector, sports industries and etc [7].

The industrial, scientific and medical sector in recent years is devoting a lot of energy in the development of the nanocomposite polymer area due to its remarkable properties with respect to pure/virgin polymer. Various nanocomposites are developed in the current era and lots of testing is currently in the process like carbon nanotubes, nano-silica, etc.

Nanoparticles used in this research such as clay, silica, zeolite and mwcnt made up of layered structure which is having the thickness of 1nm. The nanoparticles have a weaker bond so they have higher ion exchange capabilities. The mixing of nanoparticles with the pure EPDM and other chemicals is very facile till the nanometric levels due to feeble bonds and high ion exchange. The combination of nanoparticles with the polymer structure is feasible so for the homogeneous interaction of both the nanoparticles and polymer matrix we need to add some binding compounds to produce a homogenised layer structure of nanocomposite of EPDM [8].

The addition of nanocomposites to the existing polymer layered based materials as EPDM has significantly been highlighted in the field of insulating materials with its enhanced properties, particularly the mechanical, chemical and electrical properties. With increased strength levels along with the low-temperature flexibility in comparison to the other material. To complete the process of favourable interaction we need to use good surfactants that are not only compatible but also help in ion cation exchange process, so to do that we need to research a large number of surfactants. A strong interaction of ions and cations between both the polymer and nanocomposite results due to the dispersion technique [9].

Nanocomposites are used in a broad range of equipment and materials due to their improved properties than the conventional composites. These the advantages of nano-composites with respect to conventional composites [10]:

1. Nanocomposites are light in weight afterwards the addition of nanofillers also.

2. Nanocomposites have enhanced properties whether mechanical, electrical considering the size of nanocomposite in comparison to ordinary composites with very less amount of fillers

In recent time, lots of nanocomposites are prepared in terms of thermoplastic polymers but there is very less attraction towards the rubber nanocomposites. The compound rubber having low strength so with the addition of nanoparticles the product appears in the form of polymer nanocomposite of rubber having better mechanical and electrical properties. Testing on the samples also shows the increase in the tensile strength and tear properties of the nanocomposite so it will be beneficial in the construction sector, household material, agriculture, space sector, sports industries and etc [12].

2.1. Types of Nanocomposites

Generally, nanocomposites are categorised into three types which are based on the material used for their construction. The reinforcement, material, matrix used in the construction of nanocomposites breaks it into the following three categories:

1. Polymer Matrix Nanocomposites
2. Ceramic Matrix Nanocomposites
3. Metal Matrix Nanocomposites

A. Polymer Nanocomposites

These are produced from the material as polymer and reinforcement material used as nano additives exhibits in the category of polymer matrix nanocomposites. In this type of nanocomposites generally nanotubes, clay is the additives. These polymer nano-composites have better mechanical, electrical properties, flame retardancy, wear-resistance and a good amount of strength with a very small quantity of additives makes a lot of attention seeker in areas like industries and academics for both production and research respectively. Simply the polymer nanocomposite is formed by the polymer matrix as thermoplastic and the reinforcement used as carbon and glass fibre [14].

Polymer nanocomposites in comparison to ceramic and metal nanocomposites are very light in weight, having good mechanical and electrical properties, corrosion resistance, easily manufactured, high durability and economical. For achieving the intensified properties of the polymer nanocomposites, the addition of nanoparticles well immerged in the composition so that no scramble occurs.

The polymer itself is the major component of polymer nanocomposite. These are following polymers used for the manufacturing of the polymer nanocomposite.

1. Thermoplastic
2. Elastomers
3. Thermosets
4. Natural polymers

For the manufacturing of polymer nanocomposites, the choice of the polymer depends on the functionality of the application for which it is going to be used such as for mechanical, electrical, magnetic, optical, or chemical stability. Generally, the use of thermoplastic has grown from the current decade due to its better properties and respond to heat [26]. Thermoplastic has low crosslinking as compared to thermosets due to this thermoplastic melt on heating and solidify on cooling. On the other side, thermosets have cross-linkage causing less change in a compound on heat. Thermosets have good mechanical properties and have high chemical resistance and are brittle but thermoplastic getting attention due to its remoulding and recycling properties.

B. Ceramic Matrix Nanocomposites

Ceramic matrix nanocomposites are developing in engineering generation and have large applications in the industrial field. The ceramic nanocomposites have outstanding properties in the electrical and mechanical sector. For the preparation of ceramic nanocomposites generally, a lot of methodologies is present but for the micro composites conventional powder method used and in chemical methods, sol-gel process is up to the mark [15].

C. Metal Matrix Nanocomposites

They require ductile metal or alloy for its formulation. With the use of alloy or metal, these composites will behave differently from there other nanocomposites. It shows better mechanical, chemical and physical properties due to which researchers are investigating its properties. The mechanical properties improved due to its nano level combination of particles.

2.2.Preparation Techniques for Nanocomposites

In the polymer matrix nanocomposites, the process of production of nanocomposite is done by either mechanical or chemical process. For the proper fabrication, one need to make the mixture uniform and homogeneous which is the major problem. So, in the way to make the mixture uniform and homogeneous and also make nanoparticle to mix well while not deteriorating the batch of mixture following four methods are used in research [17]:

1. Interaction
2. Sol-gel
3. In situ Polymerization
4. Direct mixing

A. Interaction Method

In the interaction, method nanoparticles are dispersed into the polymer matrix generally called the incorporation of Nanofillers into polymer matrices which results in the improvement of nanocomposite in terms of shrinkage and flammability. Interaction of nanoparticle with the polymer matrix requires the mixture to be homogenised dispersion. For the homogenised dispersion of nanocomposites following techniques are required.

- Mechanical Technique: In this mechanical technique, interaction is done directly with nanoparticles and polymer by using the mixing of the solution method. Polymer and Nanofillers sheets are dipped and swollen in the co-solvent and solvent solution respectively. After that mixing of both the solutions is practised in order to intercalate the polymer chain to the Nanofillers layer by displacing the solvent asides.
- Chemical Technique: The chemical technique employs the in-situ polymerisation method for preparation. In this first nanoparticles going to intercalate with the monomers or we can say dispersed in this and then polymerization will be going to take place. The nanoparticles are swollen in the solution made of monomer and then polymer will intersect with it and then polymerization occurs [18].

i. Melt Intercalation Method

Melt interaction method produces the nanocomposites by the combination of both Nanoparticle and polymer at the heated temperature. This method is reliable and standard to use in industries. The seasoning of the mixture of Nanofillers and polymer matrix are done under stress or statically. Here the polymers used are incompatible for in situ polymerization method.

ii. Melt Bending Method

In the melt bending technique polymer (powdered form or pellets) is melted into solution in which Nanoparticles are added as fillers. This vicious result of polymer then involves the diffusion due to the increase in temperature. Here the product is made to uniform shape by using compression moulding procedure.

B. In Situ Polymerization

In situ polymerization, the Nanoparticle is socked into monomers then the polymerization is done in monomer solution either using heat or radiation [19]. Nanocomposite formed after the polymerisation of monomer between layers. There is another similar method also called as In Situ template which is very similar to this method. In this process nanoparticle incrusted in synthesised under chains of the polymer. Here both the nanoparticles as well as polymer are put together in the solution called aqueous solution and then this mixture mixed at high temperature. Due to high temperature mixing both the compounds overlap each other forming layers of nanoparticles and polymers together. In this method, some polymer will be decomposed due to the heat caused by the high-temperature dissolving.

C. Sol-gel Method

The sol-gel method is very different and opposite to the approaches that we followed earlier. This method is based on two words that are sol and gel where nanoparticles used are in the form of solid colloidal form whereas monomer used in the form of a gel. In this method, the dispersion of nanoparticle in the form of solid is made with the monomer solution producing interlayering using polymerization reaction. Here the nanocomposite is formed when polymer interconnects with the colloidal nanoparticle forming layers inside and outside the crystal.

D. Direct Mixing Method

This method is used in the fabrication of polymer using both the nanoparticles and monomer in the breaking down process together. This involves following methods for mixing the compounds one includes using solvent and other doesn't use any solvent.

Melt Compounding

In this melt compounding method, we don’t use solvent instead the nanofillers are added and mixed with the polymer at some temperature causing the state from solid to little gluey. Here shear stress is used to mixed the sample batch containing both nanofillers and polymer into nice uniform nanocomposite form [20].

Solvent Method

In the Solvent method, we mix both the nanofillers and polymer in the solvent before nanocomposite is produced. Here nanoparticles use solvent whereas polymer use cosolvent in order to recover from this solvent after the manufacturing of nanocomposite. Here shear stress is also used but very less as compared to melt compounding method.

2.3. Different Structures of Nanocomposites

The dispersion of some weight percentage of nanoparticle with the polymer results into the increased surface area of interconnected polymer with layered nanoparticles. This increase in the surface area is large in comparison to conventional polymer composites. With the interconnection of polymer with the layered nanoparticle results into following four types of polymer/ nanoparticle layered nanocomposites [21].

A. Conventional composites

The composite having large fillers such as in microns as represented in Figure 2.1(a) are categorised in conventional composites.

B. Intercalated nanocomposites

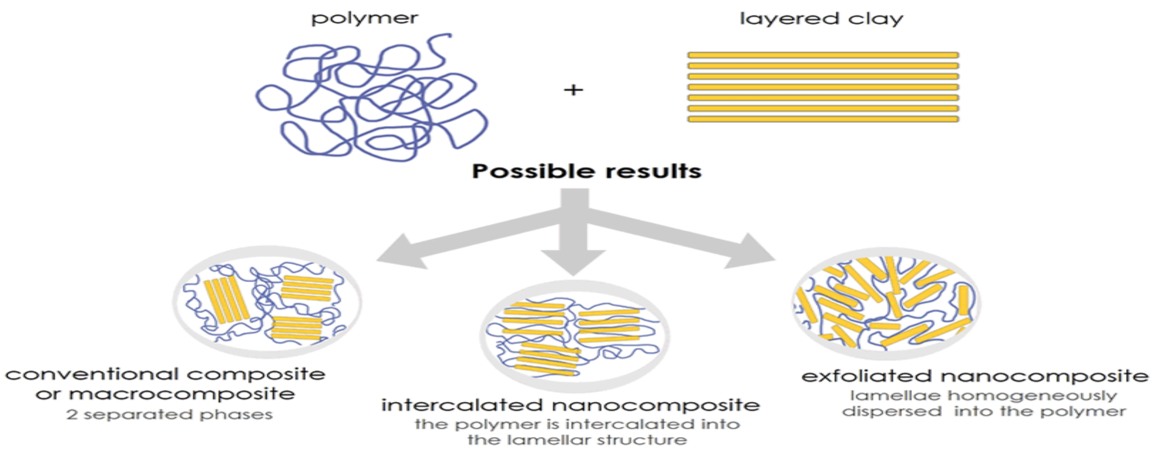
This kind of intercalated nanocomposites layers formed with the interaction of the nanoparticle layer with the rubber chain as represented in Figure 2.1(b).

C. Exfoliated nanocomposites

In the exfoliated nanocomposites the nanoparticle and rubber are totally dispersed so that the structure of the nanoparticle layer will completely loose as to be represented in Figure 2.1(c).

D. Intermediate nanocomposite

The nanocomposite formed which are in between intercalated nanocomposite and exfoliated nanocomposites are termed as intermediate nanocomposite. These are partially intercalated and partially exfoliated.



(a) (b) (c)

Figure 2.1 Structure of different nanocomposites

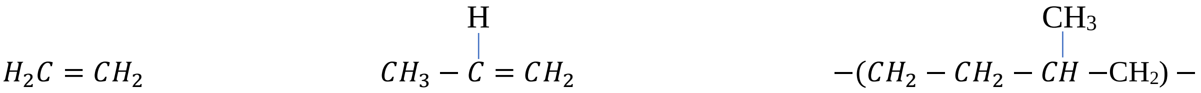
2.4. Ethylene Propylene Diene Rubber (EPDM)

Ethylene propylene diene rubber has widely used the material in both industrial as well as research work also. Its applications are automobile tyres, belts, wires, cables, insulating materials, medical equipment, rubber roofing, heat and weather resistance products [22].

EPDM now has larger space in industrial work due to its mechanical, thermal, electrical properties but it started long back in united states. After a long research on its durability, chemical resistance, heat and weather resistance it commercially produced in 1963.

In the label EPDM, E stands for ethylene, P for propylene, D for diene and M stands for polymethylene (monomer). So, EPDM is the copolymer of ethylene, propylene, diene, monomer.

Structures of ethylene, propylene and copolymer of ethylene and propylene are represented in Figure 2.



$H_2C = CH_2$

$CH_3 - \overset{\overset{H}{|}}{C} = CH_2$

$-(CH_2 - CH_2 - \overset{\overset{CH_3}{|}}{CH} - CH_2) -$

(a) (b) (c)

Figure 2.2 Formation of (a) ethylene, (b) propylene, (c) copolymer of ethylene and propylene (Source: Cicek KARŞAL, 2008)

The main purpose for the extensive use of EPDM is also the cost of vulcanisation on the sample is much lesser by using sulphur compared to the saturated nature of EPM that uses peroxides to cure which is not easy to handle and are really costly.

2.5. Properties of Ethylene Propylene Diene Rubber (EPDM)

The main properties of the EPDM are described as:

1. Thermal resistance helps EPDM withstand high temperature [24].
2. Excellent insulation properties help use in the manufacturing of electric mats that provides better insulation.
3. Chemical resistance makes EPDM use in laboratories and medical equipment.
4. Less wear and tear in the long duration of uses make EPDM as ageing resistance
5. Ozone resistance helps EPDM use in the outer atmosphere.
6. Flexible at low temperature make great demand for wires and cables.

2.6. Composition of Ethylene and Propylene

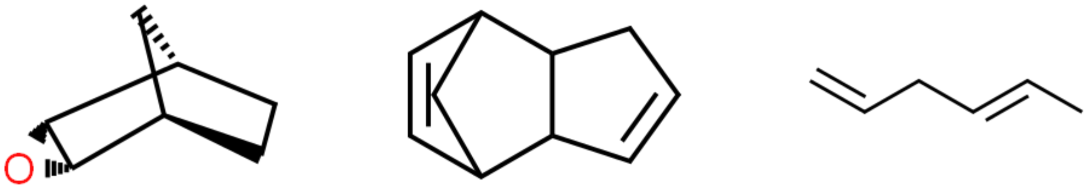
In the composition of ethylene and propylene, the major portion goes to the ethylene but not the propylene. So, the portion or content of ethylene in the monomer is randomly 40 to 80 per cent which shows larger than propylene. Due to this increase in the ethylene content more than propylene causes the structure of the monomer crystalline or hardened in the cold temperatures so in order to protect the batch of rubbers products from dispersion, we need to place them in the warm atmosphere.

With the addition of ethylene into the monomer sample, it distinguished the sample into two categories. The sample containing the percentage of ethylene content above 60 per cent falls into the semi-crystalline form of sample and the sample containing the content lesser than the 60 per cent will fall into the category of amorphous form of sample. The sample which falls in semi-crystalline form has some properties that are used according to the applications as the content of ethylene increases in creates more hardness in the batch of material which helps in better tensile strength and modulus strength of the rubber. Due to its crystalline, it exhibits hardness in the sample at lower temperatures causing difficulty in industrial milling work. Addition of oils and some micro/ nanofillers help to execute better properties of the semi-crystalline sample. The amorphous sample having lower ethylene content tends to have lower hardness due to crystallinity helps in more flexible rubber and it can be used in a colder area where we need more flexibility in rubber [25].

2.7. Diene Content

In the EPDM for the crosslinking of the sulphur for the vulcanisation process, we need to add some unsaturated monomer that helps in the crosslinking of the EPDM with sulphur after it sets at the backbone of the EPDM structure. For this diene monomer is added to the batch of the sample.

Generally, three types of dienes are used in this process that is ethylidene norbornene (ENB), dicyclopentadiene (DCDP), 1,4-hexadiene (HD) [26]. The most commonly used form all three is ENB. The chemical structures are represented in Figure 2.3.



