Graphene Cladded Fabric Reinforced Nano-Composite for Electronic Applications

By

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Graphene Cladded Fabric Reinforced Nano-Composite for Electronic Applications

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This thesis is submitted as a partial fulfillment of the requirements for the degree of

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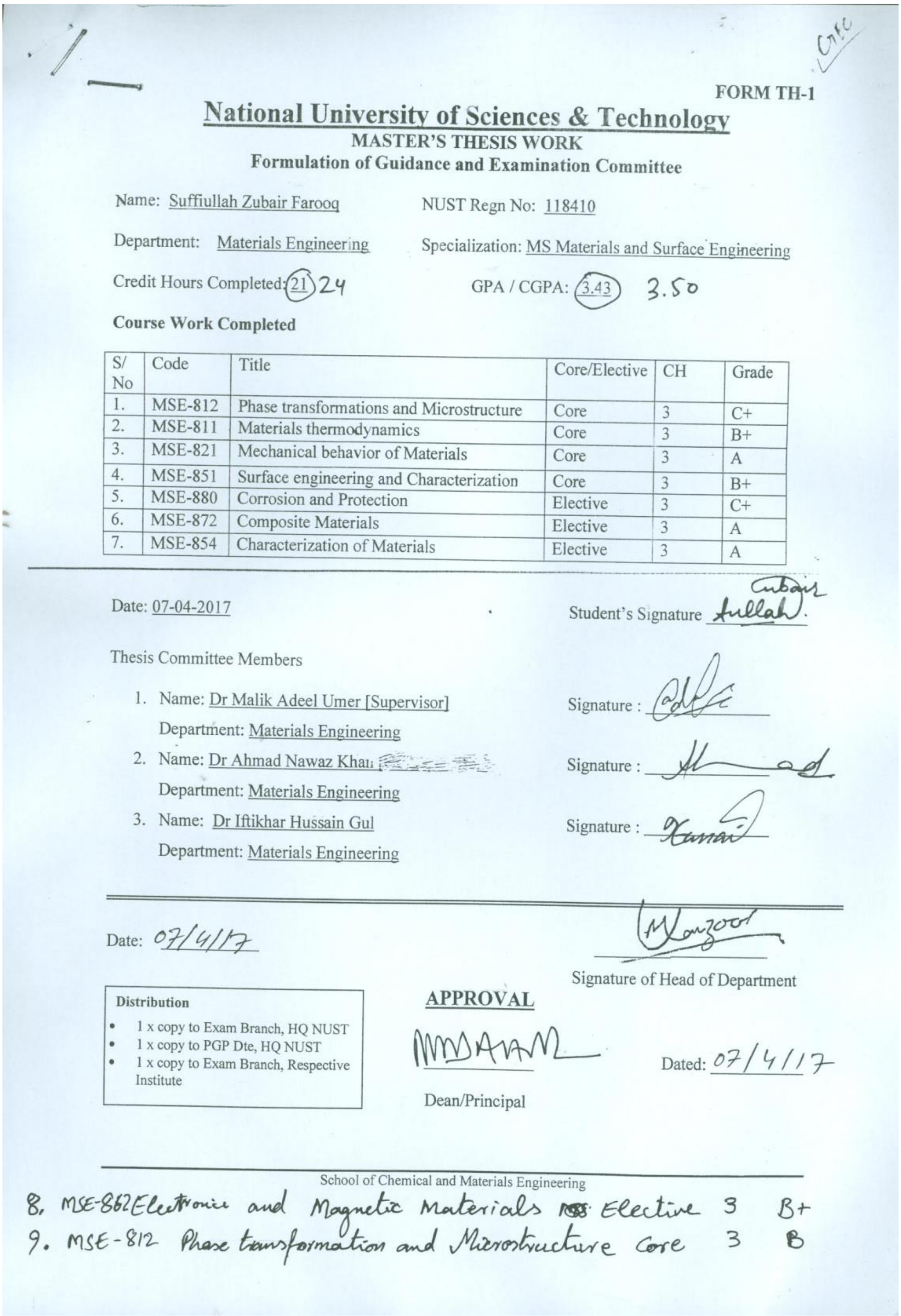

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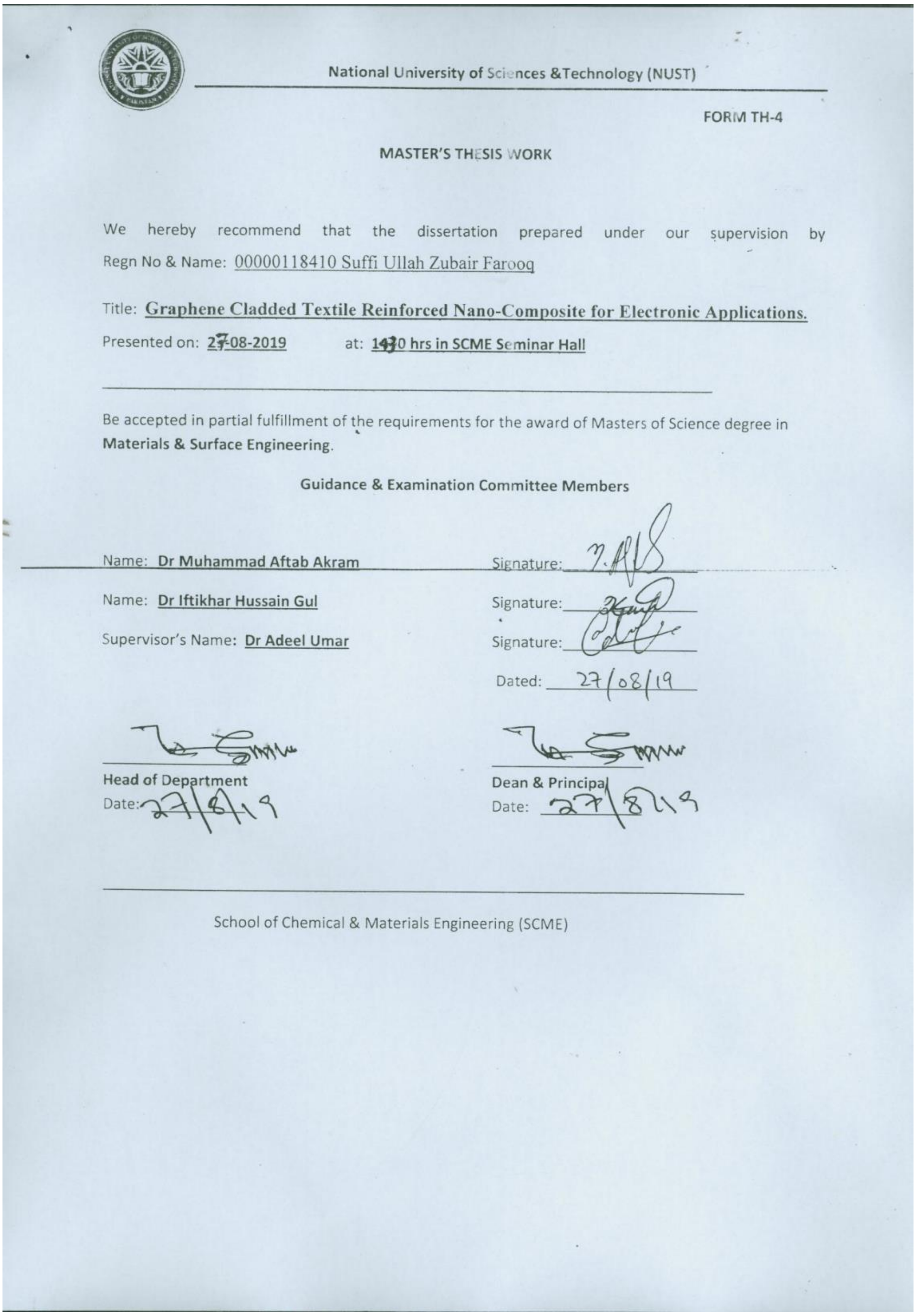

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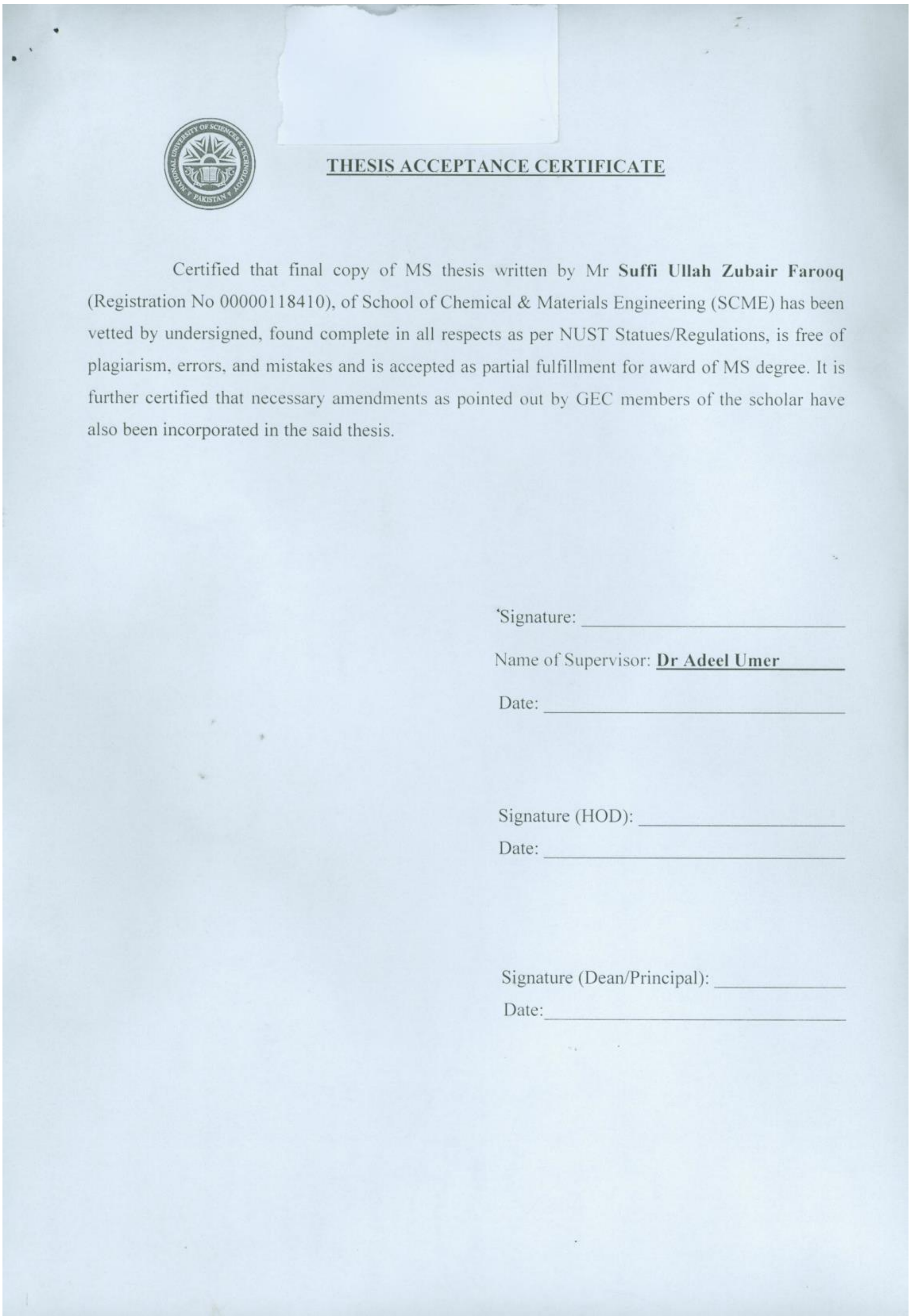

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I dedicate this thesis to my very supporting and loving Parents and Family.

Acknowledgement

"Read! In the name of your lord" (Alaq; 1st revealed ayah)

This Quranic verse sums up the entire importance of the education in the lives of humans.

It is my privilege to express my deep sense of gratitude to my research supervisor, **Dr. Malik Adeel Umer**, for his constant support and affectionate guidance throughout this research work. I would also like to thank my committee members **Dr. Iftikhar Hussain Gul** and **Dr. Muhammad Aftab Akram** for giving valuable guidance and suggestions.

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Abstract

A novel approach was idealized to overcome the dispersion problem of Graphene in usual solution cast conductive nano-composites. Significant increase in AC conductivity was observed in the rGo coated Nylon fabric embedded PMMA matrix that makes it perfect for electronic applications. $NaNO_3$-free Improved hummers method was used to synthesize Graphene Oxide involving Graphite powder as a precursor. Graphite powder was mixed with $KMnO_4$ and was stirred with H_2SO_4 and H_3PO_4 (9:1). For reaction termination H_2O_2 was used and the supernatant was subsequently washed and filtered. Nylon fabric was coated by GO solution having concentration of 2.5mg/ml and then chemically reduced using Hydrazine obtaining rGO cladded textile. Afterwards, we incorporated Graphene cladded Nylon fabric in PMMA matrix via solution casting. The XRD graphs confirmed corresponding phase and crystallinity shift of Graphite to GO and then rGO. FTIR and UV-Visible spectroscopy confirmed relative absorption and tansmission peaks characteristic for GO and rGO. SEM micrographs supported the morphology analysis for GO,rGO, cladded and uncoated fabrics. Impedance analysis predicts that our PMMA composite can behave well in smart or intelligent textile applications.

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Chapter 1

Introduction

1.1 Composite Materials

Composite is a multi-phase material that shows properties that are superior, or important in some other aspect, to the properties of the individual components. Composite Materials corresponds to every single strong material made up of more than one component. Another meaning of composite materials is that these are the strong materials, which are made up of a matrix system (i.e. polymer, metals and ceramics) that surrounds and bond together the reinforcements. In result of which better properties can be fulfilled by the mixing two or more particular materials [1].

Composite materials have commonly two phases, matrix which is the coherent phase and dispersed phase which binds together by the matrix.

Now, why to choose polymer as a matrix over other materials? The answer is due to its high specific weight and specific strength. Higher specific modulus elaborates that the weight of the system can be decreased, and this is the main concern towards the energy conservation in modern technologies. Composites can be classified according to reinforcement types as shown in Fig. 1.1.

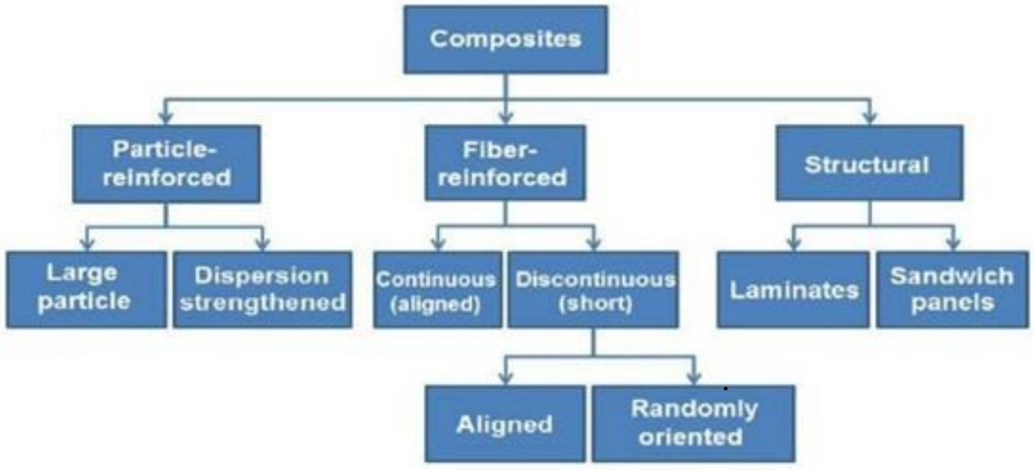


Figure 1.1: Types of Composites [2]

1.2 Nano-composites

Nano-composites are compounds having at least one component is in nanometer range dimensions. These are high-functional materials that combine unusual properties and show unusual design possibilities and are believed to be 21st century material. 18,000 publications including research papers and patents have been published on nanomaterials in the last two decades [3]. It is said that lower than 100 nanometers, properties of materials can be deeply transformed. Only reducing the size and without changing materials structure itself because of the increase in surface area. New properties can be shown by the material like elasticity, electrical conductivity, insulating behavior, different colors, higher strength, and greater\lesser reactivity that the same material do not show in either macro or micro level [4].

1.2.1 Classification of Nanocomposites

Now a days, fast-growing area of research is nanocomposites either organic or inorganic. Huge efforts are being done to control the nano-level structures by new synthetic ways. The characteristic properties of the nano-composites are not only related to the properties of their individual components, but also related to their morphology and surface interactions [5].

1.2.1.1 Organic/Inorganic Nano-composites and Lamellar Nano-composites

The overall group of organic / inorganic nano-level composites may be related to biomineralization and bio-ceramics, in which the growth of bio-polymer and inorganic matrix is done by in-situ polymerization. Ultimately, the nano-level composites of lamellar structure show maximum surface interaction between the components.

1.2.1.2 Industrial Applications

The experiments usually show that all types of nano-level composites show new and much better properties, compared to their micro-level structure material. Therefore, nanocomposites have potential in many new applications in a lot of areas such as nonlinear optics, reinforced lightweight armored components, battery cathodes, nanowires, gas sensors and some other systems as well [6].

1.2.1.3 Types of Nano-fillers

The properties of Nano composite do not simply depend upon separate ingredient’s properties, but also on their surface characteristics and morphology of fillers. Choice of nano fillers, for example, filler size and shape have a huge effect on the properties of nano composites and their applications as well. There are three broad classes of fillers depending on their geometries that have been used in polymer Nano composites.

- Particles
- Fibrils
- Sheets

Comparing these three filler geometries, lead to a conclusion that the particle Nano fillers have the maximum surface area to volume ratio. Fig. 1.2 elaborates reinforcement types and relevant surface area [7].

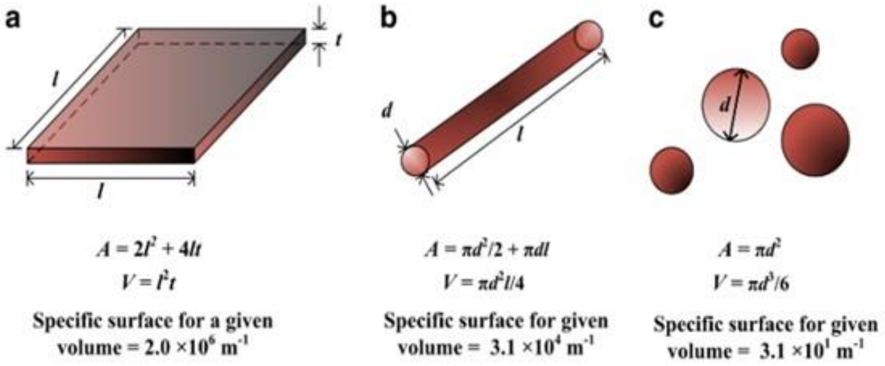


Figure 1.2: Types of fillers [7] **(a)** Sheet **(b)** Fibril **(c)** Particle

1.3 Polymers

Polymers are long-chain molecules with high molecular weight materials. These repeating units are frequently made up of carbon and hydrogen. These repeating units are chemically linked together to form polymers. Synthetic polymers term was first used in the mid twentieth century. Some example of natural polymers, that were utilized are cotton, starch, proteins, and fleece that define life and engineered polymers are Polyvinylidene fluoride (PVDF), Poly-methyl methacrylate (PMMA), Bakelite and Nylon, which are being utilized in modern technologies. Fig. 1.3 show different types of polymers. When scientist reached on point that there is strong relationship between structure and properties of polymer, they started to study about amorphous and crystalline structures of polymers due to their chain alignment [8].

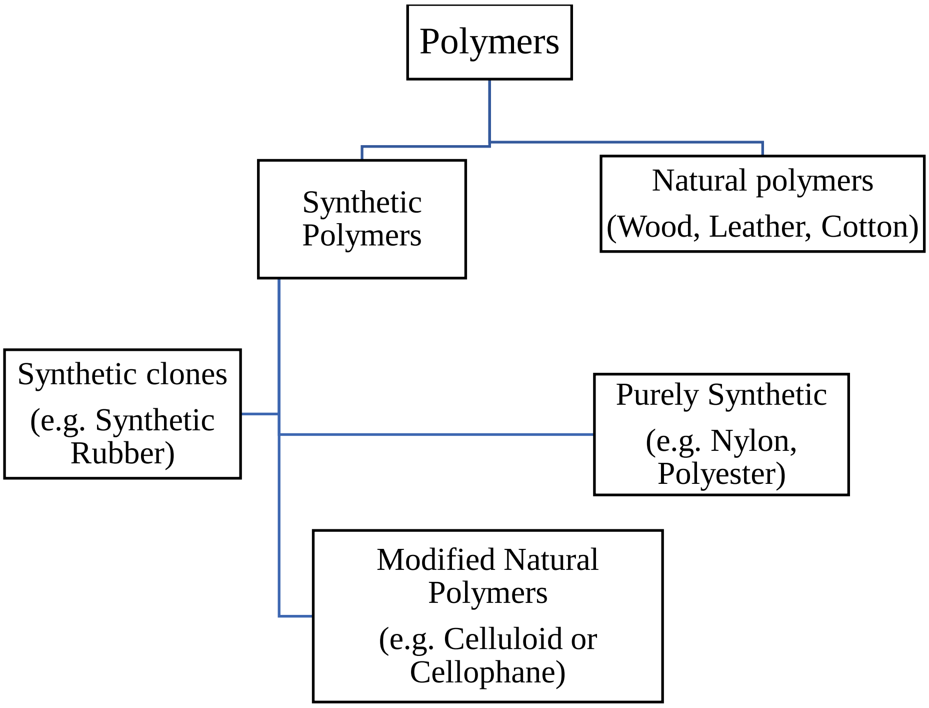


Figure 1.3: Types of Polymers [8]

1.3.1 The Polymer structure:

Polymers are composed of hydrocarbons, compounds of hydrogen and carbon. Polymers are specifically made up of bonded atoms of carbon together into long chains, which are the backbone of the polymer. As carbon atom is capable to form more than one bond with other atom, many other atoms are attached to carbon atom in the backbone.

Polyvinylidene fluoride (PVDF) and Teflon has fluorine attached to all carbon atom backbone. Chlorine is attached to carbon backbone in Polyvinyl chloride. Other common polymers have other atoms other than carbon attached to their backbone. Nylon has connected nitrogen in their backbone while polystyrene and polycarbonates contain oxygen. Some other polymers which contain silicon or phosphorus in their backbone are termed as inorganic polymer. One of the common inorganic polymers which consist of silicon backbone is Silly Putty [9].

1.3.2 Molecular Arrangement of Polymers

Linear polymers are arranged like spaghetti noodles look in a plate, if polymer lack specific order or amorphous in other words. Direct cooling of molten polymer leads to amorphous arrangement. Amorphous polymer has no proper arrangement in their molecules. Due to lack of proper arrangement, amorphous polymer is generally transparent. This property of amorphous polymer plays an important role in plastic windows, food wrap, contact lenses and headlight lenses, while crystalline polymer is usually translucent and opaque due to their arrangement of molecules. Fig. 1.4 demonstrates the different chain structure in polymers. Crystallinity and amorphousness property depend upon the condition of processing and on the type of polymers. Transparency of polymers is inversely proportion to crystallinity, more crystallinity in the polymer, less will be transparency. Crystallinity in polymers makes advantages in strength, stability, stiffness and chemical resistance. Scientist and researchers are always trying to synthesize more useful materials by controlling the molecular structure that effects in resultant polymer produced [9].

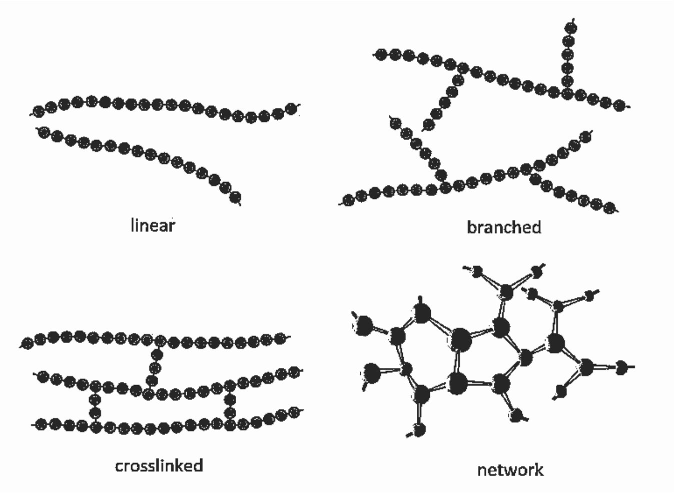


Figure 1.4: Polymer structure of **(a)** linear, **(b)** branched, **(c)** crosslinked, and **(d)** network (three-dimensional) molecular structures. Circles designate individual repeat units [9].