(a) (b) (c)

Figure 2.3 Chemical structure of (a) ethylidene norbornene (ENB), (b) dicyclopentadiene (DCDP), (c) 1,4-hexadiene (HD) (Source: chemical spider)

A. Ethylidene Norbornene (ENB)

ENB tends to be the perfect diene monomer that we use widely in industrial products due to its fastest cure rates. This monomer helps in complete binding of the sulphur cure lines with the EPDM rubber. This not only produces the linear and bounded polymer structure for various applications but helps in double bounding also. These structural changes help in the proper lining of sulphur and create better vulcanisation. The main disadvantage is the expensiveness of this monomer but due to its applications, it is widely used in respect to all others diene monomers.

B. Dicyclopentadiene (DCDP)

DCDP is the diene monomer that exhibits the slowest cure rate respective of all other monomers of the diene. This is also the least expensive diene monomer we have till today and it is easily available. This tends to produce double-bound as ENB after polymerization.

C. 1,4-hexadiene (HD):

The polymer containing the diene of HD has a slower cure rate than ENB but it has slightly better cure rate than DCDP. The monomer produce is linear in nature and has better crosslinking and it also tends to have better thermal properties than all the other monomers of diene present. For the cure line, we need to use diene monomer that has good cure rates than others from all of the above ENB has the best cure rate. After the ENB, HD and DCDP have slightly lower cure rate respective of the ENB. The curing rates are between 0.5 per cent to 12 per cent. So, to make curing fast that helps in sulphur lining and thus vulcanisation fast we need to choose the diene monomer with rates nearly above 6 per cent is preferred.

2.8. Crosslinking/Vulcanisation

In the process of crosslinking or vulcanisation generally, two types of materials are used in the process are sulphur and peroxide. This process of crosslinking is basically the chemical process which tends to improve the properties of the rubber. It helps the soft, gluey, weak bounded, plastic-like rubber structure to a strong bond and helps the product in becoming robust. The vigorous properties of rubber incline to appear from the crosslinking or curing or vulcanisation. In this process of vulcanisation, the chains of rubbers are crosslinked by the agents helping in vulcanisation causing the structure to turns from two dimensional to the three-dimensional structure which has stronger structure. The crosslinking three-dimensional structure is shown in Figure 2.4.

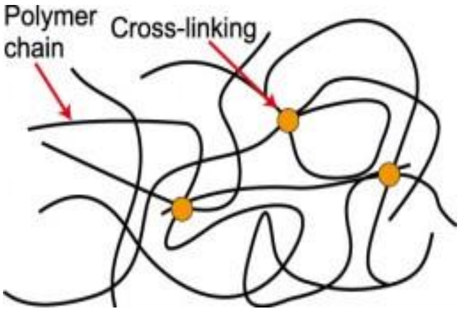


Figure 2.4 Crosslinking on the polymer chain

Before the vulcanisation or crosslinking process is done in rubber, it tends to appear less resistance to heat, water and other environmental aspects. The rubber materials such as jackets, shoes, gloves, wire, cables and other garments products have very less propensity toward heat and light as in the water they swell a lot and it causes damage to the material and in the extensive temperature it rigid or inflexible in lower temperatures and gummy or gluey in the higher temperature range this all due to the poor physical properties of the rubber [27].

Now to overcome this physical deterioration Goodyear’s and Hancock’s heated rubber with sulphur in hundred to eight ratio which helps in restoring the improved physical properties as swelling in water lessen and it tends to withstand extreme temperatures but till now its properties deteriorates after some duration called ageing. So, to improve the ageing properties of the rubber several types of research are done by mixing and heating rubber with numerous compounds. In this research, Goodyear's finds a relation with inorganic oxides so after mixing these got zinc oxide as the compound that changes a lot as it has a small cure time and it helps in binding the sulphur lines making the physical structure strong that helps in better physical properties.

With a lot of success in the field of rubber, many types of research have started in this area not only to intensify the physical strength but also to reduce the curing time also. Onslager in 1906 made the aniline which is the accelerator this is the important discovery in this area. These accelerators help in decreasing the time duration of the vulcanisation process. Aniline is toxic in nature so reaction with disulphide of carbon is made in the process to overcome the toxicity and it produces better accelerator in the industry of rubber. But with drifts, thiocarbaniliide enters the field of accelerators which is less toxic and its reaction with oxide produces much better physical properties in rubber and it also helps in decreasing vulcanisation time and the quantity of sulphur in the rubber.

In 1921, after lots of research work, a compound is discovered named as mercaptobenzothiazole (MBT) it shows much better results and becomes the first on the priority list for the vulcanisation process. After the vulcanisation process if some delinquent will be there so to remove that some fatty acids in form of hydrocarbons rubber are introduced in research that will help in accelerating the vulcanisation process but also covers the deficiency of raw rubber. Their fore addition of stearic acid is practised [28].

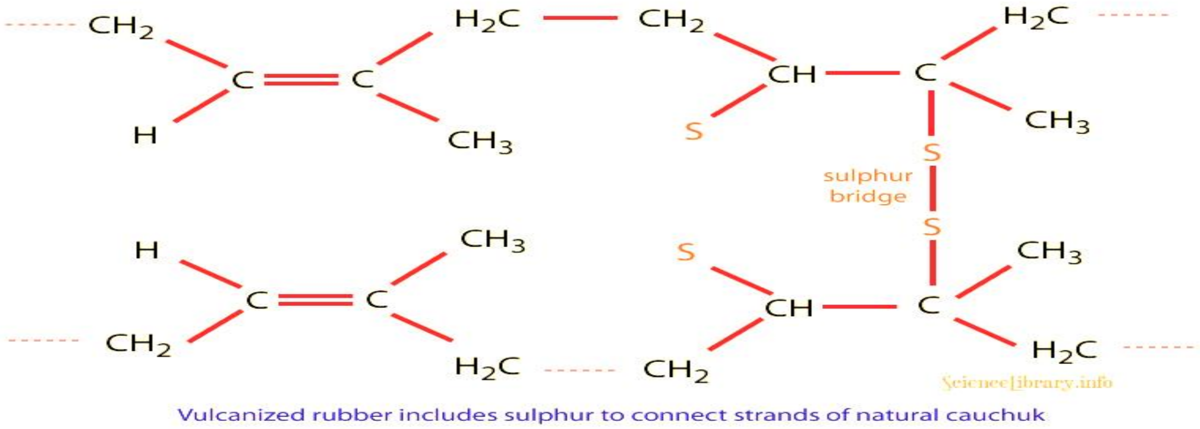
A sample of additives is shown in Table 2.1 which has the curing time of 20 minutes at 140-degree Celsius.

Table 2.1 Extracts of natural rubber

Natural rubber	100 parts
Sulphur	3 parts
Zinc Oxide	5 parts
Stearic Acid	1 part
MBT	1 part

2.8.1. Sulphur Vulcanisation

Sulphur with the help of accelerators and activators is a popular agent required for the vulcanisation process. Sulphur is very economical and easily accessible compound so it is highly recommended to use for such complex reactions. In the chemical reactions, activators help to break the ring of sulphur and with the accelerators fill the intermediate portion with double-bound of sulphur as represented in Figure 2.5. Sulphur agent in this process helps to eliminate the sulphur parts wise or fully. This helps the improvement of rubber in physical resistance, thermal and ageing resistance. In the chemical process, there are two sulphur donors which are used in the vulcanisation process that are tetramethyl thiuram disulphide (TMTD) and dithiodimorpholine (DTDM) [29].



Vulcanized rubber includes sulphur to connect strands of natural cauchuk

Figure 2.5 Structural features of sulphur vulcanised rubber

(Source: www.sciencelibrary.info)

i. Activators

The chemical compound that supports in extracting full latent of accelerators is called activators. They come in both organic as well as an inorganic form of chemicals. In the rubber industries today the most commonly used activators are zinc oxide and stearic acid. The zinc oxide comes from the family of inorganic chemical activator whereas the stearic acid is from the organic family of activators [30].

ii. Accelerators

Accelerators are the chemicals that help in increasing the cure rate from slow to medium to fast. They are categorised in two groups according to their curing time and their scorch delays (time period before vulcanisation). The chemicals that have slow to medium curing time and also produces scorch delay are categorised as a primary accelerator. Chemicals with fast curing time and are scorchy categorised as secondary accelerators [31]. Mainly used accelerators are MBT classified as primary accelerator and TMTD classified as a secondary accelerator. In the industries for rubber manufacturing, they generally use both primary and secondary accelerator in combination to gain fast cures.

2.8.2. Peroxide Vulcanisation

Peroxide vulcanisation is generally used to the saturated type of rubbers. In these types of saturated rubbers crosslinking of sulphur and accelerators is not possible because they contain no reactive group which produces links. In these types of cases peroxide, vulcanisation is preferred. Peroxide helps in forming carbon-carbon chains with adjacent polymer chains using free radicals without disturbing polymer chain. There are three steps to do peroxide vulcanisation in rubber.

(a) Homolytic cleavage
(b) Hydrogen abstraction
(c) Radical coupling

Peroxide is the chemicals that tend to have oxygen bond which cleaved or breaks when exposed to heat due to homolytic cleavage produces molecular fragments called radicals.

$$\mathrm{ROOR} \xrightarrow[\Delta]{} \mathrm{RO}^{*} + {}^{*}\mathrm{OR}$$

The radicals produced in the heat reaction are very active (unsTable). These radicals, when interacted with elastomers such as EPDM, will extract the weakly bonded hydrogen from the chain causing hydrogen abstraction and making them the backbone of rubber.

$$\mathrm{RO}^{*} + \mathrm{P} - \mathrm{H} \longrightarrow \mathrm{ROH} + \mathrm{P}^{*}$$

Due to the instability of elastomer radicals, they are very reactive. So, when there is a combination of elastomer radicals causing the crosslinking in the chains of elastomers. The compound formed as a result is a crosslink.

$$\mathrm{P}^{*} + \mathrm{P}^{*} \longrightarrow \mathrm{P} - \mathrm{P}$$

2.9. EPDM/Layered Nanocomposites Properties

The use of ethylene propylene diene monomer is very widely spread in medical, chemical, automobile industries. All these due to its properties which have a very deep impact on its mechanical or physical properties. Here study is done on the mechanical properties which depend basically on the layered nanoparticle. In the preparation of the polymer composite, there is the interaction of both the polymer and the nanoparticle resulting in the formation of the nanocomposite. This interaction of polymer and nanoparticle is not an easy job as both the compounds have their territory so there exists stress between both of these compounds. So, to improve the interaction between both the compounds and to decrease the stress a coupling agent is applied to the surface of both the polymer and nanoparticle. This will help in reducing the stress and improve the surface energy by softening the surfactant of the elastomer and make improved interaction of both the compounds. Due to this improvement in the nanoparticle, it will provide strength to the EPDM layering making modified EPDM nanocomposite [32].

The changes in the structure and properties of the surface of EPDM nanocomposite will help in improving the nanocomposite but besides all these, there are other several areas that are also be kept in vision [33]. The size of fillers is the most important rule as the size increases the interaction the polymer is not easy due to its slow dispersion and less contact area. So, to avoid this size of filler should be small.

2.9.1. Mechanical Properties of EPDM/ Layered Clay Nanocomposite

In the mechanical test, we use one of the oldest tests to check its physical properties and its mechanical strength that include tensile strength, elongation strength, breaking load and modulus strength tests. Tensile strength is the destructive test in this rubber sample is generally expanded with the force till the sample breaks or destroy in tension this is measured in kg/cm^2. The mechanic characteristics depend on the dispersion of the rubber sample that means it is directly proportional to it [34].

In this research, we use four nanoparticles that are CLAY, SILICA, MWCNT, AND ZEOLITE so the testing is done on all the samples according to its weight percentage to gets its effects on the EPDM rubber sample. The samples are prepared in 6 batches from pure sample without nanoparticle to the sample with the maximum amount of nanoparticle. These six samples are tested respectively in all the mechanical tests that include tensile strength, maximum elongation, breaking strength and modulus strength [35].

After mechanical tests are performed in different tests and then values are written tabulated form then the graphs are formed for the respective tests that include all the samples from before to after ageing.

2.9.2. Electrical Properties of EPDM/ Layered Clay Nanocomposite

The ethylene propylene diene rubber has resistance to moisture and has a lot of flexibility towards temperature range making this a suitable match for the high voltage purposes in various electrical equipment and wiring purpose. To gather that properties partial discharge test to be used. This helps in gathering the breakdown strength and partial discharge of the samples.

2.10. GAPS IN RESEARCH

The work discussed so far includes the testing of EPDM with nanoparticle layered. Following are the gaps or the less explored areas in the field of material compatibility studies and in the present study, these points have been considered.

i. Compatibility of EPDM rubber with various nanoparticles.
ii. Compatibility of nanoparticles have been focused in the literature, and very less literature is available on liquid nanoparticles
iii. Effect of mechanical and electrical tests on the EPDM nanocomposites.

2.11.OBJECTIVES OF STUDY

Based on the gaps in research, the main goal of this project has been said to study the preparation techniques and the effect of mixing of samples, ageing, mechanical and electrical properties. Thus, following objectives have been set for the project:

a) Manufacturing of EPDM and its nanocomposite containing silica, clay, mwcnt and zeolite as nanoparticles for application as high voltage insulator.
b) To apply the best technique for the preparation of the EPDM nanocomposite.
c) To perform tensile and elongation test to investigate mechanical properties of the EPDM nanocomposites.
d) To conduct the ageing test to investigate the physical wear and tear of the sample of EPDM nanocomposite.
e) To perform a Partial discharge test to investigate the insulation breakdown of the EPDM nanocomposite

CHAPTER 3
METHODOLOGY

3.1. Materials

Ethylene propylene diene monomer (EPDM) polymer matrix used to prepare nanocomposite ethylene content 48 wt. %, third monomer content of ethylidene norbornene (ENB) of 9.0 wt. %. Zinc oxide of having specific gravity 5.6 used as an activator for the vulcanisation process. Stearic acid of rubber grade having iodine value = 8 max, acid value = 190 min, SAP value = 192 min. USM = less than 1 % and melting point = 53-55 ^{0}C used as an activator for vulcanisation process. Sulphur was purchased of Four-Square Brand helps in the vulcanisation process. 2-Mercapto-benzothiazole (MBT) acts as the accelerator was supplied in powder form having melting point 170 ^{0}C, specific gravity 1.51, and bulk density between 400-440 kg/m^3. Tetramethyl thiuram disulphide (TMTD) acts as the accelerator with molecular weight 240 g/mol, melting point 140 ^{0}C, specific gravity 1.43, and bulk density between 340-380 kg/m^3. Di-octyl phthalate (DOP) with a specific gravity between 0.980-986, volume resistivity 2 ± 0.2 x 10 ^{11} ohm-cm, Mooney viscosity 82 ± 2 g/10 min, flash point 218 ^{0}C is used as softening agent in this process. Details of materials with its characteristics and suppliers are mentioned in Table 3.1. The structure of the EPDM is shown in Figure 3.1.