1.3.3 Classification of Polymers

A thermoplastic type of polymer is one which gets soften at specific higher temperature and solidify again after cooling. They are high molecular weight polymers. The polymer chains collaborate through intermolecular forces. Which enfeeble quickly with rise in temperature and are transformed into paste like fluid. Thermoplastics are dissimilar from thermosetting polymers. Thermosets don't liquefy and deteriorate at high temperature. Before jumping directly on the polymer plastics, we must have to recall brief history and need of these materials. With expansion of human population and increase in inter continent trade and business around the world and life style of people make them feel the incompetence of existing materials to make different equipment and goods of their needs. Those existing methods and materials were not enough to meet their increasing standards and needs which were increasing day by day. So, keeping in mind about naturally occurring rubber they tried to replicate these which were then used for insulation, dental and medical purposes having very low production ability. In nineteenth century developments were made to some extent which with time proved their worth and showed their importance and now they are everywhere around us, improving standards of our lives and without them now our lives are considered to be very difficult [6].

1.3.3.1 Thermoplastics

It’s a polymer material which can change or transform into viscous liquid from solid state upon heating below their melting point temperature and above the T_g glass transition temperature. It again gains its solid form upon cooling without the expense of any mechanical properties and this cycle may repeated over and over to some limited numbers which make this polymer type more affordable and suitable. From economic and environmental point of view, it can easily be recycled. Its heat cycle is shown in Fig. 1.5. With increased recycle numbers some degradation may appear. As we understood that this is recycle-able polymer and easy to mold, this made it clear to us that it is very easy to handle making it possible for re-using and molding it to any desirable shape and size, due to these reasons they are also termed as thermos softening polymer from name it is clear that they become soft on applying heat.

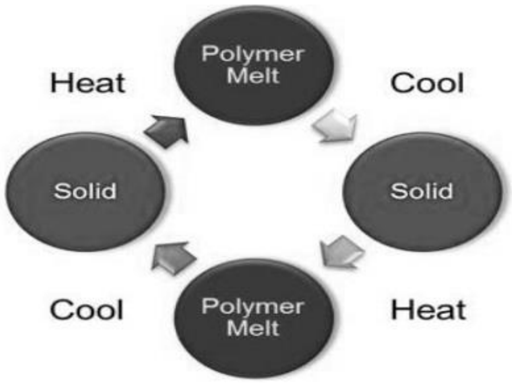


Figure 1.5: Thermoplastics

Thermo softening polymers have linear molecular structure in which molecules with high molecular weight are joined end to end which after connections form long chains due which partial crystallinity also developed in them and chain which are formed in the result are independent from each other while on temperature higher than their melting point the crystallinity diminishes due to random scattering of chains [10].

Thermoplastic polymers have properties like high strength, toughness and better hardness and durability along with waterproof and transparency with chemical resistance. This all changes with molecular structure of polymer chains. As molecular structure has influence on these properties along with optical properties which may change from transparent to opaque due to change in structure while physical properties change abruptly above the glass transition temperature.

These polymers are processed by using some of the techniques like Injection or compression molding and extrusion etc. Some of the examples include Acrylic, ABS, Nylon PLA, PMMA, PVC, Polystyrene etc. They have wide range of applications from electrical insulation to automotive and spacecraft industry.

1.3.3.2 Thermosetting

These polymer materials also share the same history as of the thermoplastic. Naturally occurring resin and rubber were present but those were not enough to meet the demands of the growing world, so synthetic plastics were developed and utilized with great scope and application in nineteenth century. Thermosetting polymer or plastic are materials which become permanently solid, hard and rigid when they cooled after the heating them on melting temperature. Unlike thermoplastics, it is very clear from here that these thermosets can be heated and shaped only once. Their form changes from liquid to permanently solid. We conclude they cannot be recycled, and it is not easy to re-use them but somehow if they are made to experience more heat they start decomposing chemically.

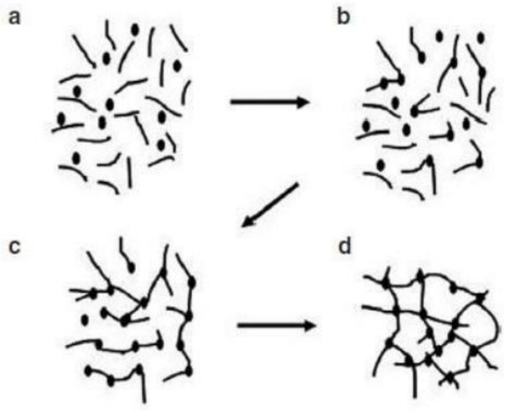


Figure 1.6: Crosslinking/Curing of polymer chains in Thermosets [11]
(a) Precursor/Resin **(b)** linear growth and branching
(c) Bridging and interlinking of Polymer chains **(d)** Cured Polymer

These polymers have very complex cross-linked structure. Curing stages are shown in Fig. 1.6. Each chain is held together with strong covalent bond and in the curing, a process of cooling and solidification of polymer, the small but high molecular weight molecules are cross-linked in network manner which is complicated. Due to presence of covalent bond in between these chains, it is very difficult for chains to slipover. The only condition possible is breaking of bonds on some high energy or force which make them permanently deformed and unable to re shape. They are synthesized using resins under the high pressure conditions while some catalyst is present in the system or at suitable temperature in atmospheric conditions, some cross linking agent is utilized for this purpose .this cross linking mostly happen during the curing and cross linking depend on the amount of the monomer and pre- polymer also some conditions around them which helps their linking like temperature and pressure.

We can say these thermosetting polymers as having high thermal performance and stability. They have resistant nature against heat, corrosion and creep. They also have better hardness and the only drawback is that they are brittle and deformed permanently. The specie of thermoset is thermoset

1.3.3.3 Elastomers

Historically Natural rubber have great importance and it is utilized from very long time which is extracted from trees .but in modern era most of development in this material in the nineteenth century with production of synthetic rubber and different methods of synthesis were adopted to synthesis them in large scale with efficient quality and on the speedy way, true synthetic rubber was developed during the world wars period and substitute of natural rubber came into race.

The specialty of elastomer is that it can return to its shape after removing applied force along with fact it they can also decompose thermally before their melting point. Due to this reason they are synthesized and utilized simultaneously. Their property of high thermal stability is due to cross linking and covalent bond which keep them to hold their shape against high temperature and thus depends on the density of crosslink's present in polymer which enable them against chemicals and give them mechanical strength and they are also processed just like thermoplastic in injection molding ,compression molding or their combination injection–compression molding etc. They also have wide and exclusive range of application from automotive, electrical, to energy etc.

The ability of polymer materials to possess properties of viscosity and elasticity together is termed as viscous – elastic behavior. Simply we say they bear elastic property, having long chains of atoms (mostly oxygen, hydrogen, carbon etc.) with weak intermolecular forces and cross link bond with adjacent chain which bring them back to original shape. Elasticity bearing materials are Elastomers mostly rubber, which is naturally occurring. This cross-linkage may be done by process called vulcanization. Elastomers have greater strain failure with smaller young's modulus in comparison to different other materials.

They are totally amorphous above T_g. If we have to distinguish between thermoplastics and Elastomers, then we can say that Elastomers are soft and stretch-able due to presence of weak binding forces which allow them to stretch on stressing and their glass transition temperature is below the room temperature while thermoplastics have higher T_g. The chains of Elastomers distribute almost uniformly due to which they can reinstate their shape. Comparative chain arrangement of thermoplastics, thermosets and elastomers is shown in Fig. 1.7.

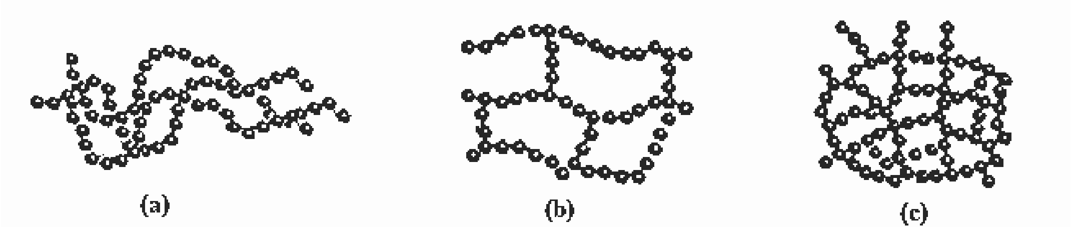


Figure 1.7: Schematic Molecular arrangement of Polymer chains [12]
(a) Thermoplastics **(b)** Elastomers **(c)** Thermosets

Presence of some covalent bond crosslinks make it possible that they return to their initial shape having flexibility and if these cross links are removed somehow, they will deform permanently. Temperature plays an affecting role in them as if Elastomers are cured in glassy phase or crystalline phase their chains are less moveable, reducing the elasticity. The whole of the elastomeric material may be consisting of just single molecule which make it difficult along with crosslinks to recycle them. New ways are carved out to synthesize thermoplastic Elastomers, which can be recycled while having properties of Elastomers. They have mostly applications like seals and adhesives.

1.3.4 Characteristics of Polymers

Major class of polymers manufactured is thermoplastic, which can be heated and reformed again and again. But the thermosets can’t be re-melted over and over again. Degradation occurs in thermoset polymers when they are reheated. Every polymer has different characteristics, but most of the polymers have following general properties [9].

1.3.4.1 Chemically resistant polymers

Polymers are used as a packaging material for cleaning fluids. Some of the polymers are readily dissolved in chemical, while other polymers provide easy, safe and non-breakable packages for such aggressive solvents.

1.3.4.2 Dielectric in nature

Due to their insulative nature, polymers are mostly used for appliances, electrical outlets, wiring and cords that are either made from polymer or fully covered by polymeric materials. Foam core of refrigerator and freezers made of polymers to provide thermal resistance. Skiers uses thermal underwear, which is made of polypropylene and acrylic and polyester are used to made fiberfill in winter jackets [3].

1.3.4.3 High strength to weight ratio

From toys to the frame structure of space stations, or to bulletproof vests which is made of Kevlar. Some polymers used in water applications as they float while some sinks. But if we compare polymers to other materials like metals, ceramics, they are light weight and high specific strength.

1.3.4.4 Extensively diverse processability

Extrusion and Injection molding process are used to produce thin fibers or bottles or heavy pipes, or large part of car body. Plastics can also be mixed with solvents to make paints and adhesives. Elastomers and some other polymers stretch to very large percentage and commonly used in tires.

1.3.4.5 Wide variety in colors and attributes

Polymer properties can be further enhanced by mixing additives to increase their uses and applications. Now a days, polymers can largely made to mimic silk, marble, cotton, wool fibers, porcelain, aluminum and zinc. As polymers are easily moldable, this property of polymer makes it valuable in large number of applications that do not readily come from natural world.

1.3.4.6 Not always composed of petroleum

Many polymers are made of units derived from crude oil, coal and natural gas. But the primary repeating block units can be created from bio-materials such as cellulose from cotton linters or polylactic acids from corn.

1.3.4.7 No alternative option

Waterproof and clear films can be made from polymers. Tubing for medical and bags for blood that increase the blood life are made from polyvinyl chloride (PVC). PVC easily transfers fire-catching oxygen in inflammable flexible tubing. For open dialysis, collection of blood and heart surgery, Anti-thrombogenic components such as heparin, can be introduced into flexible PVC catheters. Polymers play an important role in many medical devices to permit effective functioning.

1.4 Poly methyl methacrylate (PMMA)

Polymethyl methacrylate (PMMA), also known as acrylic glass, is a straightforward and nonflexible thermoplastic material generally utilized as a breakproof swap for glass. It has large specialized points of interest over other straightforward polymer (e.g. PC, polystyrene.), few include:

- High protection from UV light.
- High transmission of light
- Boundless shading alternatives

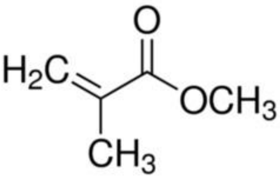


Figure 1.8: MMA Monomer [13]

PMMA is obtained from methyl methacrylate monomer. Fig. 1.8 show chemical formula of MMA. PMMA is a reasonable, dry plastic accessible in pellet, little sheet and granule structures, which can be shaped with every thermoplastic processing strategy (counting infusion forming, pressure embellishment, and expulsion). The most noteworthy quality PMMA sheets are delivered by cell throwing, however for this situation, the polymerization and trim advances happen simultaneously. It is normally called acrylic glass [14].