Table 3.1 Details of materials used in the manufacturing process

Material	**Supplier**	**Characteristics**
EPDM Trade name Keltan® 6950C	LANXESS India Pvt. Ltd	Mooney viscosity of 65 g/10 min at the test conditions ML (1+4) 125 ^{0}C
Zinc oxide	Upper India smelting & Refinery works	Specific gravity 5.6
Stearic acid	Amrit oils & chemicals	Iodine value = 8 max, acid value = 190 min, SAP value = 192 min. USM = less than 1 % and melting point = 53-55 ^{0}C
Sulphur	Four Square	Melting point 115.2^{0}C

MBT	NOCIL limited	Powder form having melting point 170 ^{0}C, specific gravity 1.51, and bulk density between 400-440 kg/m^3
TMTD	NOCIL limited	Molecular weight 240 g/mol, melting point 140 ^{0}C, specific gravity 1.43, and bulk density between 340-380 kg/m^3
DOP Trade name Kanatol 800	KLJ Group	Specific gravity between 0.980-986, volume resistivity 2 ± 0.2 x 10 ^{11} ohm-cm, Mooney viscosity 82 ± 2 g/10 min, flash point 218 ^{0}C

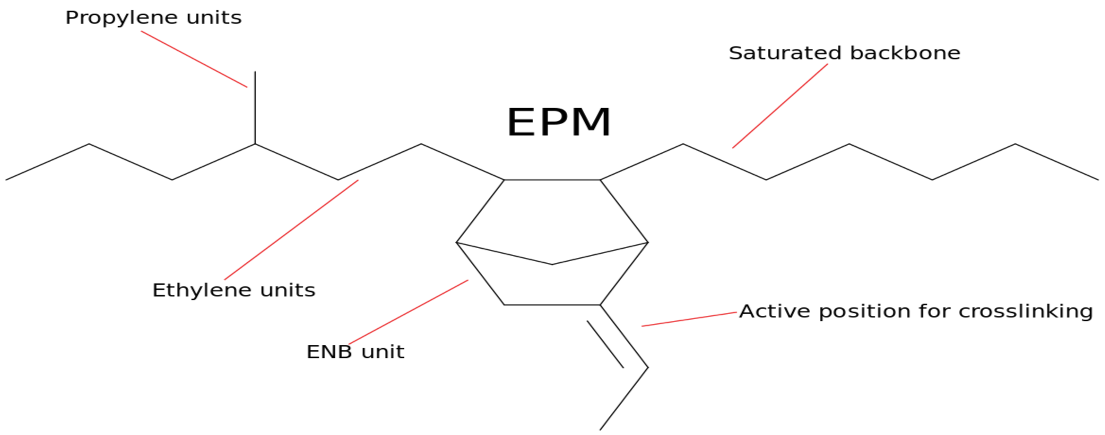


Figure 3.1 Structure of EPDM

(Source: www.sciencelibrary.info)

3.2. Formulation of components

In this study, for the preparation of samples the quantity of rubber was in the ratio of (100 phr) for each sample and respectfully the ratios of other materials were such as sulphur (2 phr) each, zinc oxide (5 phr) each, stearic acid (1 phr) each, mbt (1 phr) each, tmtd (1 phr) each, dop (2 phr) each. The number of nanocomposites was in the ratio of 1 phr. The Nanocomposites used for the study are SILICA, MWCNT, CLAY, ZEOLITE. Table 3.2 (a), (b), (c), (d) represents the formulation of components with different nanoparticles. Model 0-5 represents the weight of the nanoparticle (in phr) used the mixing process. Model 0 represents pure sample without nanoparticle while model 5 represents a sample with the highest amount of nanoparticle.

Table 3.2 (a) Components with SILICA

COMPONENTS	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
EPDM	100	100	100	100	100	100
SULPHUR	2	2	2	2	2	2
Zinc OXIDE	5	5	5	5	5	5
STEARIC ACID	1	1	1	1	1	1
MBT	1	1	1	1	1	1
TMTD	1	1	1	1	1	1
DOP	2	2	2	2	2	2
SILICA	0	1	2	3	4	5

Table 3.2 (b) Components with MWCNT

COMPONENTS	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
EPDM	100	100	100	100	100	100
SULPHUR	2	2	2	2	2	2
Zinc OXIDE	5	5	5	5	5	5
STEARIC ACID	1	1	1	1	1	1
MBT	1	1	1	1	1	1
TMTD	1	1	1	1	1	1
DOP	2	2	2	2	2	2
MWCNT	0	1	2	3	4	5

Table 3.2 (c) Components with CLAY

COMPONENTS	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
EPDM	100	100	100	100	100	100
SULPHUR	2	2	2	2	2	2
Zinc OXIDE	5	5	5	5	5	5
STEARIC ACID	1	1	1	1	1	1
MBT	1	1	1	1	1	1
TMTD	1	1	1	1	1	1
DOP	2	2	2	2	2	2
CLAY	0	1	2	3	4	5

Table 3.2 (d) Components with ZEOLITE

COMPONENTS	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
EPDM	100	100	100	100	100	100
SULPHUR	2	2	2	2	2	2
Zinc OXIDE	5	5	5	5	5	5
STEARIC ACID	1	1	1	1	1	1
MBT	1	1	1	1	1	1
TMTD	1	1	1	1	1	1
DOP	2	2	2	2	2	2
ZEOLITE	0	1	2	3	4	5

3.3. Preparation of EPDM nanocomposite using SILICA, CLAY, MWCNT, TMTD

In the preparation first, all the components are weight carefully. The mixing of components was performed on a two rolling pin machine working on 400 volts at a constant temperature of 40 degrees Celsius. The rubber was first rolled to make smoother to work for 10 minutes then the addition of nanocomposite, stearic acid, dop, zinc oxide was done. The rolling keeps on combining the sample for another 15 minutes and the addition of another batch of samples mbt, tmtd and sulphur was added and rolled the sample till rotor torque becomes constant. The samples after preparation were mould to sheets and buttons on the respective moulding machine at the temperature of 160 degrees Celsius for the time period of 14 minutes. Table 3.2 above represents the formulation of each component.

3.3.1. Weighing of each component

a) The weight of each component measured very precisely as any error will affect the properties of the material.
b) Figure 3.3 represents measuring machine having calibration to four zeros after decimal place for precise measurement.

Figure 3.2 Measuring machine
(Source: Vibracoustic India Pvt. Ltd. Mohali)

3.3.2. Mixing of each component

a) For the mixing process, we use two rolling pins machine as shown in Figure 3.4 this machine works at 400 volts at a temperature of 40-degree Celsius rotor speed of 60 RPMs.
b) First, we put the rubber (300gms) into the machine as rubber is hard at room temperature. We make it smooth by rolling for 10 minutes.
c) In the second step after rubber smoothing, we add our first batch of chemicals that include stearic acid, zinc oxide (3 phr), dop (6 phr), and our nanoparticle (0,1,2,3,4,5 phr) at ambient temperature according to the model of the sample as in Table 3.2.
d) Here dop helps as a binding agent also as it is the only liquid component.

e) After complete binding, we add another batch of chemicals [mbt (3 phr), tmtd (3 phr) acts as accelerators], sulphur (6 phr) as vulcanisation agent.

f) For the mixture to completely binds together we keep on rotating mixture in the two rolling machines at 60 RPMs for another 10 minutes.

Figure 3.3 Two rolling machines

(Source: Vibracoustic India Pvt. Ltd. Mohali)

3.3.3. Making of buttons

a) The buttons are made in the moulds in the moulding machine in Figure 3.5 with the help of the hydraulic press for 20 minutes.
b) Here we have two moulds with twelve button capacity.
c) The temperature of this equipment is 320˚F.
d) The buttons are made in dimensions as (50 mm × 50 mm × 5mm)

3.3.4. Making of sheets

a) The sheet is made in a single mould with the help of the hydraulic press in Figure 3.6.
b) The temperature of equipment is 160˚ C.
c) This machine has the capacity of compression of 5 tons.
d) The sheet is manufactured in (150 mm × 150 mm × 2mm)

Figure 3.4 Button moulding machine

Figure 3.5 Sheet moulding machine

(Source: Vibracoustic India Pvt. Ltd. Mohali)

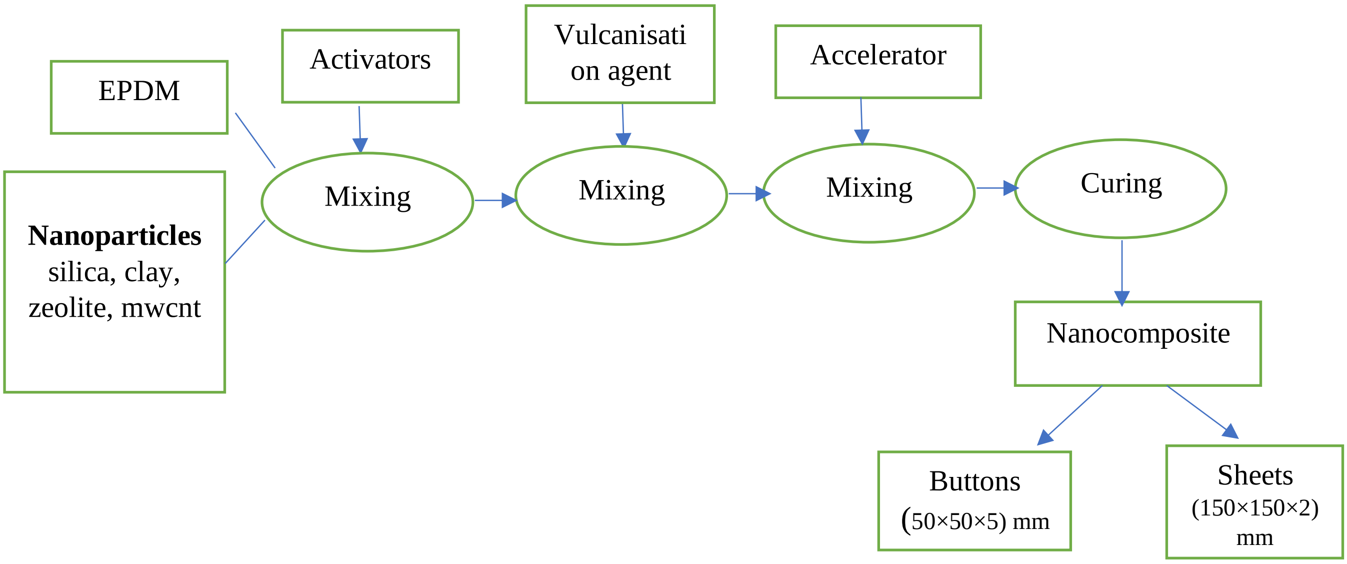


Figure 3.6 Systematic illustration of the production of EPDM nanocomposite

Activators used are stearic acid and zinc oxide, the accelerator is MBT AND TMTD, the vulcanisation agent is sulphur.

3.4. Nanocomposites Testing Techniques

The effect of the addition of layered clay, silica, zeolite and mwcnt to EPDM nanocomposites are investigated through comparison of mechanical and electrical techniques of both pure and layered EPDM samples.

3.4.1. Mechanical Properties Measurement

3.4.1.1 Tensile and Elongation Test

Tensile and elongation tests were determined using the universal testing machine at 25 degrees Celsius at a test speed of 500mm per minute according to DIN 53504. Gage length, standard travel was 25mm at a preload of 0.1 N [36].

a) First, for the test, we need to cut moulded sheets five in number into dumble or dog bone shape as shown in Figure 3.8.
b) These dumbles thickness are measured thrice with the precision of 0.01 mm.
c) Measured dumbles of vulcanised rubbers are inserted into the automatic tensile and elongation test machine as shown in Figure 3.9.
d) From the testing, we measure tensile strength, elongation, modulus and breaking load.

Figure 3.7 Dumble or dog bone shape of EPDM nanocomposite cuttings

(Source: Vibracoustic India Pvt. Ltd. Mohali)

Figure 3.8 Automatic Tensile and Elongation test machine

(Source: Vibracoustic India Pvt. Ltd. Mohali)

3.4.1.2. Standard Test Method for Rubber Vulcanisation Properties

The standard test for rubber properties and vulcanisation characteristics is done using rotor less cure machine MDR-3000 as shown in Figure 3.10 at the temperature of 180 degrees Celsius for the frequency of 1.7 Hz according to ASTM D5289 specifications using 90 degrees die piece [37].

1. In this method, we measure vulcanization characteristics of rubber compound using rotor less shear cure meter.
2. Rotor less cure meter is the class of meter that uses dies to sense torque or stress during strain application.

A. Measurements

a.) Minimum torque: - Here it measures the stiffness of unvulcanised rubber at specified vulcanized temperature, taken at the lowest point in the curve b.) Maximum torque: - Here it measures the stiffness of unvulcanised rubber at specified vulcanized temperature, measured within the specified time.

B. Test methods

1. Here rubber test piece contained in die cavity almost closed and maintained at elevated temperature.
2. The cavity formed by two dies one is oscillating through rotary amplitude.
3. This produces sinusoidal alternating strain in test piece which is recorded.

C. Uses of the test method

1. This helps to identify the vulcanization characteristics of rubber compound.
2. This helps to make quality control in the rubber manufacturing process.
3. In the rotor, less cure meter test sample reaches test temperature in a shorter time and there is better temperature distribution.

Figure 3.9 Standard testing machine

(Source: Vibracoustic India Pvt. Ltd. Mohali)

3.4.1.3. Ageing Test

The ageing test of EPDM nanocomposite sample was carried out in AIR AGEING OVEN at 70 degrees Celsius for the time period of 70 hours. This test ensures the effect of wear and tear of sample and change in its properties due to ageing. After the ageing of sample is done the sample is again tested for mechanical properties as done in the above tests to investigate the effect of ageing due to the environment on its mechanical properties. The standard equipment for the ageing test is shown in Figure 3.11.

Figure 3.10 Standard equipment for ageing test

(Source: Vibracoustic India Pvt. Ltd. Mohali)

3.4.2. Electrical Testing

Partial Discharge (PD), is considered to be a significant phenomenon for accessing insulation performance. The test was conducted as per IEC (International Electrotechnical Commission) Standard 60270, PD is localised electrical discharge that only partially bridges the insulation between conductors [38]. Observation, collections, and evaluations of results of pure and samples with nanoparticles are included.

All the work regarding testing of samples and calculations were performed in the H V Lab, Electrical Engineering, Thapar Institute of Engineering and Technology. A block-shaped pure sample and with nanoparticles (in phr) as shown in formulation Table 3.3 used in the testing procedure.

Table 3.3 List of Samples for PD

Sample	Description
0	100% EPDM
1	100% EPDM + 1% Nanoparticle
2	100% EPDM + 2% Nanoparticle
3	100% EPDM + 3% Nanoparticle
4	100% EPDM + 4% Nanoparticle
5	100% EPDM + 5% Nanoparticle

Measurement and hardware setup of PD shown in Figure 3.12 (a), (b), (c), (d) respectively and the components required with specifications were shown in Table 3.4. The voltage applied was set from 5 kV to 100 KV or till full breakdown occurs. Duration of setup is 30 seconds.

Table 3.4 Components with Specifications for PD

Components	Rating
HV transformer	0.230/5 KV,50 KVA
HV coupling transformer	100 µF
HV measuring capacitor	200/1500 pF
Capacitance of Detector circuit	0.50 µF
Resistance of Detector circuit	60 Ω
Inductance of Detector circuit	0.65 mH

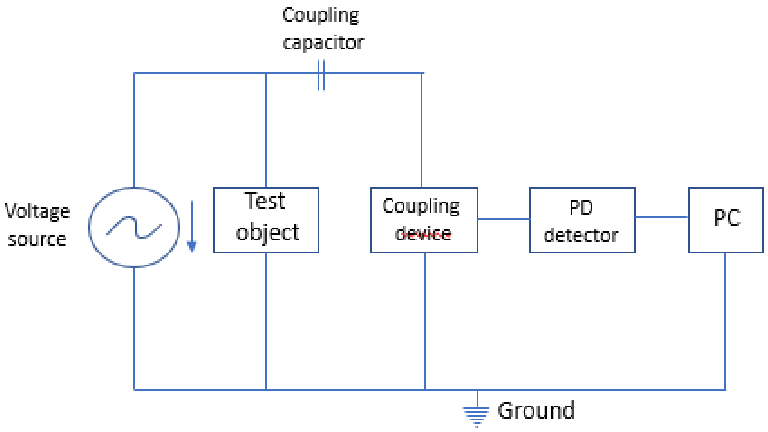


Figure 3.11(a) Block diagram of PD

Figure 3.11 (b) Hardware setup of PD

(At High Voltage Lab, EIED, TIET, Patiala)

HAEFELY TRENCH TETTEX PD-DETECTOR

PD-Scope

Setup: HARLEEN Date: 31.01.19 Time: 11:33

☑ Plot Level > 4.9 nC

☑ Voltage 24.3 kV
50 Hz

PD-Range: 5.0 nC/div
Noise Suppression: 30 %
Synchronisation: Auto
Gain: Auto

Alt-F9 Load New Setup
ESC Quit PD-DETECTOR

PD Trigger ☑

Figure 3.11 (c) Measuring system Figure 3.11 (d) Measured value of PD

(At High Voltage Lab, EIED, TIET, Patiala)

The results obtained from this PD test has been presented in the result section Chapter 4 which are performed on different sections.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1. Mechanical Properties of EPDM Nanocomposite

In the mechanical properties, we measure the effect of the use of different nanoparticle layers in the EPDM nanocomposite (Clay, Silica, Zeolite, MWCNT). In this mechanical testing, we measure the tensile strength, elongation, modulus, and breaking load. We measure all are samples from model 0 to model 5 in which model 0 represents pure EPDM sample without the addition of nanoparticle and the sample representing model 5 have the highest ratio of nanoparticle used in EPDM layering. So, from these models, we can compare both pure EPDM sample with the sample having 1 phr, 2 phr, 3 phr, 4 phr, 5 phr of nanoparticles present inside them. In this we also measure the effect of ageing due to the environment this helps in comparing the after-ageing sample with before ageing sample. The graphs and data are shown to justify each testing.

4.1.1. Tensile Strength

In this research, we measure the tensile strength of EPDM nanocomposite of Clay, Silica, MWCNT, Zeolite before and after ageing.

(a) Tensile strength of EPDM/ CLAY Nanocomposite

In this measurement of tensile strength for EPDM/CLAY nanocomposite Table, 4.1 shows the tensile strength of the samples from model 0 to model 5 before ageing and Table 4.2 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.1 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.1 and Table, 4.2 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The maximum tensile strength for before ageing is 34 kg/cm^2 for maximum filler in model 5 whereas the tensile strength for after ageing sample with maximum filler loading in model 5 is 28.4 kg/cm^2. The sample without the filler of nanoparticle or pure sample have a least tensile strength that is for before ageing of model 0 is 17.8 kg/cm^2 whereas the after ageing tensile strength of model 0 is 16.5 kg/cm^2.

Table 4.1 Tensile strength in kg/cm^2 of CLAY nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	16.5	28.6	28	27.9	30.1	33.7
2	17.8	25.5	31.6	31.2	36.2	34.0
3	15.7	25.4	30.9	29.6	33.2	35.9

Table 4.2 Tensile strength in kg/cm^2 of CLAY nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	17.2	28.6	31.4	23.9	24.7	32.0
2	16.5	24.4	26.8	25.1	34.4	28.4
3	18.1	21.4	25.5	25.5	30.4	28.5

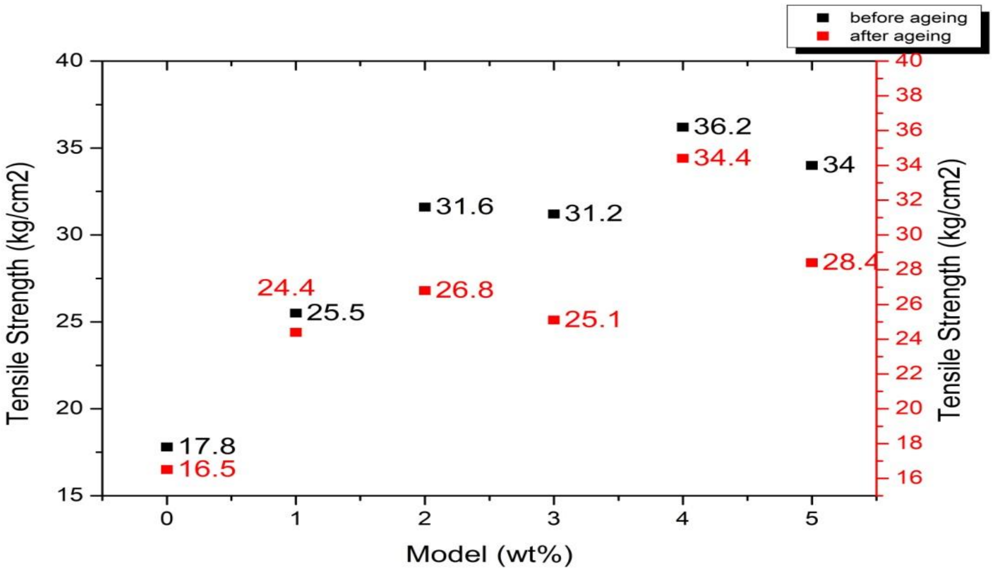


Figure 4.1 Tensile strength of clay nanocomposite for both before and after ageing with different filler loading.

(b) Tensile strength of EPDM/ SILICA Nanocomposite

In this measurement of tensile strength for EPDM/SILICA nanocomposite Table, 4.3 shows the tensile strength of the samples from model 0 to model 5 before ageing and Table 4.4 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.2 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of

Table 4.3 and Table, 4.4 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The maximum tensile strength for before ageing is 40 kg/cm^2 for maximum filler in model 5 whereas the tensile strength for after ageing sample with maximum filler loading in model 5 is 32.3 kg/cm^2. The sample without the filler of nanoparticle or pure sample have a least tensile strength that is for before ageing of model 0 is 14.9 kg/cm^2 whereas the after ageing tensile strength of model 0 is 15.9 kg/cm^2.

Table 4.3 Tensile strength in kg/cm^2 of SILICA nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	14	19.3	19.7	26.2	24.8	31
2	14.9	16.5	17.2	21.7	23.9	40.2
3	14.8	15.8	17.6	22.3	25.7	37.7

Table 4.4 Tensile strength in kg/cm^2 of SILICA nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	16.1	16.7	23.2	24.3	26	34.2
2	15.9	19.1	22.4	21.9	29.1	32.3
3	17.9	18.2	23.5	25.2	30.6	39.8

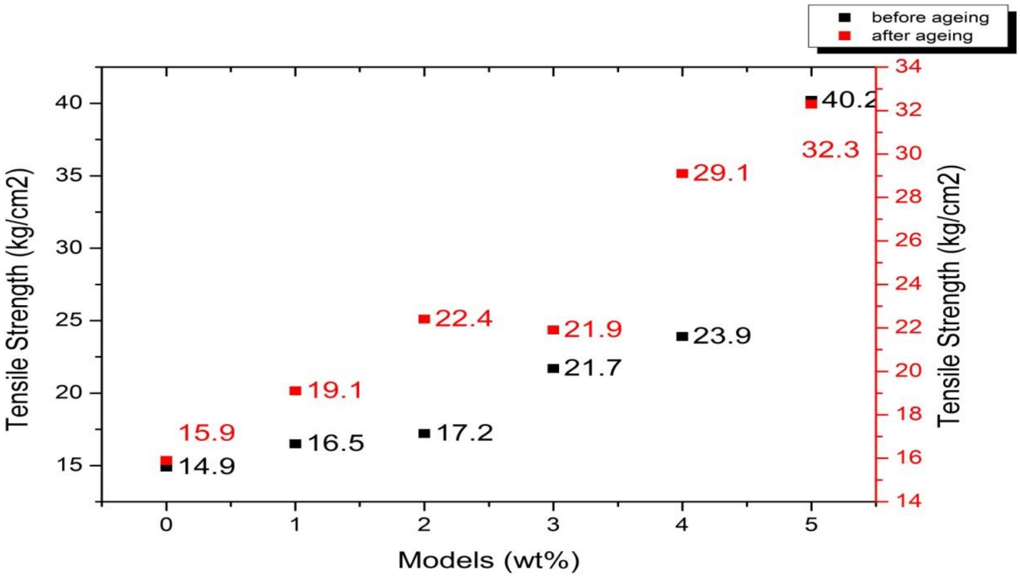


Figure 4.2 Effect of different filler loading on tensile strength of silica nanocomposite for both before and after ageing.

(c) Tensile strength of EPDM/ MWCNT Nanocomposite

In this measurement of tensile strength for EPDM/MWCNT nanocomposite Table, 4.5 shows the tensile strength of the samples from model 0 to model 5 before ageing and Table 4.6 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.3 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.5 and Table, 4.6 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The maximum tensile strength for before ageing is 33.2 kg/cm^2 for maximum filler in model 5 whereas the tensile strength for after ageing sample with maximum filler loading in model 5 is 38 kg/cm^2. The sample without the filler of nanoparticle or pure sample have a least tensile strength that is for before ageing of model 0 is 15.6 kg/cm^2 whereas the after ageing tensile strength of model 0 is 15.8 kg/cm^2.

Table 4.5 Tensile strength in kg/cm^2 of MWCNT nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	16.3	20.9	23.5	25.0	30.7	32.3
2	15.6	19.2	20.7	24.6	32.4	33.2
3	14.7	20.2	20.5	21.5	30.2	34.7

Table 4.6 Tensile strength in kg/cm^2 of MWCNT nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	12.1	22.1	27.1	25.8	31.3	36.6
2	15.8	18.5	22.4	26.8	34.6	38.0
3	16.8	17.6	22.2	28.6	36.3	35.7

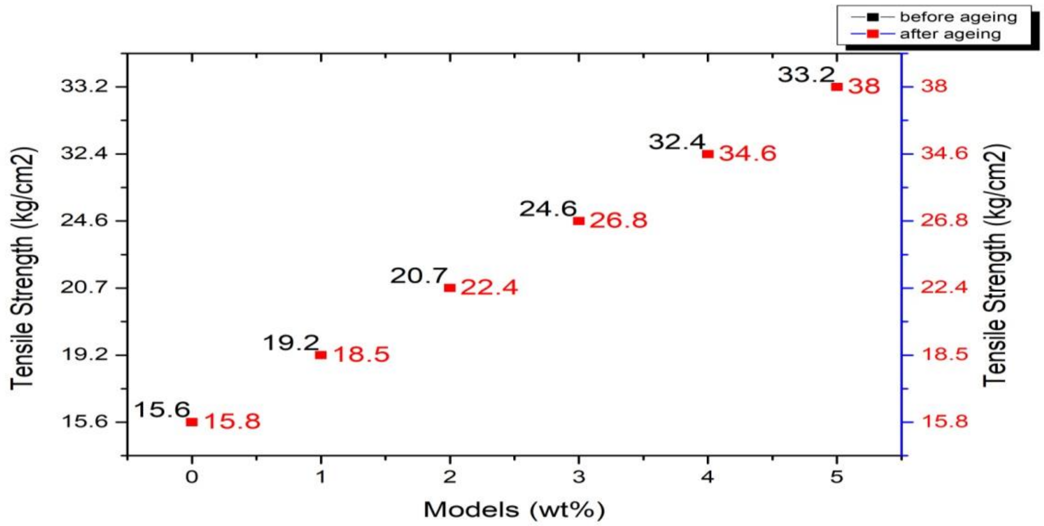


Figure 4.3 Effect of different filler loading on tensile strength of mwcnt nanocomposite for both before and after ageing.

(d) Tensile strength of EPDM/ ZEOLITE Nanocomposite

In this measurement of tensile strength for EPDM/ ZEOLITE nanocomposite Table, 4.7 shows the tensile strength of the samples from model 0 to model 5 before ageing and Table 4.8 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.4 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.7 and Table, 4.8 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The maximum tensile strength for before ageing is 20.4 kg/cm^2 for maximum filler in model 5 whereas the tensile strength for after ageing sample with maximum filler loading in model 5 is 29.5 kg/cm^2. The sample without the filler of nanoparticle or pure sample have a least tensile strength that is for before ageing of model 0 is 16.1 kg/cm^2 whereas the after ageing tensile strength of model 0 is 16.6 kg/cm^2.

Table 4.7 Tensile strength in kg/cm^2 of ZEOLITE nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	14.0	18.9	19.2	22.3	27.1	25.4
2	16.1	18.7	18.1	23.4	25.0	20.4
3	15.8	17.7	17.2	22.0	28.6	22.8

Table 4.8 Tensile strength in kg/cm^2 of ZEOLITE nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	17.3	19.3	20.1	20.7	24.8	29.0
2	16.6	17.7	18.9	19.5	21.0	29.5
3	17.6	17.9	21.0	20.7	22.1	22.3

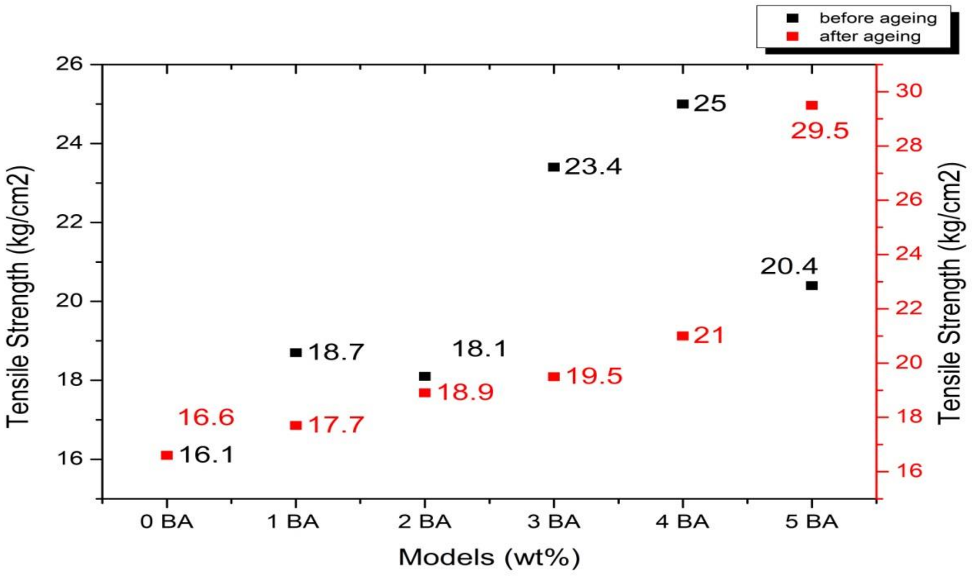


Figure 4.4 Effect of different filler loading on tensile strength of zeolite nanocomposite for both before and after ageing.

From all the Figures 4.1 to Figure 4.4 graphs shows the variation of tensile strength for both before and after ageing. The reinforcing ability of fillers follows the order as for the before ageing as **SILICA>CLAY>MWCNT>ZEOLITE**. The after ageing the order follows as **MWCNT>CLAY>SILICA>ZEOLITE**.

4.1.2. Maximum Elongation

In this research, we measure the max elongation strength of EPDM nanocomposite of Clay, Silica, MWCNT, Zeolite before and after ageing.

(a) Maximum Elongation strength of EPDM/ CLAY Nanocomposite

In this measurement of tensile strength for EPDM/ CLAY nanocomposite Table, 4.9 shows the max elongation strength of the samples from model 0 to model 5 before ageing and Table 4.10 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.5

samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.9 and Table, 4.10 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The maximum max elongation strength for before ageing is 164 % for maximum filler in model 5 whereas the max elongation strength for after ageing sample with maximum filler loading in model 5 is 138%. The sample without the filler of nanoparticle or pure sample have a least max elongation strength that is for before ageing of model 0 is 138 %whereas the after ageing max elongation strength of model 0 is 112%.

Table 4.9 Maximum elongation in % age of CLAY nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	122	170	124	138	138	143
2	138	156	158	157	158	164
3	125	157	144	141	139	154

Table 4.10 Maximum elongation in % age of CLAY nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	116	143	153	116	115	151
2	112	111	172	135	176	138
3	122	97	127	134	135	131

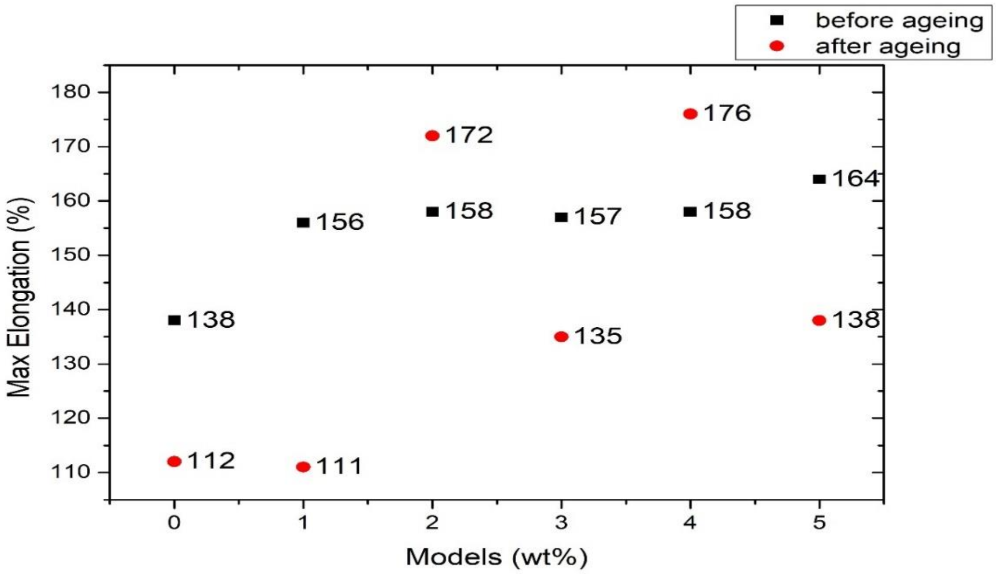


Figure 4.5 Effect of different filler loading on max elongation strength of clay nanocomposite for both before and after ageing.