The quality of the material is higher than embellishment evaluations inferable from its amazingly high atomic mass. Elastic toughening has also been utilized to build the strength of PMMA. It is 100% recyclable.

1.4.1 Properties of PMMA Polymer

PMMA polymer displays glass-like characteristics (e.g. clearness, brightness, straightforwardness, translucence). It is less hazardous. We can talk about PMMA properties and advantages in detail below [15].

1.4.1.1 Transmittance

Having a Refractive Index of 1.49, the PMMA, gives high light transparency. PMMA enable 92% fraction of light to go through it, better than glass or different plastics. The plastics can undoubtedly be thermally formed with no misfortune in transparency. When contrasted with polyethylene and polystyrene, the PMMA is prescribed for most outside application on account of its natural security.

1.4.1.2 Surface Hardness

PMMA is an intense, strong and light thermoplastic. Thickness of acrylic extends between 1.17 g/cm^3-1.20 g/cm^3 which is less than 50% for that of glass. It has great scratch obstruction in competition with other polymers like Polycarbonate, but not like glass. It has low absorption and water retention, regarding that items formed by PMMA have high dimensional solidness.

1.4.1.3 UV Stability

Highly safe from UV light and high absorption. PMMA is okay for outdoor applications planned for long haul.

1.4.1.4 Chemical Resistance

Not affected by fluid arrangements of quite a number of research center synthetic compounds, by cleansers, and aliphatic hydrocarbons, cleaners, alkalis and weaken inorganic acids. Be that as it may, acrylics are not suggested for use with chlorinated or sweet-smelling hydrocarbons, esters, or ketones.

1.4.2 Preparation Methods of PMMA

Poly (methyl methacrylate) is obtained by free-radical polymerization of methyl methacrylate in mass (when it is in sheet structure) or suspension polymerization. Fig. 1.9 show schematic for free radical polymerization.

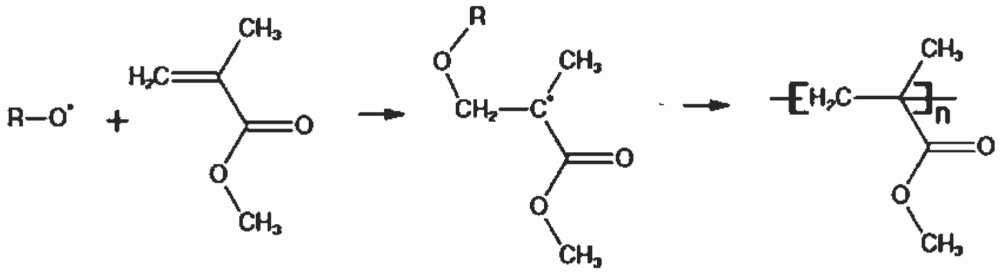


Figure 1.9: Free radical polymerization of MMA [13]

1.4.3 Conditions for PMMA processing

PMMA can be processed by extrusion, extrusion blow molding, casting and thermoforming, injection molding [16]. Pre-drying isn't important if a vented chamber is utilized, however, if a typical chamber is utilized then PMMA must be handled dry and it is prudent to pre-evaporate the granules up to 8 hours holding on 70 °C -100 °C. Surface deformities and rankles will shape if sodden granules are handled.

1.4.3.1 Injection Molding

Important process parameters include:

- Temperature for Melting: 200 °C -250 °C
- Temperature for Moulding: 40 °C -80 °C
- Large Pressures for injection are required because the flow properties of PMMA are poor and to achieve the correct flow the slow injection of polymer is needed
- Heating of PMMA at 80 °C is required to eliminate internal stresses.

1.4.3.2 Extrusion

Important process parameters include:

- Temperature: 180 °C -250 °C
- Screw for degassing having an L/D ratio of twenty to thirty is optimal.

1.4.4 Applications of PMMA

Because of its glass like properties, lightweight and even better durability than glass, it became famous in World War II. It was broadly utilized making flying machine windshields, coverings and firearm barrels. Few other business applications were created for PMMA, for example, glass material, façade configuration, publicizing, car headlamps, and so on.

Nowadays several grades acrylic polymer are used in diverse multipurpose applications. Major applications are in the sector of architecture and construction, lighting, automotive and transportation, electronics, medical and healthcare and furniture [17].

1.5 Nylon and Nylon Fabric

The polymer of Nylon is a plastic with very-long, overwhelming particles developed of short, perpetually rehashing segments of molecules likewise as a substantial metal chain is made of repeating rehashing connections. Nylon isn't a single entity, yet the name given to a group of polyamides [18].

Nylon fabric is a polymer fabric made up of Nylon fiber strands, which implies that it is made from a long chain of carbon-based particles called monomers. There are many various kinds of nylon, however the greater part of them are gotten from polyamide monomers that are extricated from unrefined crude oil, which is otherwise called petroleum.

Mostly, a monomer known as hexamethylenediamine is used in the production of nylon, and this substance is occasionally referred as diamine acid for short. This monomer is obtained from petroleum.

1.5.1 Preparation Method

For the synthesis of famous nylon, diamine acid for example hexamethyl diamine ($H_2N(CH_2)_6NH_2$) reacts with adipic acid ($C_6H_{10}O_4$). Fig. 1.10 show Hexamethyl diamine chemical formula. The polymer of this type is ordinarily known as PA 6,6, and it’s the basic polymer to be utilized for nylon fabric. PA 6,6 is a called nylon salt, and this solidified substance is then warmed to shape a liquid mass [19].

Figure 1.10: Hexamethyl diamine [19]

This flowing mass is then expelled through a spinneret, which is a gadget that appear to have many minor openings. Upon expulsion via the spinneret, mass quickly solidifies, and the subsequent strands are then prepared to be stacked onto bobbins.

Strands then extended to build their quality and versatility, and then are rounded onto another spool in a procedure called "drawing." This procedure makes the polymer particles orchestrate in a parallel fashion, and in the end of illustration procedure, the subsequent filaments are prepared to be formed into articles of clothing or different types of filaments.

Now and again, nylon might be spun into fabric all alone, however it is generally joined with different textures to make blended materials. It is then colored to create the shading that is wanted for the finished result [17].

1.5.2 Different Types of Nylon Fabric

There are many artificially extraordinary polymers textiles that are altogether called "nylon." Some sorts of these fabrics include:

1.5.2.1 Nylon 6,6

One of the primary, completely engineered fabricated polymers textile, and it come into form by joining hexamethylenediamine and dicarboxylic acid. The subsequent salt can be dissolved to frame filaments or solidified for cleansing purpose.

1.5.2.2 Nylon 46

Only created by universal organization DSM, and it is known by the name Stanyel. While this polymer isn't normally utilized in textiles, it is famous for protection from ungracious situations, and it is regularly found in motor segments, for example, transmissions, air cooling frameworks and brakes.

1.5.2.3 Nylon 510

In the beginning, created by DuPont in contrast to nylon 6,6. In any case, creation costs at last precluded the large-scale manufacturing of this polymer for textile, and its currently fundamentally utilized in modern and logical applications.

1.5.2.4 Nylon 1,6

This polymer is blend of formaldehyde, water and adiponitrile, but it isn't normally utilized in textile [18].

1.5.3 Application of Nylon Fabric:

Until now, clothing of women is one major applications of this type of textile and it is also used in yoga pants, tights and other types of form-fitting bottoms.

This kind of textile is additionally normally utilized in sportswear, yet it is substandard compared to natural or semi-manufactured filaments in this application. In any case, a few parts of this fabric, for example, its versatility, are wanted in sportswear. Regardless of whether they are principally made out of different fabrics, some sportswear producers incorporate nylon texture in their material mixes for improved flexibility and softness [5, 20].

1.6 Graphene

Graphene, having a remarkable two-dimensional structure, contains numerous planar sheets of carbon atoms which are sp^2-hybridized. Its discovery was made in 2004. It has become the most attractive material of today and the future due to his extraordinary properties. Few of its mostly utilized applications are in polymer nanocomposites, polymer framework composites which consolidate nanoscale filler materials [21].

1.6.1 Synthesis of Graphene

Some of the methods are as follows [22]:

- Adhesive tape or scotch tape method
- Liquid phase sonication
- Chemical approach
- Epitaxial growth
- Chemical vapor deposition

1.6.1.1 Scotch Tape Method

In this technique graphene layer is extracted from crystal lattice of Graphite with the assistance of a sticky tape. As the tape is removed from graphite, the graphene with multi layers is still on the tape. So, if we do the peeling repeatedly so the multilayer graphene can be converted into few layers of graphene. So, after the peeling process is completed, we attach the tape to a substrate, the glue will be dissolved by acetone, in order to remove the tape. At the end, we do peel for the last time with an unused tape.

1.6.1.2 Liquid Phase Method

This method which will permit us to upscale the yield, with a specific end goal to get a much higher quantity of graphene. One of the least difficult strategies will be, to disperse it in an organic nature solvent with just about the same surface reactivity as graphite. So, this is how we reduce the energy barrier, which must be overcome so as to separate a graphene layer from the lattice. Sonication of the solution in ultrasound bath for a few hours for this purpose. After that, the solution must be centrifuged with a specific end goal to discard the thicker flakes.

1.6.1.3 Epitaxial Growth

Graphene is synthesized by simultaneous heating and cooling down a SiC crystal. Where Few-layer graphene has started growing on C face. The consequent results are dependent on the condition such as temperature, pressure or rate of heating. Indeed, Synthesis of nanotubes can occur instead of graphene by increase in temperature and pressure up to certain limit. SiC was graphitized in 1955, while it was a unfavorable result rather than a technique for making graphene [23].

1.6.2 Properties of Graphene

Graphene has a very large theoretical surface area that gives enormous variety of possibilities for its functionalization while retaining mechanical properties and flexibility. Table 1 highlights some of the promising properties for Graphene. It has very high electrical and thermal conduction comparable to metals due to high mobility of charge carriers. Mechanical properties are extraordinary as well [24].

Table 1: Graphene Properties [25, 26]

NO.	Properties	Average Values
1	Young’s modulus	1TPa
2	Fracture strength	1.25TPa
3	Elastic Modulus	0.25TPa
4	Thermal Conductivity	$5000 Wm^{-1}K^{-1}$
5	Mobility of charge carriers	$2x10^5 cm^2V^{-1}s^{-1}$
6	Specific surface area	$2630 m^2g^{-1}$

1.6.3 Applications

Graphene has amazed the world due to its extraordinary properties and applications. It can be used in structural applications due to its remarkable mechanical properties. It is also utilized for semi-conducting applications, advanced materials, nano-composites, electromagnetic shielding, antistatic coatings, electroluminescent displays, Field effect transistors and many more applications as shown in Fig. 1.11 [26].