(b) Maximum Elongation strength of EPDM/ SILICA Nanocomposite

In this measurement of tensile strength for EPDM/ CLAY nanocomposite Table, 4.11 shows the max elongation strength of the samples from model 0 to model 5 before ageing and Table 4.12 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.6 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.11 and Table, 4.12 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The maximum max elongation strength for before ageing is 398 % for maximum filler in model 5 whereas the max elongation strength for after ageing sample with maximum filler loading in model 5 is 283%. The sample without the filler of nanoparticle or pure sample have a least max elongation strength that is for before ageing of model 0 is 114 %whereas the after ageing max elongation strength of model 0 is 122%.

Table 4.11 Maximum elongation in % age of SILICA nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	102	153	167	206	244	287
2	114	128	149	173	240	398
3	112	125	150	173	243	373

Table 4.12 Maximum elongation in % age of SILICA nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	118	115	180	177	245	357
2	122	152	169	186	246	283
3	137	138	182	186	260	367

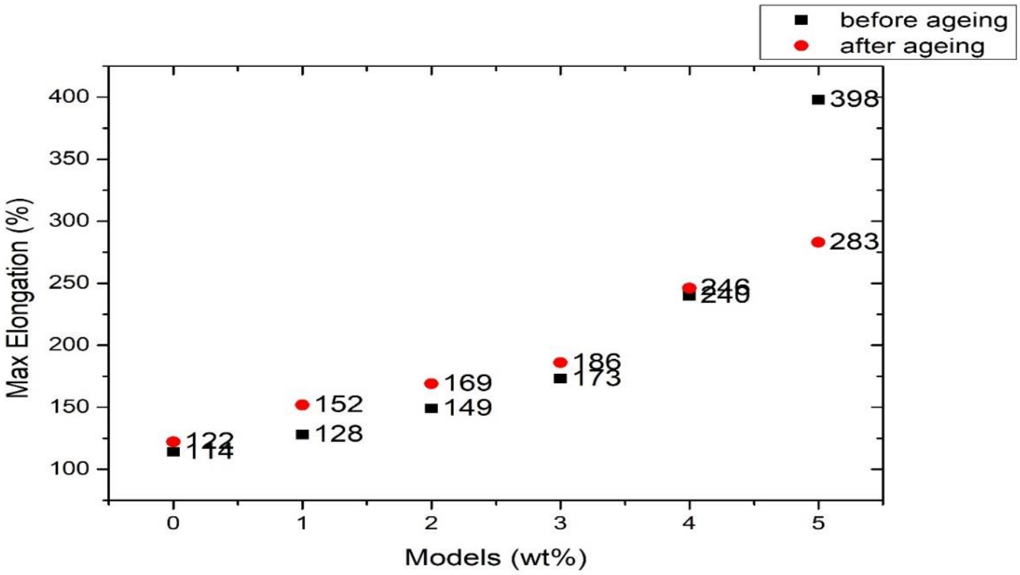


Figure 4.6 Effect of different filler loading on Maximum elongation strength of silica nanocomposite for both before and after ageing.

(c) Maximum Elongation strength of EPDM/ MWCNT Nanocomposite

In this measurement of tensile strength for EPDM/ MWCNT nanocomposite Table, 4.13 shows the Maximum elongation strength of the samples from model 0 to model 5 before ageing and Table 4.14 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.7 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.13 and Table, 4.14 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The maximum elongation strength for before ageing is 146 % for maximum filler in model 5 whereas the maximum elongation strength for after ageing sample with maximum filler loading in model 5 is 163%. The sample without the filler of nanoparticle or pure sample have a least maximum elongation strength that is for before ageing of model 0 is 126 %whereas the after ageing maximum elongation strength of model 0 is 121 %.

Table 4.13 Maximum elongation in % age of MWCNT nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	136	142	138	137	161	141
2	126	133	132	150	160	146
3	118	133	134	120	159	165

Table 4.14 Maximum elongation in % age of MWCNT nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	78	150	159	137	143	152
2	121	117	137	144	156	163
3	137	108	134	159	168	149

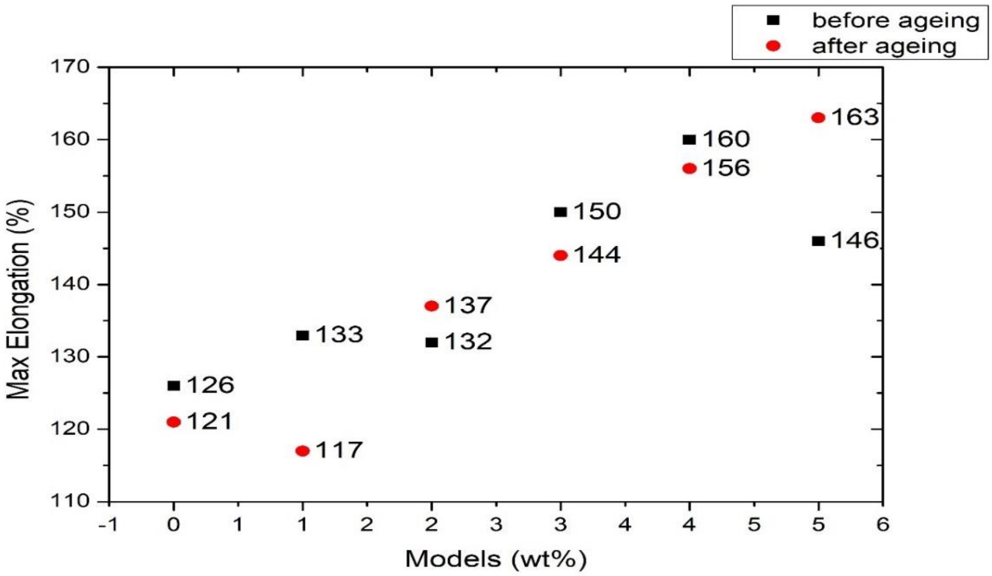


Figure 4.7 Effect of different filler loading on maximum elongation strength of mwcnt nanocomposite for both before and after ageing.

(d) Maximum Elongation strength of EPDM/ ZEOLITE Nanocomposite

In this measurement of tensile strength for EPDM/ ZEOLITE nanocomposite Table, 4.15 shows the maximum elongation strength of the samples from model 0 to model 5 before ageing and Table 4.16 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.8 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.15 and Table, 4.16 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The maximum max elongation strength for before ageing is 180 % for maximum filler in model 5 whereas the max elongation strength for after ageing sample with maximum filler loading in model 5 is 184%. The sample without the filler of nanoparticle or pure sample have a least max elongation strength that is for before ageing of model 0 is 134 %whereas the after ageing max elongation strength of model 0 is 127 %.

Table 4.15 Maximum elongation % age of ZEOLITE nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	113	171	170	132	179	223
2	134	172	162	145	145	180
3	136	158	149	123	160	211

Table 4.16 Maximum elongation % age of ZEOLITE nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	129	123	157	124	181	157
2	127	125	147	144	140	184
3	128	141	169	139	155	157

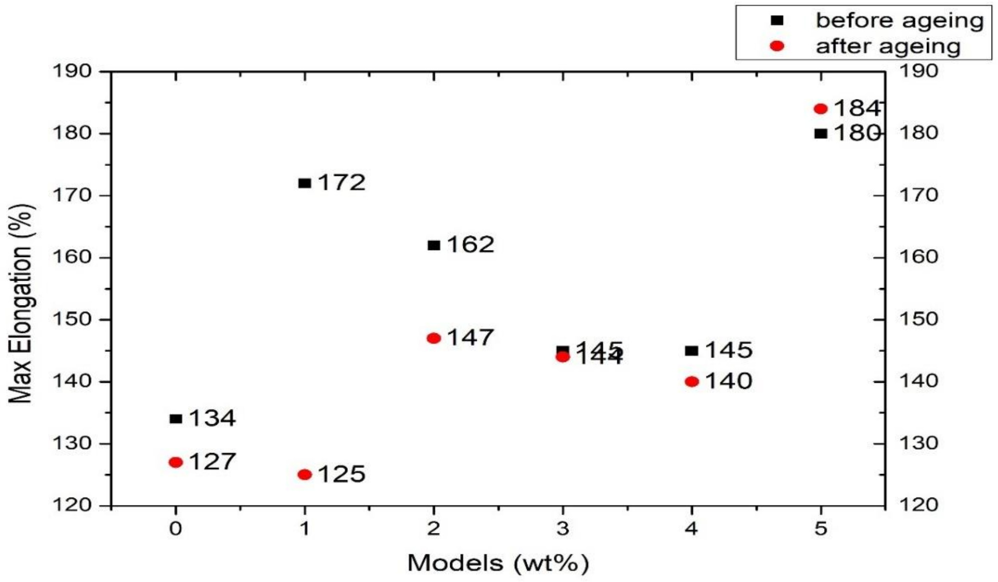


Figure 4.8 Effect of different filler loading on max elongation strength of zeolite nanocomposite for both before and after ageing.

In Figure 4.5 to Figure 4.8 shows the variation of maximum elongation strength. The order of elongation strength before and after ageing was similar to **SILICA>ZEOLITE>CLAY>MWCNT**

4.1.3. Breaking Load

In this research, we measure the breaking load strength of EPDM nanocomposite of Clay, Silica, MWCNT, Zeolite before and after ageing

(a) Breaking Load strength of EPDM/ CLAY Nanocomposite

In this measurement of breaking load for EPDM/ CLAY nanocomposite Table, 4.17 shows the breaking load strength of the samples from model 0 to model 5 before ageing and Table 4.18 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.9 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.17 and Table, 4.18 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The maximum breaking load strength for before ageing is 4.9 kg for maximum filler in model 5 whereas the breaking load strength for after ageing sample with maximum filler loading in model 5 is 4.4 kg. The sample without the filler of nanoparticle or pure sample have a least breaking load strength that is for before ageing of model 0 is 2.1 kg whereas the after ageing breaking load strength of model 0 is 2.1 kg also.

Table 4.17 Breaking load in Kg of CLAY nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	2.0	4.5	3.7	4.0	4.5	4.5
2	2.1	4.2	4.6	4.3	5.1	4.9
3	1.8	4.2	4.3	3.9	4.5	5.0

Table 4.18 Breaking load in Kg of CLAY nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	2.2	4.0	4.7	3.7	3.7	4.8
2	2.1	3.5	4.2	3.9	5.4	4.4
3	2.3	3.1	4.0	3.9	4.5	4.3

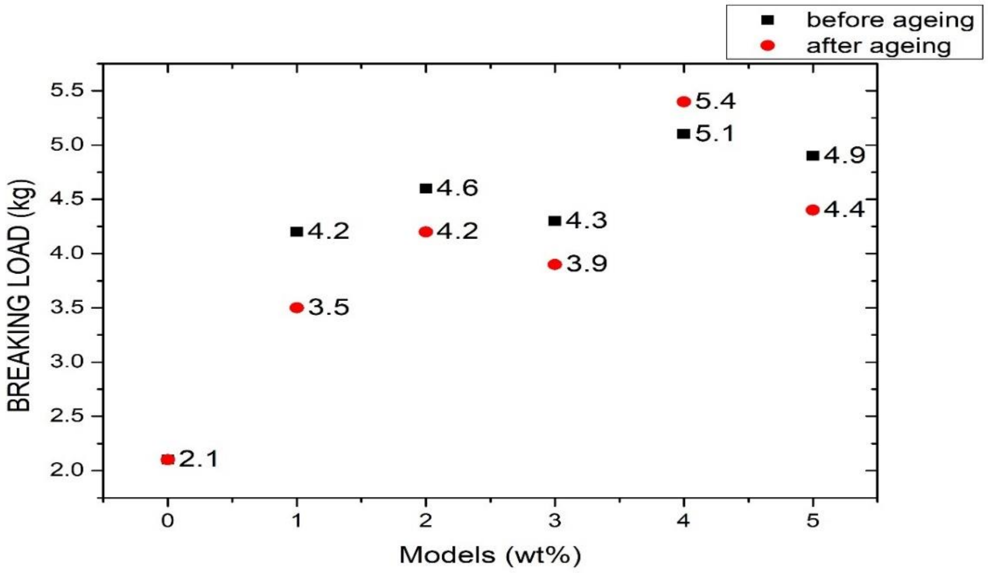


Figure 4.9 Effect of different filler loading on breaking load strength of clay nanocomposite for both before and after ageing.

(b) Breaking Load strength of EPDM/ SILICA Nanocomposite

In this measurement of breaking load for EPDM/ CLAY nanocomposite Table, 4.19 shows the breaking load strength of the samples from model 0 to model 5 before ageing and Table 4.20 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.10 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.19 and Table, 4.20 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The maximum breaking load strength for before ageing is 5.7 kg for maximum filler in model 5 whereas the breaking load strength for after ageing sample with maximum filler loading in model 5 is 4.7 kg. The sample without the filler of nanoparticle or pure sample have a least breaking load strength that is for before ageing of model 0 is 2 kg whereas the after ageing breaking load strength of model 0 is 2.2 kg also.

Table 4.19 Breaking load in Kg of SILICA nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	1.8	2.6	2.6	3.5	3.3	4.4
2	2.0	2.3	2.1	3.0	3.2	5.7
3	1.9	2.2	2.2	2.9	3.4	5.4

Table 4.20 Breaking load in Kg of SILICA nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	2.2	2.2	3.3	3.4	3.7	4.8
2	2.2	2.7	3.1	3.2	4.1	4.7
3	2.4	2.5	3.2	3.6	4.4	5.7

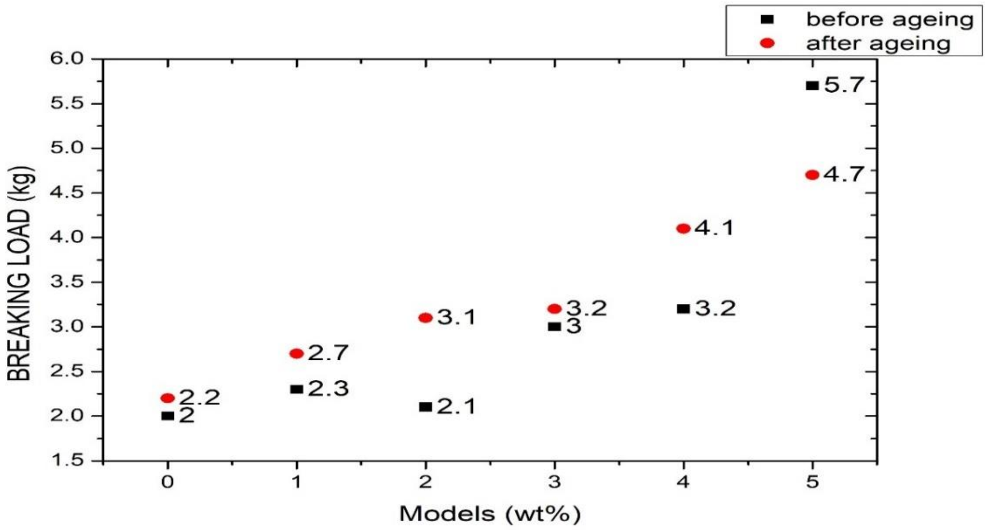


Figure 4.10 Effect of different filler loading on breaking load strength of silica nanocomposite for both before and after ageing.

(c) Breaking Load strength of EPDM/ MWCNT Nanocomposite

In this measurement of breaking load for EPDM/ MWCNT nanocomposite Table, 4.21 shows the breaking load strength of the samples from model 0 to model 5 before ageing and Table 4.22 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.11 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.21 and Table, 4.22 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The maximum breaking load strength for before ageing is 4.8 kg for maximum filler in model 5 whereas the breaking load strength for after ageing sample with maximum filler loading in model 5 is 6.2 kg. The sample without the filler of nanoparticle or pure sample have a least breaking load strength that is for before ageing of model 0 is 2.2 kg whereas the after ageing breaking load strength of model 0 is 2.1 kg also.

Table 4.21 Breaking load in Kg of MWCNT nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	2.3	2.7	3.2	3.5	4.9	4.4
2	2.2	2.6	2.8	3.5	5.0	4.8
3	2.0	2.8	2.8	2.9	4.7	5.2

Table 4.22 Breaking load in Kg of MWCNT nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	1.6	3.2	3.9	3.9	4.7	5.8
2	2.1	2.7	3.4	4.1	5.0	6.2
3	2.1	2.5	3.3	4.3	5.2	5.6

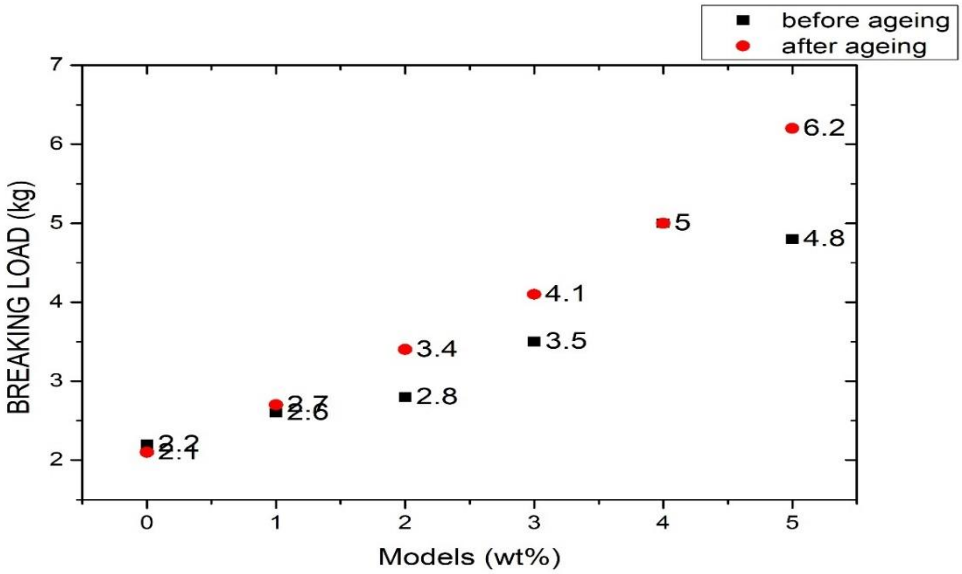


Figure 4.11 Effect of different filler loading on breaking load strength of mwcnt nanocomposite for both before and after ageing.

(d) Breaking Load strength of EPDM/ ZEOLITE Nanocomposite

In this measurement of breaking load for EPDM/ ZEOLITE nanocomposite Table, 4.23 shows the breaking load strength of the samples from model 0 to model 5 before ageing and Table 4.24 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.12 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.23 and Table, 4.24 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The maximum breaking load strength for before ageing is 3.2 kg for maximum filler in model 5 whereas the breaking load strength for after ageing sample with maximum filler loading in model 5 is 4.3 kg. The sample without the filler of nanoparticle or pure sample have a least breaking load strength that is for before ageing of model 0 is 2.2 kg whereas the after ageing breaking load strength of model 0 is 2.3 kg also

Table 4.23 Breaking load in Kg of ZEOLITE nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	2.0	2.6	2.6	3.2	3.9	3.9
2	2.2	2.6	2.3	3.4	3.4	3.2
3	2.2	2.5	2.3	3.1	3.8	3.6

Table 4.24 Breaking load in Kg of ZEOLITE nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	2.3	2.5	2.8	2.9	3.7	4.2
2	2.3	2.4	2.6	2.8	3.1	4.3
3	2.4	2.5	3.0	2.9	3.3	3.4

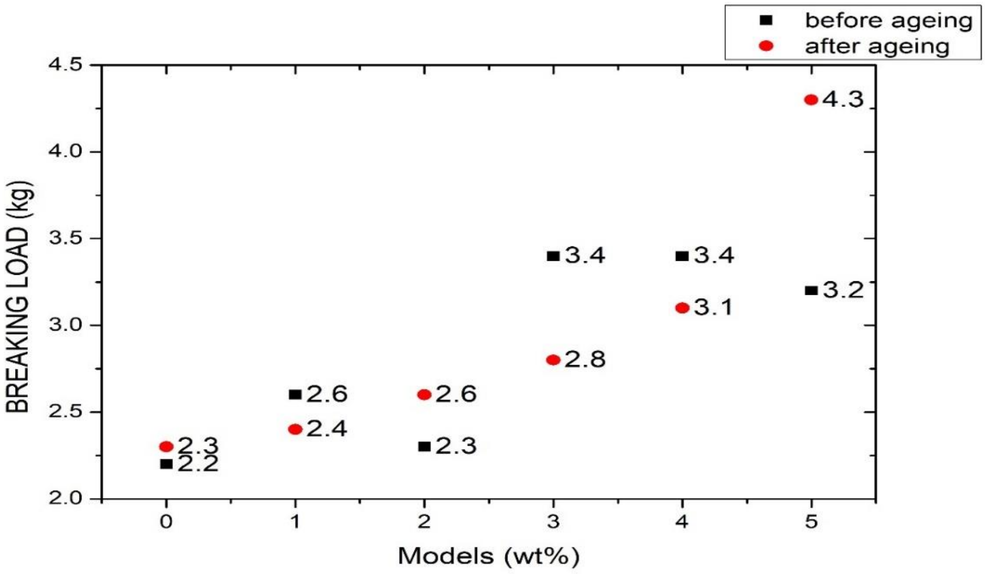


Figure 4.12 Effect of different filler loading on breaking load strength of zeolite nanocomposite for both before and after ageing.

In Figure 4.9 to 4.12 shows the variation of the breaking load of all the 4 nanoparticles. The order of breaking load for before and after ageing was respectively **SILICA>CLAY>MWCNT>ZEOLITE, MWCNT>CLAY>SILICA>ZEOLITE**

4.1.4. Modulus (100%)

In this research, we measure the modulus strength of EPDM nanocomposite of Clay, Silica, MWCNT, Zeolite before and after ageing

(a) Modulus strength of EPDM/ CLAY Nanocomposite

In this measurement of modulus strength for EPDM/ CLAY nanocomposite Table, 4.25 shows the modulus strength of the samples from model 0 to model 5 before ageing and Table 4.26 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.13 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.25 and Table, 4.26 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The modulus strength for before ageing is 22.7 kg/cm^2 for maximum filler in model 5 whereas the modulus strength for after ageing sample with maximum filler loading in model 5 is 22.2 kg/cm^2. The sample without the filler of nanoparticle or pure sample has the least modulus strength that is for before ageing of model 0 is 14.1 kg/cm^2 whereas the after-ageing modulus strength of model 0 is 15.2 kg/cm^2 also.

Table 4.25 Modulus strength in Kg/cm^2 of CLAY nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	14.2	17.7	23.3	21.3	23.1	25.5
2	14.1	17.2	21.2	21.5	24.8	22.7
3	13.5	17.1	22.5	22.2	25.2	25.3

Table 4.26 Modulus strength in Kg/cm^2 of CLAY nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	15.5	20.6	21.5	21.1	22.1	23.1
2	15.2	22.3	17.0	19.8	21.4	22.2
3	15.6	-	20.9	20.1	23.8	23.0

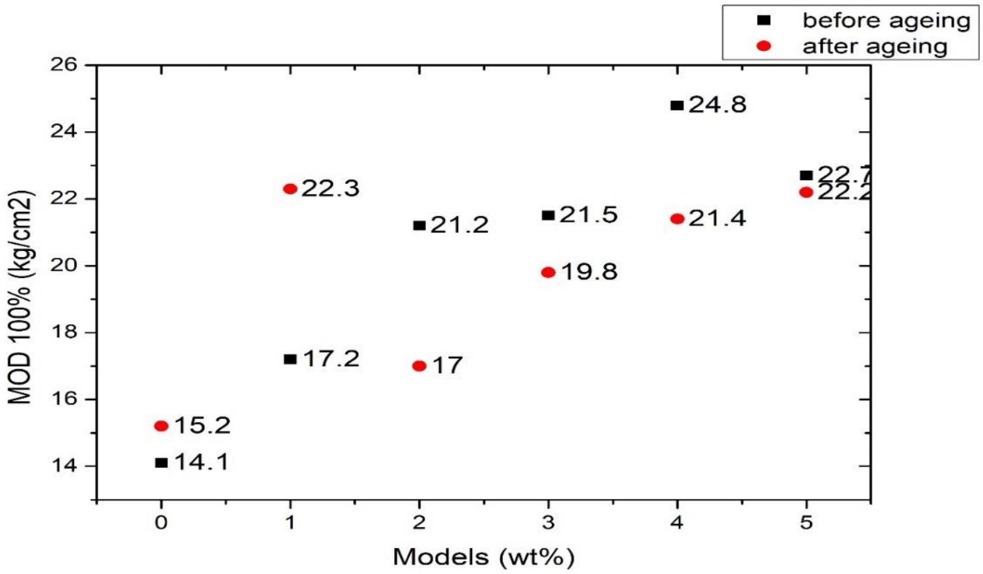


Figure 4.13 Effect of different filler loading on modulus strength of clay nanocomposite for both before and after ageing.

(b) Modulus strength of EPDM/ SILICA Nanocomposite

In this measurement of modulus strength for EPDM/ SILICA nanocomposite Table, 4.27 shows the modulus strength of the samples from model 0 to model 5 before ageing and Table 4.28 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.14 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.27 and Table, 4.28 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The modulus strength for before ageing is 13.3 kg/cm^2 for maximum filler in model 5 whereas the modulus strength for after ageing sample with maximum filler loading in model 5 is 14.4 kg/cm^2. The sample without the filler of nanoparticle or pure sample has the least modulus strength that is for before ageing of model 0 is 13.7 kg/cm^2 whereas the after-ageing modulus strength of model 0 is 14.0 kg/cm^2 also.

Table 4.27 Modulus strength in Kg/cm^2 of SILICA nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	13.8	14.3	13.9	14.6	13.0	14.0
2	13.7	14.0	13.3	14.3	12.6	13.3
3	13.8	13.7	13.5	14.5	13.2	13.2

Table 4.28 Modulus strength in Kg/cm^2 of SILICA nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	14.5	15.2	14.9	15.5	13.3	12.7
2	14.0	14.4	15.1	13.9	14.3	14.4
3	14.5	14.7	14.9	15.4	14.1	13.7

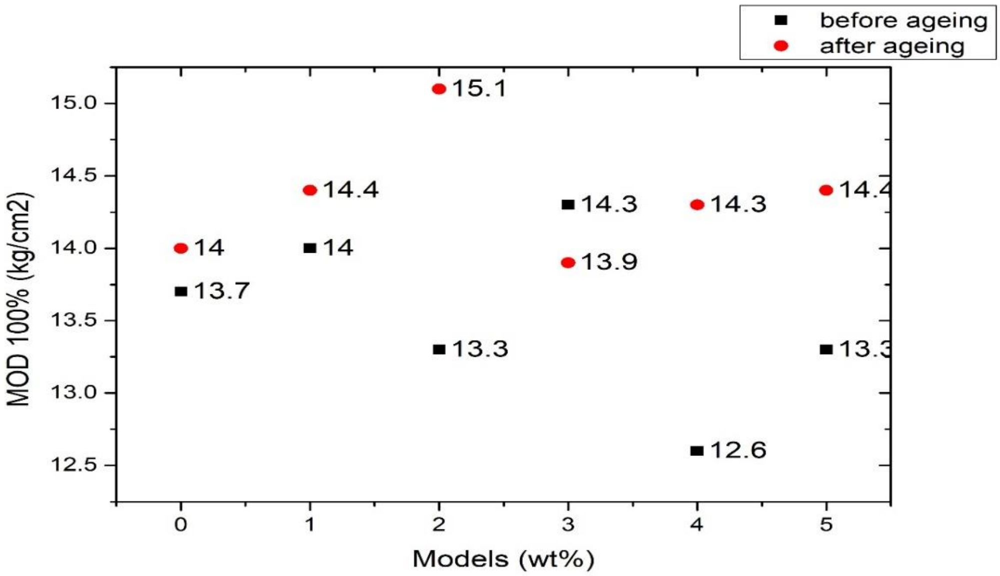


Figure 4.14 Effect of different filler loading on modulus strength of silica nanocomposite for both before and after ageing.

(c) Modulus strength of EPDM/ MWCNT Nanocomposite

In this measurement of modulus strength for EPDM/ MWCNT nanocomposite Table, 4.29 shows the modulus strength of the samples from model 0 to model 5 before ageing and Table 4.30 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.15 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.29 and Table, 4.30 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The modulus strength for before ageing is 24.3 kg/cm^2 for maximum filler in model 5 whereas the modulus strength for after ageing sample with maximum filler loading in model 5 is 24.8 kg/cm^2. The sample without the filler of nanoparticle or pure sample has the least modulus strength that is for before ageing of model 0 is 13.5 kg/cm^2 whereas the after-ageing modulus strength of model 0 is 14.0 kg/cm^2.

Table 4.29 Modulus strength in Kg/cm^2 of MWCNT nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	13.5	16.0	18.4	19.3	20.7	24.4
2	13.5	15.7	16.8	17.7	21.5	24.3
3	13.3	16.3	16.5	18.6	20.5	23.0

Table 4.30 Modulus strength in Kg/cm^2 of MWCNT nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	-	16.3	18.3	19.6	22.9	25.2
2	14.0	16.5	17.6	19.6	23.4	24.8
3	13.8	16.7	17.7	19.2	22.8	25.2

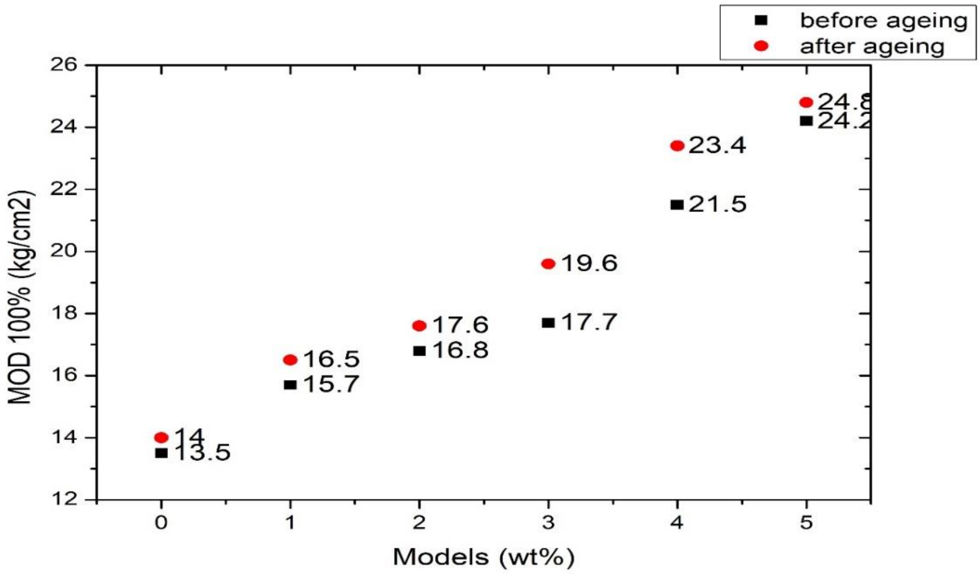


Figure 4.15 Effect of different filler loading on modulus strength of mwcnt nanocomposite for both before and after ageing.