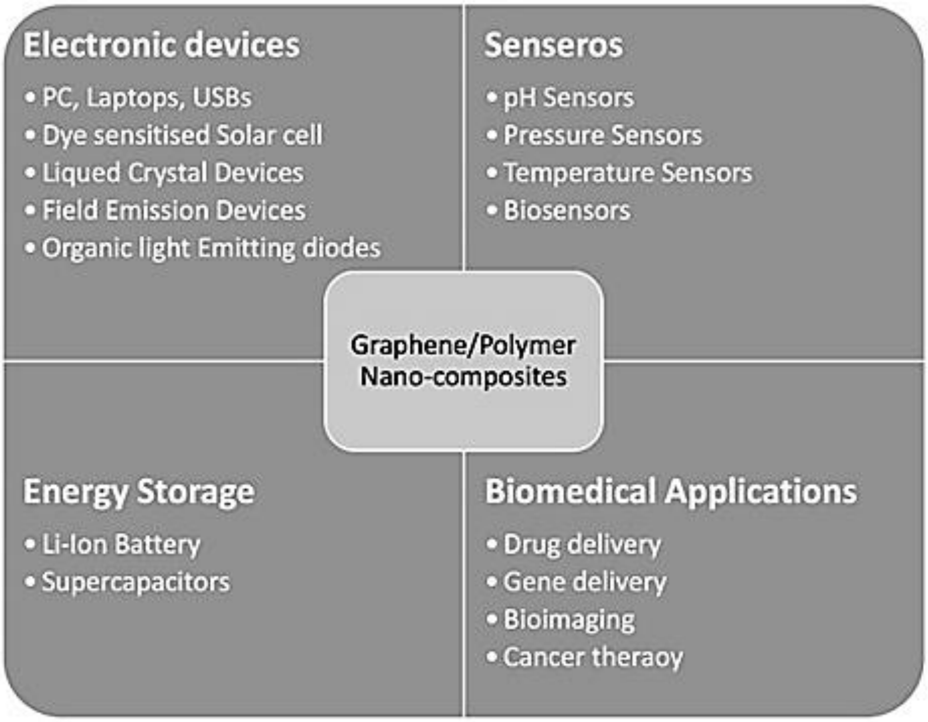


Figure 1.11: Applications of Graphene [23, 24, 26, 27]

Chapter 2

Literature Review

Nariman Yousefi et al. [28] made RGO/epoxy nanocomposites with anisotropic mechanical and electrical properties showing particularly high dielectric constants and better EMI Shielding Efficiencies [EMI SE]. The large rGO sheets and their arrangement into a layered structure is significant in accomplishing a dielectric constant value 14000 with 3 wt.% of rGO at 1 kHz, which is known to be among the most elevated dielectric constants announced in open writing. The almost parallel rGO sheets inside epoxy film maintain a system comprising of various nanocapacitors that offer a high charge stockpiling ability. The nanocomposites can be successful EMI protecting materials because of their higher attribution of exceptional EMI SE up to 38dB. The examination of EMI SEs between different materials features the upsides of the exceedingly adjusted rGO/epoxy composites which can fulfill different necessities of elite EMI protecting materials, for example, light weight and magnificent mechanical properties.

Z. J. Li et al. [29] integrated Few layer graphene (FLG) sheets of high electricall conductivity by a multi-step intercalation decline strategy. The electrical conductivity of the as-fused FLG is evaluated to be $\sim 3.2 \times 10^4$ Sm^{-1}, for all intents and purposes indistinguishable from that of perfect graphite. SEM, TEM and Raman examination reveal that the as-fused FLG sheets have gigantic regions with single moreover, twofold layers. The specific capacitance of 180 Fg^{-1} is gained for the FLG in a 1M Na_2SO_4 liquid electrolyte by organizing the cyclic voltammogram. The extraordinary capacitance of the FLG is promising for the application to become predominant electrochemical supercapacitors.

T. Kuila et al. [30] portrayed their work as Poly(methyl methacrylate) (PMMA)/graphene nanocomposites were set up by in situ emulsion polymerization. Raman and FTIR exhibited that PMMA polymer contained little graphite oxide. Dynamic mechanical assessment and differential filtering calorimetry examination exhibited that graphene in the PMMA structure went as filler; it improved the capacitance moduli and glass change temperatures of the nanocomposites.

Thermogravimetric examination showed that the warm steadfastness of the nanocomposites extended by 35 °C. The electrical conductivity of nanocomposite with 3 wt.% graphite oxide was 1.5 S m^{-1} at room temperature.

G. Kandho et al. [31] were accounted for as The composite films of Poly (vinyl liquor) (PVA) and Reduced graphene oxide (RGO) with 1wt%, 1.5wt% and 2wt% stacking of RGO utilizing arrangement blending have been readied by means of evaporative throwing procedure. For this reason, RGO has been synthetically blended utilizing modified Hummer's strategy and portrayed utilizing UV–Visible ingestion, Fourier Transform Infrared (FTIR) spectroscopy and Transmission electron Microscopy (TEM). Further, the compound communication of PVA chains with RGO has been uncovered utilizing FTIR spectroscopy. The variety in dielectric parameter (ε′ and ε′′) with recurrence has been learned at different temperatures (320K–400K) so as to encapsulate the dielectric unwinding conduct in the readied composite films. The estimations of ε′ and ε′′ have been found to increment with ascend in temperature and stacking of RGO. The practically direct variety in air conditioning conductivity (σac) with recurrence at each considered temperature recommends the perception of general power law in these composites. Be that as it may, the incline of these bends was found to diminish with ascend in temperature proposing the Correlated Barrier Hopping (CBH) model as charge transport component in these composites. The non-symmetric state of electric modulus (M″) variety show the presence of non-Debye kind of unwinding. The unwinding time(τ) decided from M ″variation with recurrence is observed to be diminished with ascend in temperature. This clarifies the change of long range to short range versatility of accuse bearers of ascend in temperature. Further, as an effect of increment in RGO focus in composites, increment in τ has been discovered delineating their utilization in vitality stockpiling applications where materials with longer releasing time are required.

C. Zhao et al. [32] announced the coming of self-fueled utilitarian pieces of clothing has offered ascend to an interest for stretchable vitality stockpiling gadgets that are amendable to coordination into material structures. The electromaterials (anode, cathode and separator) are relied upon to continue a misshapening of 3% to 55% related with body development. Here, we report a stretchable texture supercapacitor terminal utilizing usually accessible nylon lycra textile as the substrate and graphene oxide (GO) as a dyestuff. It was readied by means of an easy coloring methodology pursued by a gentle synthetic decrease. This decreased graphene oxide (rGO) covered texture anode holds conductivity at a connected strain of up to 200%. It conveys a particular capacitance of 12.3 Fg^{-1} at an output pace of $5mVs^{-1}$ in 1.0 M lithium sulfate fluid arrangement. The capacitance is essentially expanded to 114 Fg^{-1} with the expansion of a synthetically orchestrated polypyrrole (PPy) covering. This PPy-rGO-texture anode exhibits an improved cycling soundness and a higher capacitance at half strain when contrasted with the presentation saw with no strain.

S. N. Tripathi et al. [33] the effect of filler incorporation techniques on the electrical and mechanical properties of reduced graphene oxide (rGO) filled poly(methyl methacrylate) (PMMA) nanocomposites. Composites were set up by three extraordinary systems. One was in situ. polymerisation of MMA monomer in presence of rGO, mass polymerization of MMA in proximity of PMMA granules/rGO and by in situ polymerization of MMA in proximity of rGO sought after by sheet tossing. In particular, the effect of wire of fluctuating totals (for instance running from 0.1 to 2 % w/w) of rGO on the electrical, thermal, morphological and mechanical properties of PMMA was analyzed. The electrical conductivity was seen to be in a general sense subject to the proportion of rGO. The electrical conductivity of 2 wt% RGO-stacked PMMA composite was extended by factor of 107, when composites were set up by in situ polymerization of MMA inside rGO and PMMA, while, on different occasions increase in conductivity was seen at the equal RGO content when composites were set up by tossing method. FTIR and Raman spectra proposed the similarity of compound as among rGO and PMMA system, however XRD models, SEM and HRTEM studies exhibit that among three procedures, the sheet-tossing has better interconnectivity.

Chapter 3

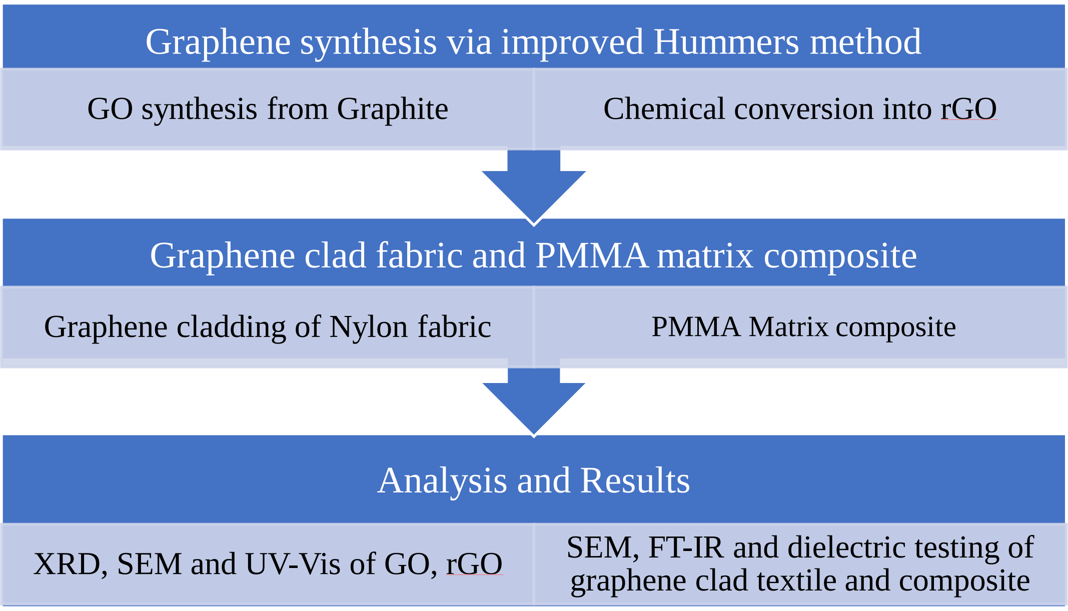
Experimental Work

3.1 Material Selection

Materials required for the Nano-composite were 1,1,2,2-dichloroethane, Tetrahydrofuran, Dimethylformamide, Styrene Acrylonitrile, Polymethylmethacrylate, Graphite powder, Nylon Fabric, Potassium permanganate, Sulfuric acid, Phosphoric acid, hydrochloric acid, De-ionized water and distilled water. First, compatibility of solvents with materials was established. DMF proved to be best for rGO dispersion.

3.2 Approach

For synthesis of PMMA matrix nano-composite, firstly, I synthesized Graphene oxide and reduced Graphene oxide via chemical route named as improved Hummers method [34]. After necessary analysis with XRD, UV-Vis spectroscopy and SEM, I dip and dry coated Nylon with GO solution of 2.5mg/ml to get GO cladded Nylon fabric. GO coated Nylon fabric was reduced by Hydrazine. Finally, rGO cladded fabric was embedded in PMMA matrix by solution casting method.



3.3 Methodology

3.3.1 GO synthesis

GO was synthesized via improved hummers method [35]. First, 3g of Graphite and 18g of Potassium permanganate are mixed inside a 1000ml beaker. Sulfuric acid and Phosphoric acid in ratio of 9:1 i.e. 360ml and 40ml respectively are mixed and stirred. Then acid solution is poured inside Graphite beaker and stirring is maintained at 60 ℃ for 16hours. After that a slightly dense purple shading liquid is obtained. That liquid is poured inside a 400ml DI-ice containing beaker. After that reaction is terminated by dropwise adding 5ml of Hydrogen-peroxide in it. The solution surface color turns yellowish. The solution is then allowed to settle down for seven days. After that the supernatant liquid is washed out. Same washing cycle was repeated. After that washing of supernatant via centrifuge is pursued. For this purpose, the liquid was washed by centrifuging at rpm of 6500 for one hour with 100ml of DI water, 100ml of 30% HCl and 100 ml of ethanol. This process is repeated. After removal of supernatant liquid again and again, the solution was washed by 100ml ethanol and 100ml DI water repeatedly to attain pH of 6. In the end relatively dense brownish shading liquid was attained. This liquid was then filtered out via filtration assembly through Nylon filter paper and DI water. Finally, the paste was dried in vacuum oven at 60 ℃ for 24hours. Dark colored sticky Graphite oxide powder was obtained. Graphite Oxide powder was then sonicated then for 3hours to separate out the graphite layers from each other forming Graphene Oxid [34].

The steps are described below schematically.