(d) Modulus strength of EPDM/ZEOLITE Nanocomposite

In this measurement of modulus strength for EPDM/ ZEOLITE nanocomposite Table, 4.31 shows the modulus strength of the samples from model 0 to model 5 before ageing and Table 4.32 represent the samples from model 0 to model 5 after ageing. From the graph in Figure 4.16 samples from both before and after ageing are depicted differently. Here the variation due to the increase in filler loading is clearly represented for model 0 to model 5. From the reading of Table 4.31 and Table, 4.32 graphs are made by taking the average of all 3 reading from both the before and after ageing Tables. Clearly, the graphs represent the difference in the reading of both the sample models for before and after ageing. The modulus strength for before ageing is 13.2 kg/cm^2 for maximum filler in model 5 whereas the modulus strength for after ageing sample with maximum filler loading in model 5 is 17.1 kg/cm^2. The sample without the filler of nanoparticle or pure sample has the least modulus strength that is for before ageing of model 0 is 13.4 kg/cm^2 whereas the after-ageing modulus strength of model 0 is 14.3 kg/cm^2 also

Table 4.31 Modulus strength in Kg/cm^2 of ZEOLITE nanocomposite before ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	13.0	13.2	13.2	17.6	16.2	13.2
2	13.4	13.1	13.0	17.1	18.1	13.2
3	13.1	13.1	13.2	18.4	18.9	12.9

Table 4.32 Modulus strength in Kg/cm^2 of ZEOLITE nanocomposite after ageing

Sr.no.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
1	14.6	16.4	14.7	17.5	15.4	19.3
2	14.3	15.1	14.5	14.9	16.1	17.1
3	14.7	14.3	14.4	16.1	15.6	15.6

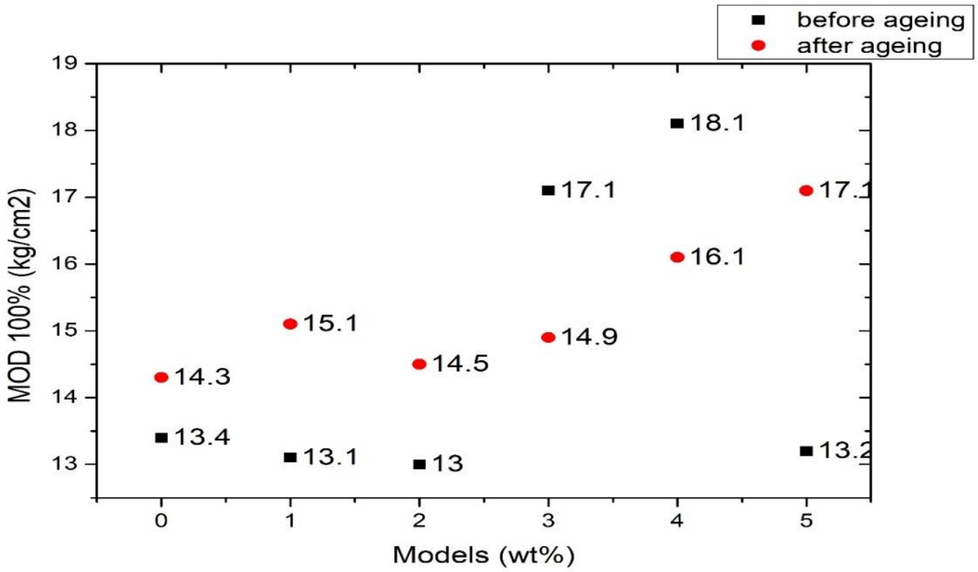


Figure 4.16 Effect of different filler loading on modulus strength of ZEOLITE nanocomposite for both before and after ageing.

Figure 4.13 to Figure 4.16 show that the variation of modulus 100%. The order of elongation strength before and after ageing was respectively **CLAY>MWCNT>ZEOLITE>SILICA, MWCNT>CLAY>ZEOLITE>SILICA.**

4.2. Standard Test Results for Rubber Vulcanisation Properties

These tests are conducted in rotor less cure meter in we generally analyse the rubber properties by using rubber analyser. This is the advanced rotor less rotational shear rheometer that can analyse rubber, polymer or rubber compound at any manufacturing point. Through this the test conducted include scorch time, scorch time delay, tan delta angles, and peak rates. Below the Tables including all parameters and with graphs are included for complete verification process. The Table 4.33 represents graph of clay, 4.34 represent silica, 4.35 mwcnt and 4.36 represent zeolite. The Figures 4.17 to Figure 4.20 represent graphs of clay, silica, mwcnt and zeolite respectively. Here scorch time is the time duration before the vulcanisation process start. The scorch time is delayed or improved by using the accelerators such as mbt or tmtd or combination of both mbt and tmtd.

Table 4.33 Vulcanisation properties of NANOCLAY

Model	S' Min	S' Max	Scorch Time	TC 10	TC 90	tan delta@90	Peak rate	Rheo Pass/fail
Unit	Lbin	Lbin	minutes	minutes	minutes	rad	Lbin/min	-
1	0.81	13.60	0.99	0.91	2.99	-	12.64	Pass
2	1.09	13.80	0.54	0.50	1.89	0.01	22.00	Pass
3	1.12	14.57	0.53	0.49	2.00	0.01	22.66	Pass
4	1.12	14.86	0.53	0.49	2.09	0.01	22.44	Pass
5	1.10	15.27	0.55	0.51	2.22	0.01	22.06	Pass
6	1.21	16.18	0.54	0.51	2.41	0.02	21.73	Pass

Average value	1.075	14.713	0.613	0.568	2.267	0.012	20.555
Minimum	0.81	13.600	0.530	0.490	1.890	0.010	12.640
maximum	1.210	16.180	0.990	0.910	2.990	0.020	22.660
Standard deviation	0.137	0.957	0.185	0.168	0.398	0.004	3.890
Coefficient of variation	12.711	6.503	0.502	0.492	0.292	37.268	18.924
Process capability Cp	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Process capability cpk	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000

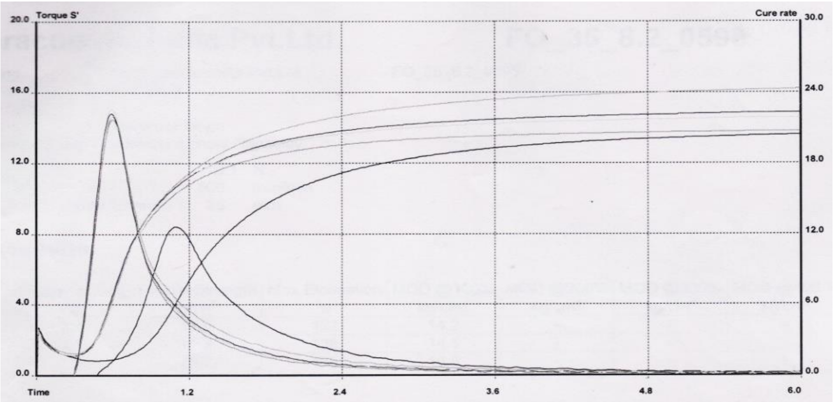


Figure 4.17 Vulcanised properties of nano clay as obtained from standard test machine MDR 3000

Table 4.34 Vulcanisation properties of NANOSILICA

Model	S' Min	S' Max	Scorch Time	TC 10	TC 90	tan delta@90	Peak rate	Rheo Pass/fail
Unit	Lbin	Lbin	minutes	minutes	minutes	rad	Lbin/min	-
1	0.76	13.53	0.84	0.76	2.44	-	14.77	Pass
2	0.80	13.56	0.78	0.71	2.38	-	16.51	Pass
3	0.77	13.29	0.79	0.72	2.79	-	15.96	Pass
4	0.84	12.76	0.79	0.71	3.35	0.01	14.82	Pass
5	0.91	11.63	0.78	0.68	3.86	0.02	12.14	Pass

Average value	0.816	12.954	0.796	0.716	2.964	0.015	14.840
Minimum	0.760	11.630	0.780	0.680	2.380	0.010	12.140
maximum	0.910	13.560	0.840	0.760	3.860	0.020	16.510
Standard deviation	0.061	0.807	0.025	0.029	0.632	0.000	1.684
Coefficient of variation	7.485	6.227	0.053	0.067	0.335	0.000	11.345
Process capability Cp	0.000	0.000	0.000	0.000	0.000	-1.000	0.000
Process capability cpk	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000

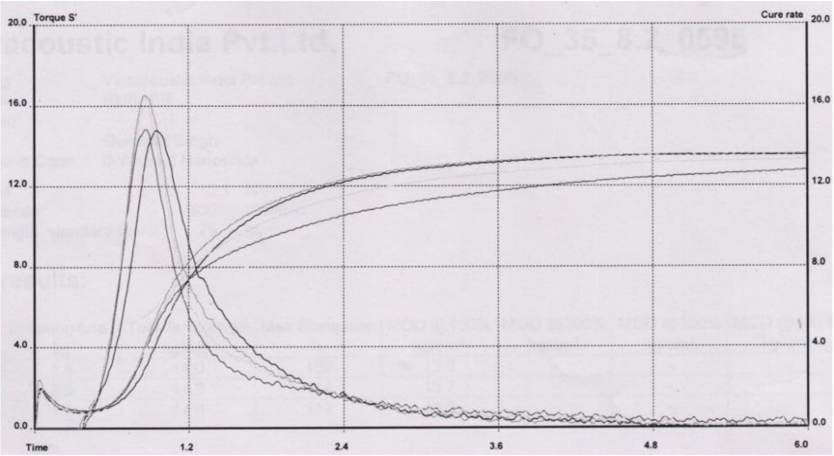


Figure 4.18 Vulcanised properties of nano silica as obtained from standard test machine MDR 3000

Table 4.35 Vulcanisation properties of MWCNT

Model	S' Min	S' Max	Scorch Time	TC 10	TC 90	tan delta@90	Peak rate	Rheo Pass/fail
Unit	Lbin	Lbin	minutes	minutes	minutes	rad	Lbin/min	-
1	0.76	13.35	0.85	0.76	2.45	-	14.03	Pass
2	0.89	14.03	0.74	0.67	2.30	0.01	15.91	Pass
3	0.97	14.85	0.72	0.66	2.42	-	16.31	Pass
4	1.12	15.21	0.71	0.65	2.35	0.01	16.92	Pass
5	1.21	16.77	0.64	0.60	2.25	0.01	21.13	Pass
6	1.35	18.08	0.61	0.58	2.34	0.02	22.18	Pass

Average value	1.050	15.382	0.712	0.653	2.352	0.013	17.747
Minimum	0.760	13.350	0.610	0.580	2.250	0.010	14.030
maximum	1.350	18.080	0.850	0.760	2.450	0.020	22.180
Standard deviation	0.218	1.759	0.084	0.063	0.074	0.005	3.195
Coefficient of variation	20.717	11.437	0.197	0.161	0.053	40.000	18.003
Process capability Cp	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Process capability cpk	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000

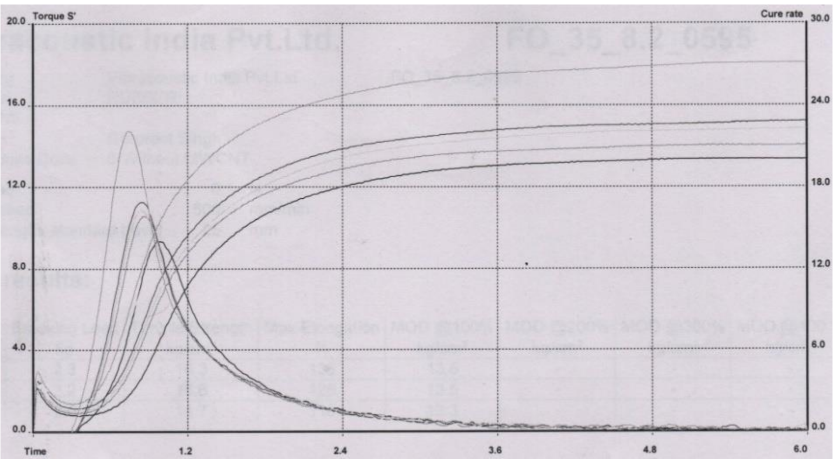


Figure 4.19 Vulcanised properties of mwcnt as obtained from standard test machine MDR 3000

Table 4.36 Vulcanisation properties of ZEOLITE

Model	S’ Min	S’ Max	Scorch Time	TC 10	TC 90	tan delta@90	Peak rate	Rheo Pass/fail
Unit	Lbin	Lbin	minutes	minutes	minutes	rad	Lbin/min	-
1	0.81	13.61	0.90	0.82	2.76	-	13.59	Pass
2	0.81	13.56	0.92	0.85	3.02	-	13.11	Pass
3	0.83	13.37	0.84	0.77	3.17	0.01	12.95	Pass
4	0.90	13.33	0.78	0.71	3.25	0.01	13.32	Pass
5	0.96	13.02	0.75	0.68	3.5	0.01	12.75	Pass
6	1.04	12.51	0.69	0.62	3.65	0.01	13.21	Pass

Average value	0.892	13.233	0.813	0.742	3.227	0.010	13.155
Minimum	0.810	12.510	0.690	0.620	2.760	0.010	12.750
maximum	1.040	13.610	0.920	0.850	3.650	0.010	13.590
Standard deviation	0.094	0.411	0.089	0.088	0.324	0.000	0.292
Coefficient of variation	10.507	3.107	0.183	0.197	0.167	0.000	2.223
Process capability Cp	0.000	0.000	0.000	0.000	0.000	-1.000	0.000
Process capability cpk	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000	-1.000

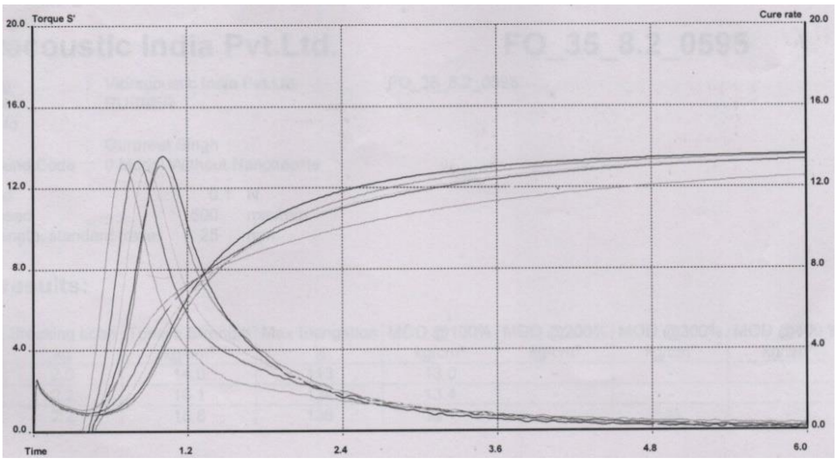


Figure 4.20 Vulcanised properties of zeolite as obtained from standard test machine MDR 3000

4.3. Electrical Properties of EPDM Nanocomposite

PARTIAL DISCHARGE(PD), is considered to be a significant phenomenon for accessing insulation performance. The objective was to study the insulation performance, to collect and analyse data of partial discharge and breakdown voltage of a pure EPDM and the EPDM with nanoparticles. The test was conducted as per IEC (International Electrotechnical Commission) Standard 60270, PD is localised electrical discharge that only partially bridges the insulation between conductors. Observation, collections, and evaluations of results of pure and samples with nanoparticles are included.

Testing and experimental work were carried out in the High Voltage Laboratory, Faculty of Electrical Engineering, Thapar Institute of Engineering and Technology. A block-shaped pure sample and with nanoparticles (in phr) as shown in formulation Table 4.37 used in the testing procedure.