Step 1: Mixing and Stirring

3g of Graphite+ 18g $KMnO_4$ | H_2SO_4:H_3PO_4=360ml:40ml

Step 2: Exfoliation

Acids + $KMnO_4$ mixture | Stir on Hotplate at 60 ℃ for 16h

Step 3: Exothermic reaction

Purple liquid into 400ml Ice | Add dropwise 5ml H_2O_2

Step 4: Termination

Surface colour turns Yellowish | Settling down for 7 days

Step 5: Washing

Remove excess liquid from top | Repeat last washing step

Step 6: Centrifugation

2x250ml Vials; 40mins at 6500rpm | 100ml HCl↔Ethanol swap

Step 7: Cleaning

After every wash, stir and weigh | Repeat till pH~6

Step 8: Filtration

Residue into filtration assembly | Nylon filter of 45µm sieve size

Step 9: Sonication

100ml Ethanol↔DI water repeatedly | Sonicate for 3hours

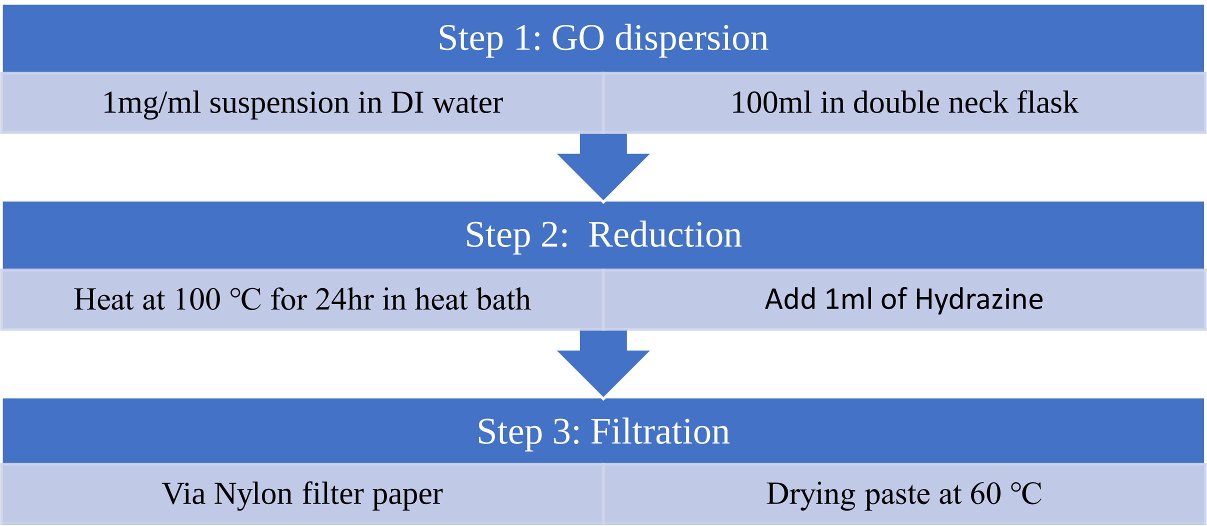
Step 10: Drying

Vacuum drying at 60 ℃ for 24h | Dark brownish powder in petridish

3.3.2 Reduced GO synthesis

GO was reduced under condensation chamber inside a heat bath. Graphene oxide was dispersed in DI water having concentration of 1mg/ml. Then the 100ml solution was poured into a double neck flask inside a heat bath. 1ml Hydrazine was added to the flask and the condensation chamber was closed. Then we heated the solution to 100 °C for 24hours. After that black precipitates were obtained. These were then filtered via Vacuum suction thorough Nylon filter paper, DI water and Ethanol in a filtration assembly. Lastly the paste obtained was dried in a vacuum oven for 24hours at 60 °C [36].

GO reduction procedure is described in schematic [37-39].



3.3.3 Nylon fabric cladding

Nylon fabric was washed by ethanol and dried before cladding. Graphene Oxide was dissolved in DI water in concentrations of 1mg/ml, 2mg/ml and 2.5mg/ml. Nylon fabric was dipped inside the GO solution for 10mins and then dried in PTFE mold at 70 °C for 12hours. After that GO cladded fabric was tested for electrical applications [40, 41]. Fig. 3.1 show picture of uncoated and GO cladded Nylon fabric.

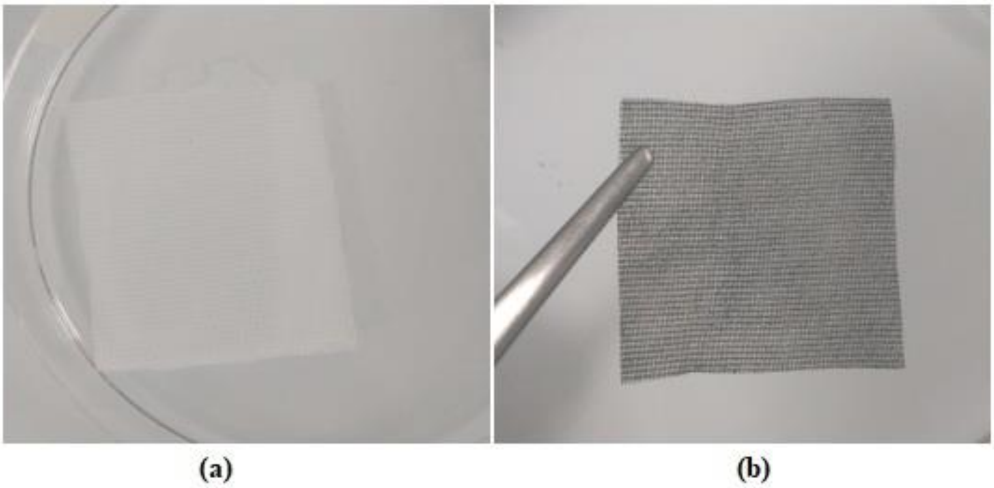


Figure 3.1: (a) pure Nylon fabric **(b)** GO cladded Nylon fabric

3.3.4 Reduction of GO coated Nylon textile

GO coated nylon fabric was then placed in reducing atmosphere with Hydrazine inside a petri dish for 2hours at 60 ℃. After that the sample is dried and then washed with ethanol [39]. Fig. 3.2 is showing GO and rGO cladded Nylon textile fabric.

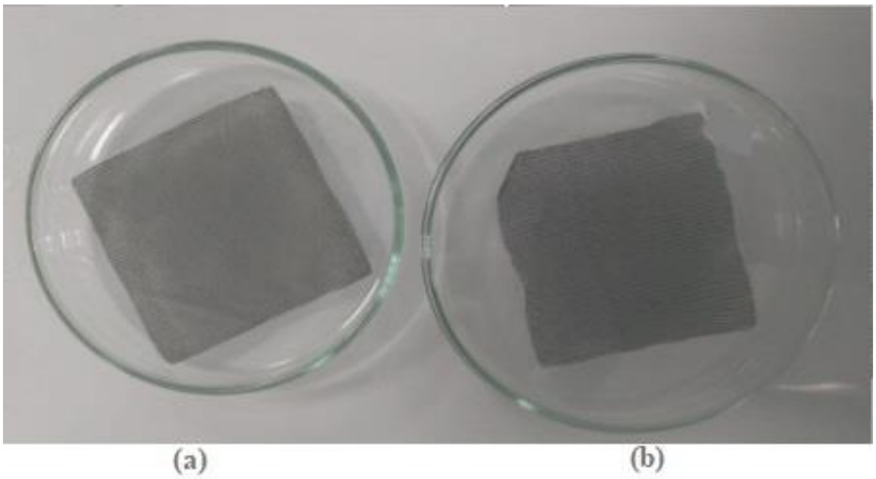


Figure 3.2: (a) GO coated textile fabric **(b)** Graphene cladded textile

3.3.5 PMMA based Nano-composite

The GO coated and rGO coated fabric were then solution casted in PMMA matrix in DMF solvent [42]. For this purpose, 2g PMMA was dissolved in 50ml DMF for 2hours at 300rpm over hot plate with stirring magnetic stirrer. After that the solution was poured into petri dish and textile was dipped inside the solution. The solution was dried in an oven at 80°C for 24 hours. The film was taken out of the petri dish. At last the Graphene cladded textile reinforced PMMA Nano-composite was formed. Fig. 3.3 is picture of final transparent PMMA based nano-composite.

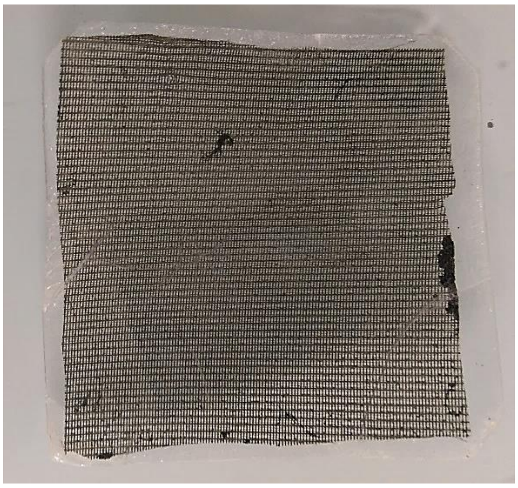


Figure 3.3: rGO cladded textile reinforced PMMA matrix

Chapter 4

Analysis Technique

4.1 Scanning electron microscope (SEM)

In this technique, fine beam of electrons is focused over a specimen’s surface. These electrons interact with the sample surface and photons or electrons are knocked off from material’s surface in result. These knocked off electrons are then focused on the detector. The output from the detector modulates the brightness of cathode ray tube. For every point where the electron beams are focused and interact, it is plotted on consequent point on CRT and material’s image is produced. The electron-surface interaction causes the release of secondary electrons (SE), backscattered electrons (BSE) and X-rays. Common SEM mode for detection is via secondary electrons. Schematic for SEM is shown in Fig. These electrons are emitted from near the sample surface. So, a pronounced and clear image of sample is obtained. It can reveal sample detail even less than 1nm in size. Also, elastic scattering of incident electrons also takes place and release back scattered electrons. They emerge from deeper locations as compared to secondary electrons. So, their resolution is comparatively low. Characteristic x-rays also emit from the atoms when an inner shell electron knocks off from its shell. We use SEM as it has easy sample preparation and we can figure our sample’s morphology, chemistry, crystallography and orientation of planes. Magnification of SEM can be controlled from 10 to 500,000 times [43].

SEM analysis was performed utilizing (JEOL-JSM- 6490LA) with operating voltage of 10kV-20 kV, spot size of 35-60, and working distance of 10mm. Fig. 4.2 shows view of the machine from the front and Fig. 4.1 show schematic mechanism of SEM.

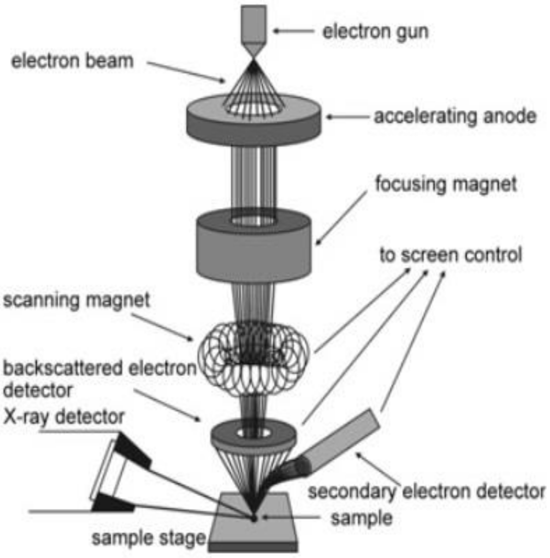


Figure 4.1: Schematic for SEM working mechanism [44]

Figure 4.2: JSM 6490LA SEM present in SCME, NUST, Islamabad.

4.2 X-ray diffraction (XRD)

It is an analysis procedure which is used to determine the crystalline phase of the material. It provides fingerprints of Bragg's reflections of crystalline materials. Diffractometer consist of 3 basic elements. A cathode tube, sample holder and x-ray detector. X-rays are produced by heating filament element which accelerates electrons towards a target which collide with target material with electrons. Crystal is composed of layers and planes. So, x-ray which has wavelength having like these planes is reflected that that angle of incidence is equal to angle of reflection. “Diffraction” takes place and it can be described as by Bragg’s Law:

$$2d\sin\theta = n\lambda$$

Bragg’s law fulfillment leads to constructive interference and “Bragg’s reflections” will be picked up by the detector. These reflections positions tell us about inter-layer spacing. X-ray diffraction tells us about the phase, crystallinity and sample purity. By this technique one can also determine lattice mismatch, dislocations and unit cell dimensions. Fig. 4.3 show schematic for XRD. X-ray diffractions were performed by STOE diffractometer at SCME-NUST.

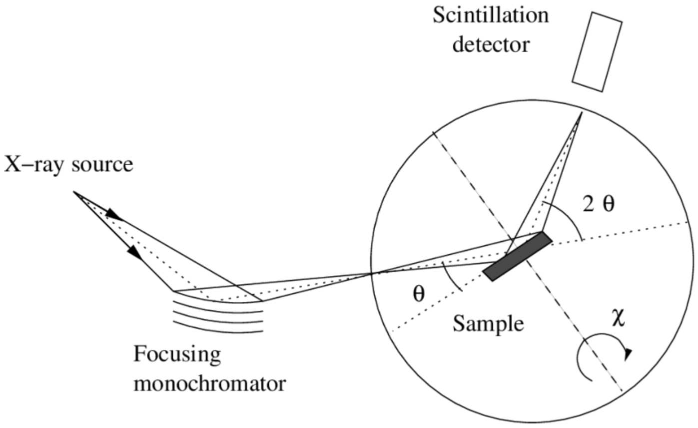


Figure 4.3: Schematic of a typical XRD [45]

4.3 UV-Vis spectroscopy

This technique is used to determine the optical properties of materials. Absorbance, transmittance and resistance properties can be studied by this technique. It measures the absorption of light beam after it passes from a sample. This absorption can be of single wavelength or of extended spectral range.

These radiations are made to fall on the sample. A spectrometer is present in this instrument which records this absorption by a sample at different wavelengths. The resultant plot of absorbance (A) versus wavelength (λ) is obtained which is called a spectrum. The mechanism is elaborated in Fig.4.5.

Absorption and % Transmission data was recorded using “Jenway 7315 UV-Vis spectrometer” in wavelength range of 200-800 nm of electromagnetic spectrum present at SCME- NUST as shown in Fig 4.4.

Figure 4.4: Jenway 7315 UV-Vis spectrometer present in SCME, NUST, Islamabad.

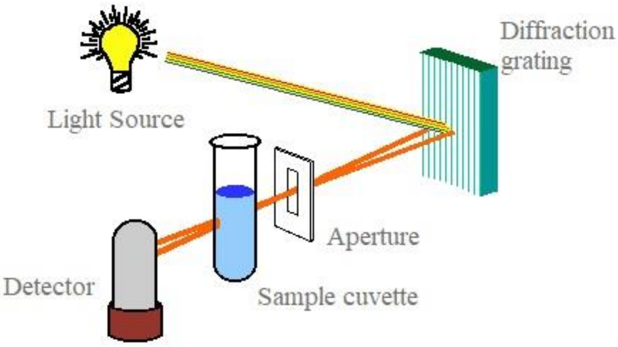


Figure 4.5: Schematic of a typical UV-Vis spectrometer [46]

4.4 Impedance analysis

The dielectric behavior was analyzed at room temperate using impedance analyzer (Wayne Kerr 6500B). The dissipation D and capacitance C were obtained in the frequency range of 100Hz-5MHz. Circular cross sectional area samples were prepared of 13mm diameter. Thickness was of 0.133mm for GO coated fabric, 125mm for pure Nylon fabric and 128mm for reduced GO coated fabric [37, 47].

Chapter 5

Results and Discussion

5.1 X-ray Diffraction patterns

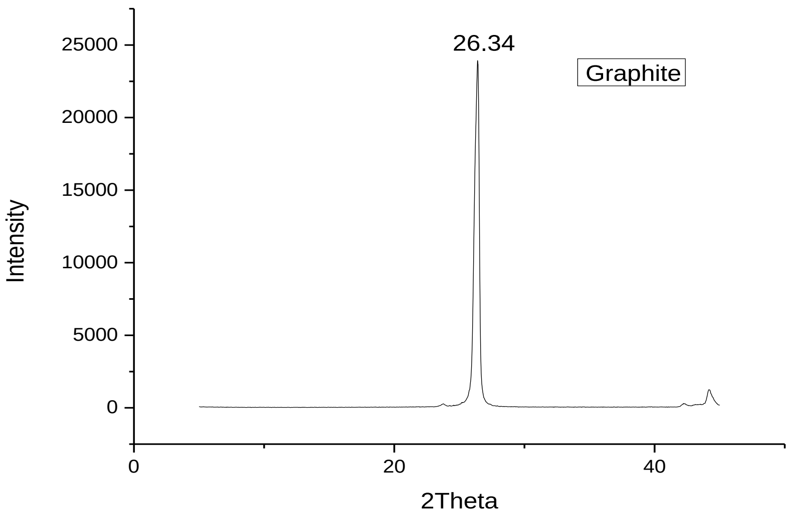


Figure 5.1: XRD graph for Graphite powder

Sharp peak at 26.34˚ indicate crystalline structure of Graphite powder in Fig. 5.1. It relates to d-spacing of around 3.38Å. Intensity of the peak depicts the hexagonal closepacking crystalline structure. Nano-rystallite size for Graphite powder is 19.85nm.

The relatively broad peak for graphene oxide Fig. 5.2 represents amorphous trend in crystalline structure of Graphite. However, the angle 10.54˚ clearly verifies the presence of graphene oxide which corresponds to d-spacing of 8.38 Å. GO have hydrophilic oxygenated graphene sheets that have functional oxygen groups on edges and base planes.

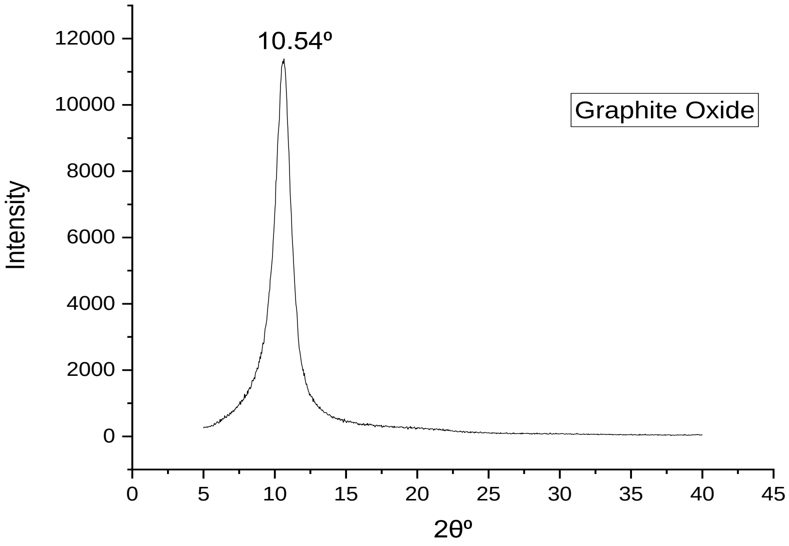


Figure 5.2: XRD graph for GO

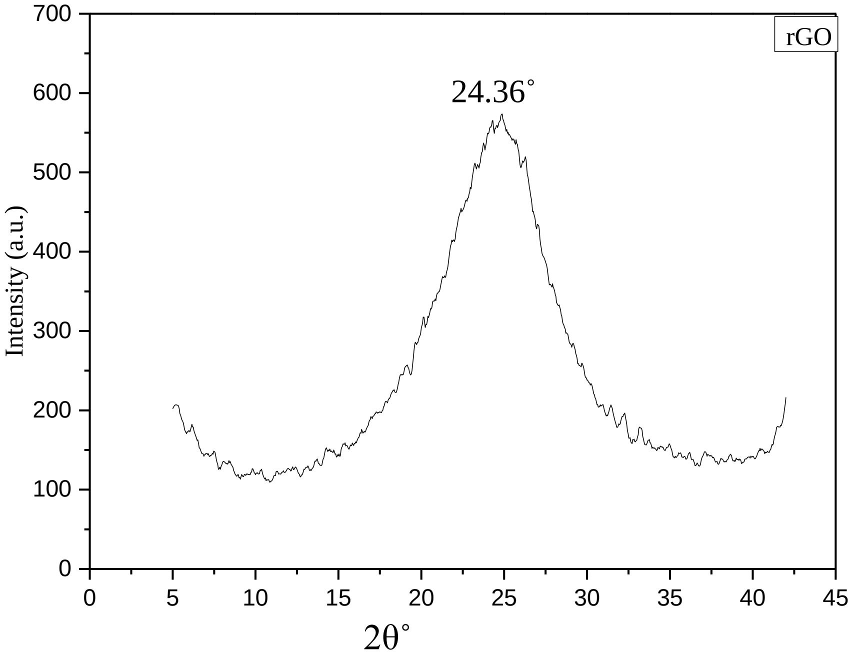


Figure 5.3: XRD graph for reduced Graphene Oxide

The rGO XRD graph in Fig. 5.3 presents amorphous structure of Graphene starting from 19˚ to 28˚ with highest center at 24.36˚ corresponding to d-spacing of 3.66Å. The transition froom Graphite [002] plane to GO [002] plane and then rGO validates Graphene synthesis. Nano-crystallite average size for rGO is 24.37nm.

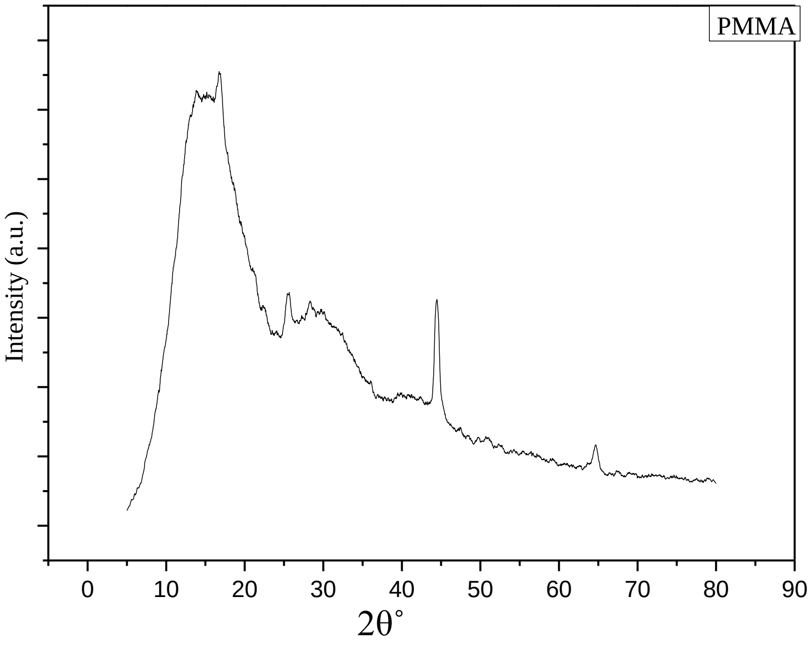


Figure 5.4: XRD graph for PMMA

XRD Graph for PMMA represents amorphous nature of the polymer in Fig. 5.4. The peak at 44˚ corresponds to sample holder.

5.2 Scanning Electron Micrographs

SEM micrographs for Graphene Oxide show stacked sheets of graphene Oxide over one another in Figures 5.5,5.6, 5.7 and 5.8. GO sheets are accumulating due to higher concentration of GO in drop cast sample prepared for SEM. GO micrographs show vast surface area of GO sheets. Step like morphology represents stacking of sheets over one another while having minor wrinkles as well.