Table 4.37 List of Samples for PD testing

Sample	Description
0	100% EPDM
1	100% EPDM + 1% Nanoparticle
2	100% EPDM + 2% Nanoparticle
3	100% EPDM + 3% Nanoparticle
4	100% EPDM + 4% Nanoparticle
5	100% EPDM + 5% Nanoparticle

The partial discharge test has been done on all the samples from model 0 to mode 5 for all the nanoparticles fillers CLAY, SILICA, MWCNT, ZEOLITE. The detail of all the results are formed in Table 4.38 for silica, Table 4.39 for clay, Table 4.40 for zeolite and 4.41 for mwcnt there is some space in the Table which is not filled due to the breakdown occurrence with no insulation. According to the higher values in Tables of respective nanoparticle, the graph is represented in Figure 4.21 for silica, Figure 4.22 for clay, Figure 4.23 for zeolite and Figure4.24 for mwcnt.

Table 4.38 Results of PD test of all the samples of SILICA

SAMPLE NO.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
BREAKDOWN STRENGTH (KV)	30.7	26.4	31.4	27.1	30.7	30.7
PARTIAL DISCHARGE (nc)	8.4	6.45	5.8	1.3	8.4	8.2

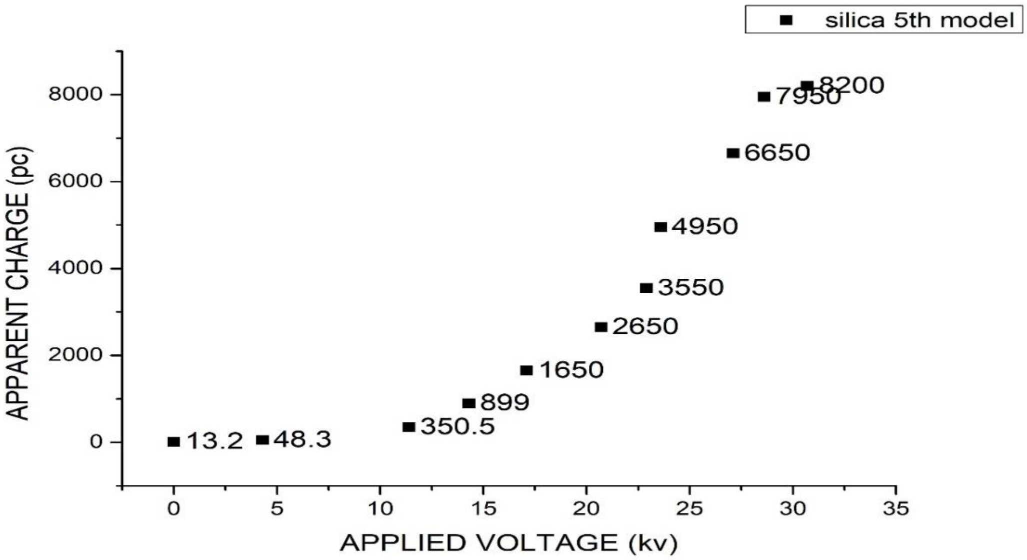


Figure 4.21 Graph of silica 5th model

Table 4.39 Results of PD test of all the samples of CLAY

SAMPLE NO.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
BREAKDOWN STRENGTH (KV)	30.1	31.4	28.7	30.7	24.3	30.7
PARTIAL DISCHARGE (nc)	8.8	7.1	8.2	.723	4.8	6.7

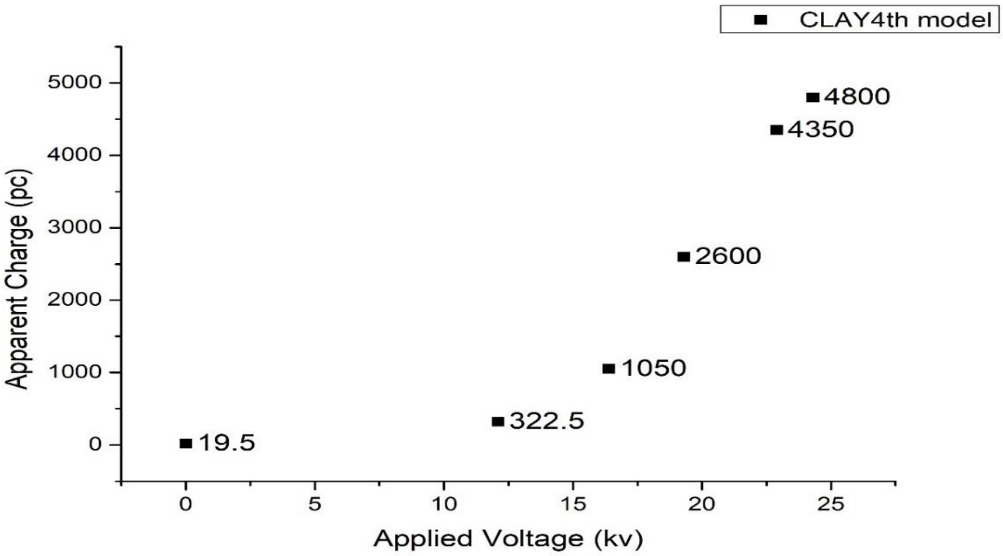


Figure 4.22 Graph of clay 4th model

Table 4.40 Results of PD test of all the samples of ZEOLITE

SAMPLE NO.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
BREAKDOWN STRENGTH (KV)	30	30.7	30.7	-	27.1	30.7
PARTIAL DISCHARGE (nc)	5.8	8.2	7.2	-	3.2	3.6

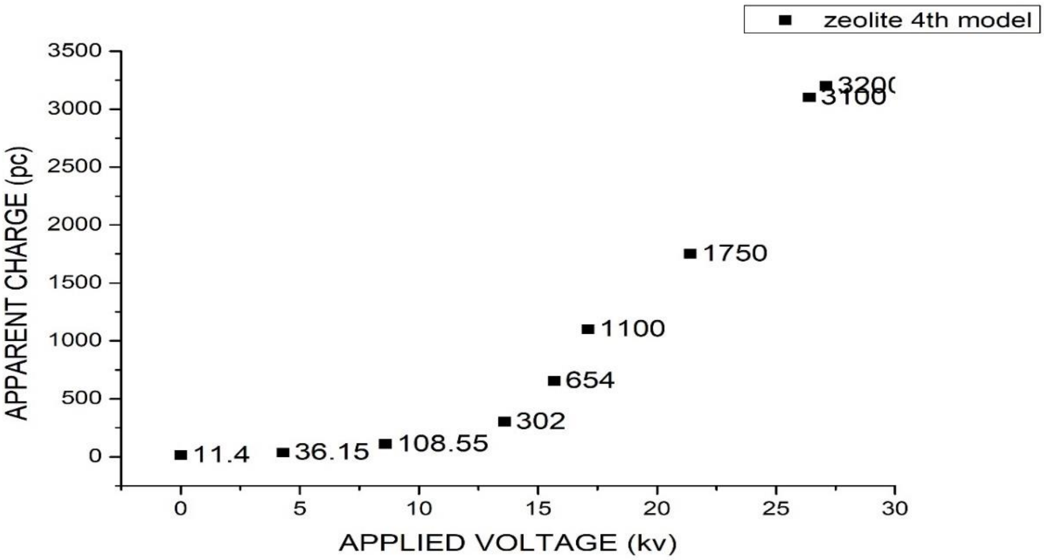


Figure 4.23 Graph of zeolite 4th model

Table 4.41 Results of PD test of all the samples of MWCNT

SAMPLE NO.	MODEL 0	MODEL 1	MODEL 2	MODEL 3	MODEL 4	MODEL 5
BREAKDOWN STRENGTH (KV)	21.4	31.4	24.3	29.3	-	-
PARTIAL DISCHARGE (nc)	.362	6.1	2.4	8.7	-	-

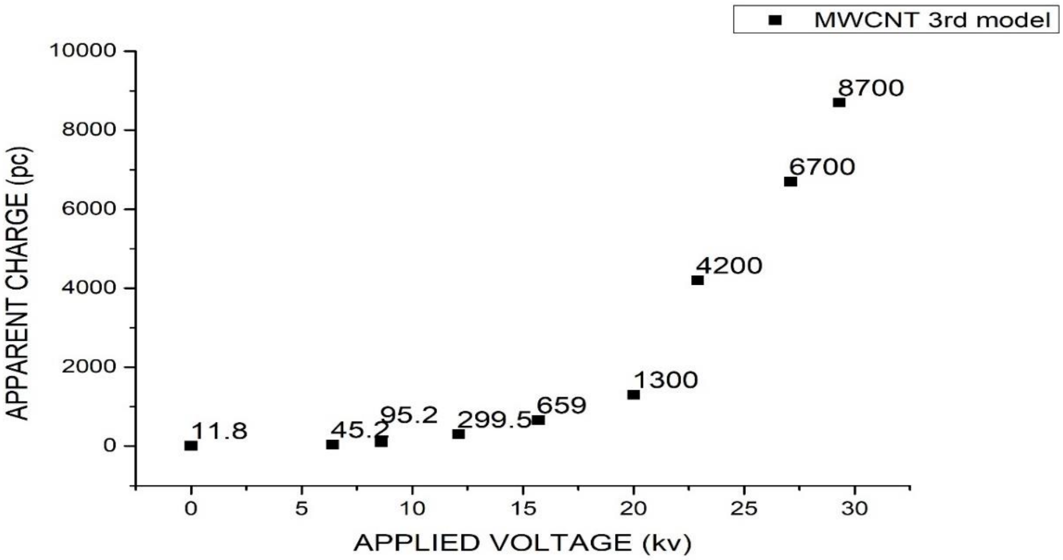


Figure 4.24 Graph of mwcnt 3rd model

The graphs of PD testing were shown in Figure 4.21 to 4.24 which implicit that the breakdown voltage of all the nanoparticle. From testing the results shows the highest breakdown strength is 31.4 KV with clay, silica and mwcnt. The partial discharge has the highest range is 8.7 with mwcnt. So, with the results we can easily rectify the filler for our demand.

CHAPATER 5
CONCLUSION AND FUTURE SCOPE

The research work discovered the effect of nanoparticle content, mixing time, and condition, ageing on mechanical and electrical behaviour of ethylene propylene diene rubber (EPDM nanocomposite).

In the research work, the EPDM nanocomposite using SILICA, CLAY, MWCNT and ZEOLITE in different weight percentage from 1phr to 5 phr are formed using the two-rolling machine. While missing stearic acid and zinc oxide act as activators and DOP as binding agent. The sulphur used for vulcanisation process and MBT, TMTD acts as accelerator. After the mixing process overs, the batch of sample is prepared which then compressed and moulded to sheets and buttons.

Now the testing part appears where first mechanical testing is conducted on the samples. The tensile and elongation test, breakdown load test and modulus test are performed to test the mechanical or physical strength of the sample prepared. The mechanical tests show that the addition of nanoparticle layer to EPDM has improved the physical strength of the EPDM nanocomposite. From the results we concluded highest tensile strength, elongation test breaking load test and modulus tests results. All these results are for both before and after ageing tests from this we can easily select our sample according to our requirement. Then the electrical tests are performed on the sample to investigate the insulation properties of the EPDM nanocomposite. The graphs show the results and the sample best suited for our requirement.

From the research work presented we can conclude that EPDM samples/composites formed with proper processing including proper mixing time and temperatures. With the addition of nanoparticle layer has improved both mechanical and electrical properties of the EPDM.

With the advancement in technology, we are shifting from conventional to renewable sources of energy. In future, the work will focus on new nanoparticles that will help in getting better intersection of materials and will provide good dispersion. They help in better electrical, mechanical, thermal properties. The thermal, hardness, tear test, X-Ray Diffraction, Scanning Electron Microscopy, are tests that will be going to performed employing EPDM nanocomposites in future aspects.

LIST OF PUBLICATIONS

- Research paper titled as **‘‘Preparation and characterization of EPDM nanocomposites for high voltage insulations applications’’** has been presented and published in the proceedings of 7th International Conference on Advancements in Engineering and Technology, held on 15-16 March, 2019 at Bhai Gurdas Institute of Engineering and Technology, Sangrur with collaboration in Springer.
- The Research paper titled as **‘‘Preparation and characterization of EPDM nanocomposites for high voltage insulations applications’’** has been communicated to the journal of ICAET, Springer and is under review.

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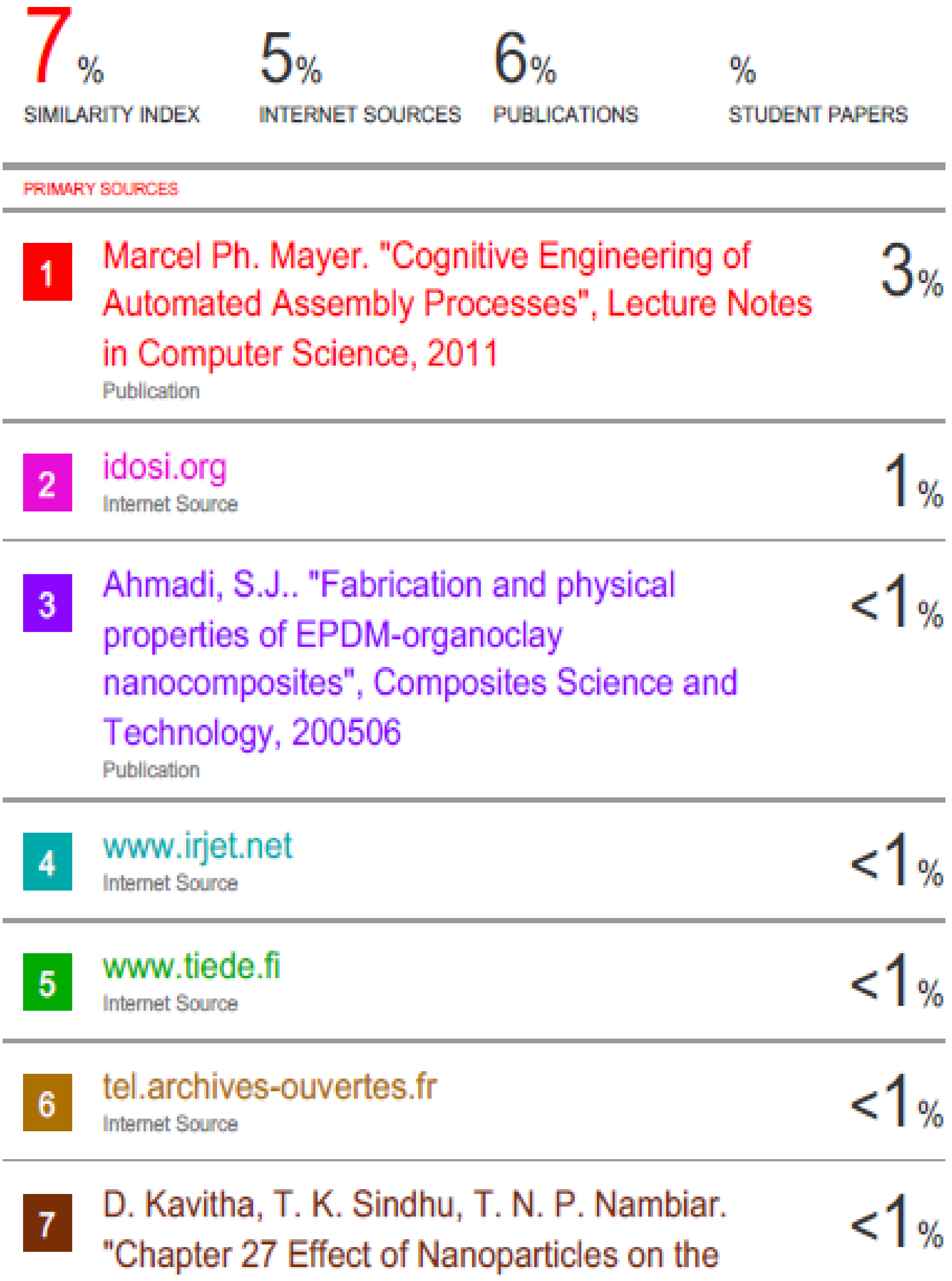

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