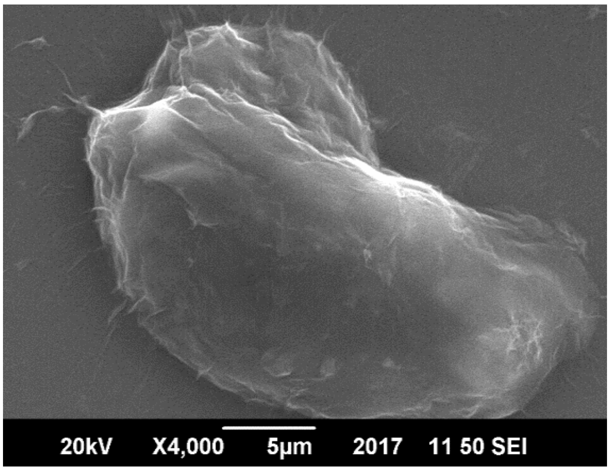


Figure 5.5: SEM micrograph for GO at X4000

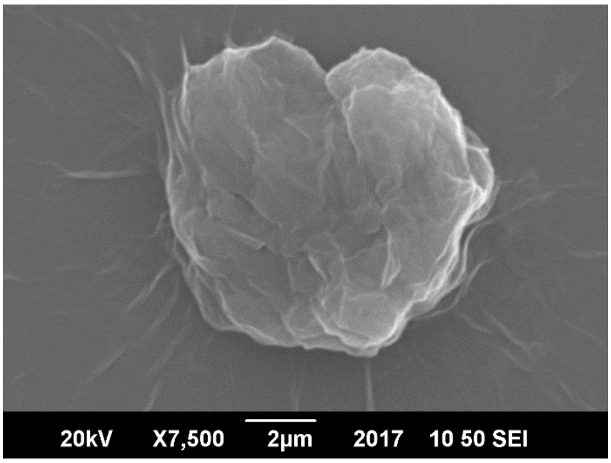


Figure 5.6: SEM micrograph for GO at X7500

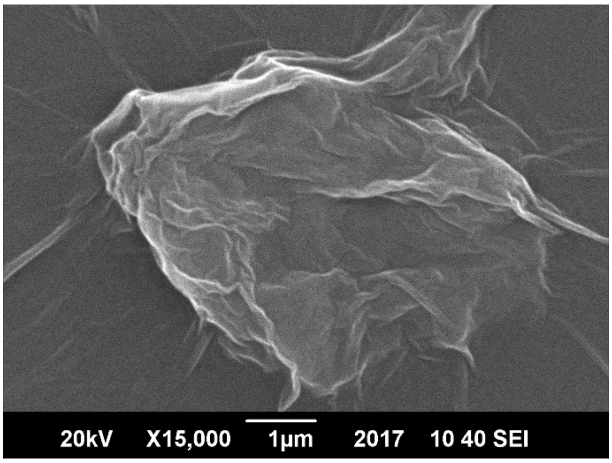


Figure 5.7: SEM micrograph for GO at X15000

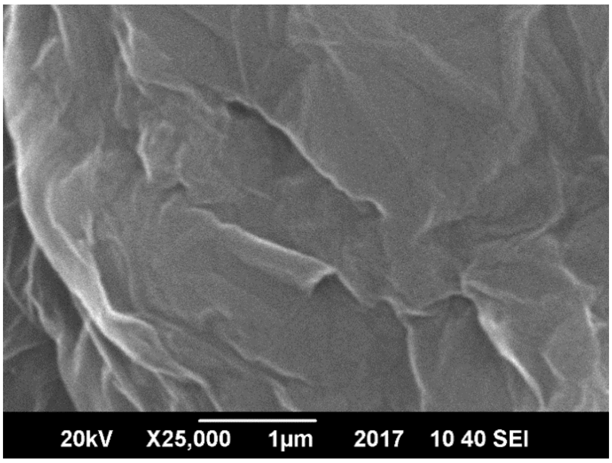


Figure 5.8: SEM micrograph for GO at X25000

SEM micrographs for rGO show sheet like morphology in Figures 5.9, 5.10, 5.11 and 5.12. The sheets are accumulated over one another due to lesser stable dispersion in solvent. Wrinkled single sheets are visible at higher magnification as well. rGO sheets tend to wrinkle due to higher surface area.

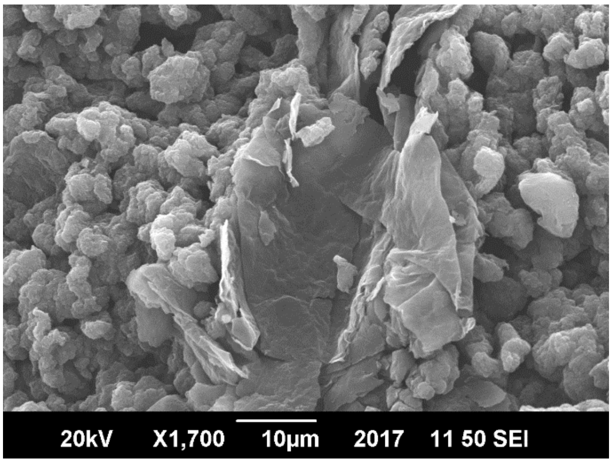


Figure 5.9: SEM micrograph for rGO sheets at X1700

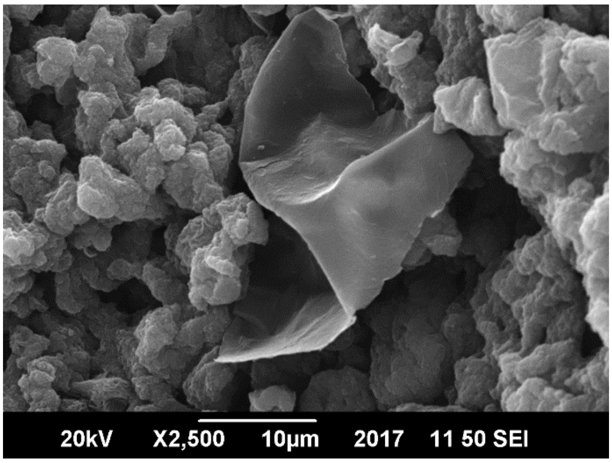


Figure 5.10: SEM micrograph for rGO sheets at X2500

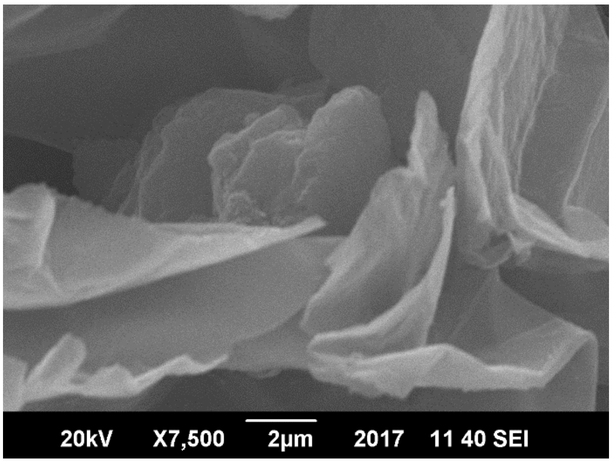


Figure 5.11: SEM micrograph for rGO sheets at X7500

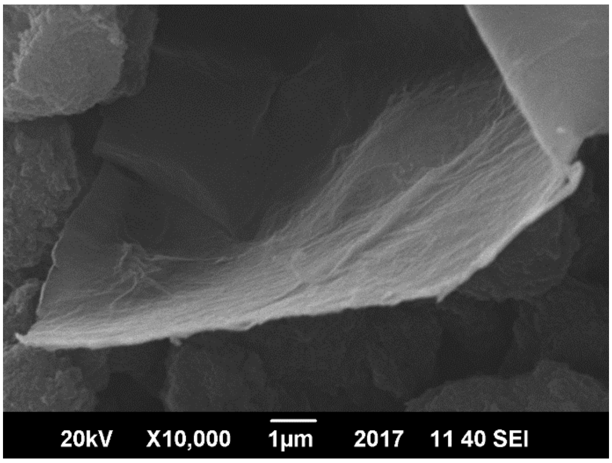


Figure 5.12: SEM micrograph for rGO sheets at X10000

SEM graphs for Nylon fabric show woven nylon fibers into fine fabric in Figures 5.13, 5.14, 5.15 and 5.16. Woven nylon fibers with average fibril diameter of 46.8µm. The surface of Nylon fabric is readily available for coating.

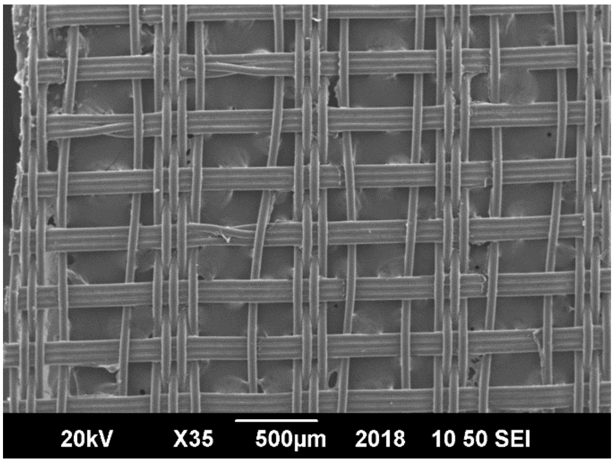


Figure 5.13: SEM micrograph for Nylon textile at X35

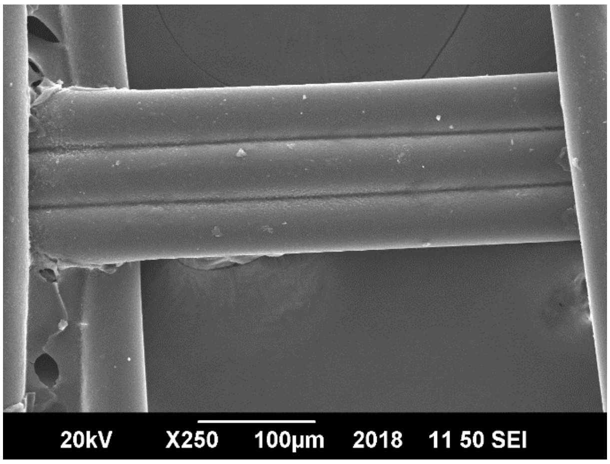


Figure 5.14: SEM graph for Nylon textile at X250

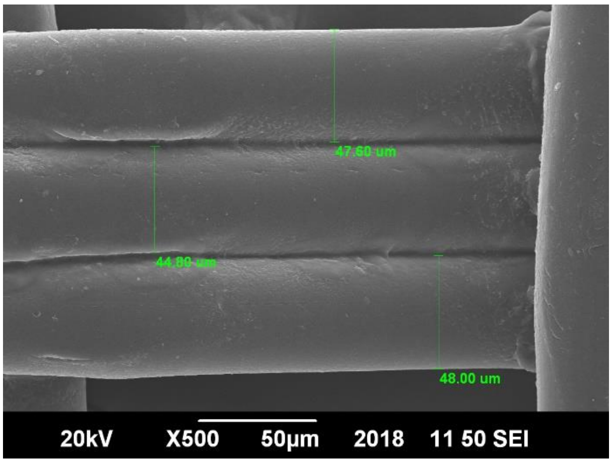


Figure 5.15: SEM micrograph for Nylon textile fabric at X500

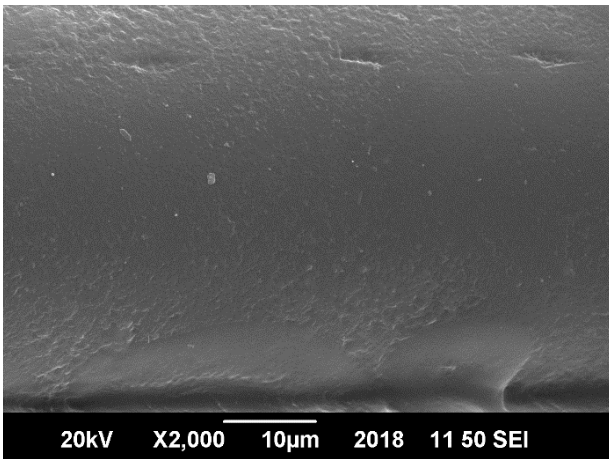


Figure 5.16: SEM micrograph for Nylon fabric at X2000

The micrographs vividly explain the diverse morphology of Nylon, GO and rGO respectively. The beautifully scattered rGO sheets represent its unique properties. The agglomeration of sheets is because of wrinkle effect in the Graphene sheets.. Now lets examine the after effects of the GO coating on Nylon fabric.

SEM micrographs for GO coated Nylon textile show good adhesion of GO sheets to Nylon fabric strands in Figures 5.17, 5.18, 5.19 and 5.20. GO sheets have quite finely cladded the Nylon fibers. GO sheets have cladded the strands multiple times in some places.

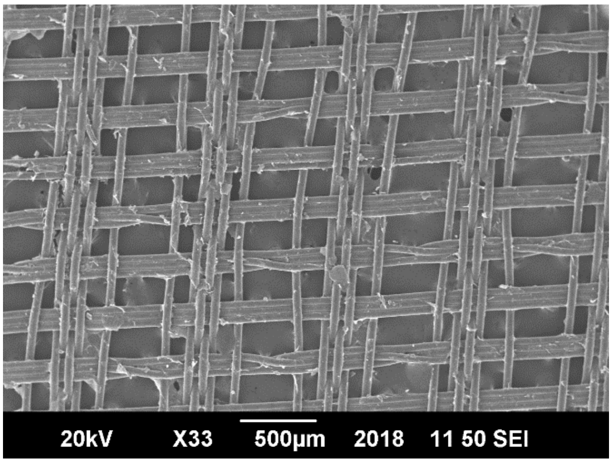


Figure 5.17: SEM micrograph for GO coated Nylon fabric at 33X

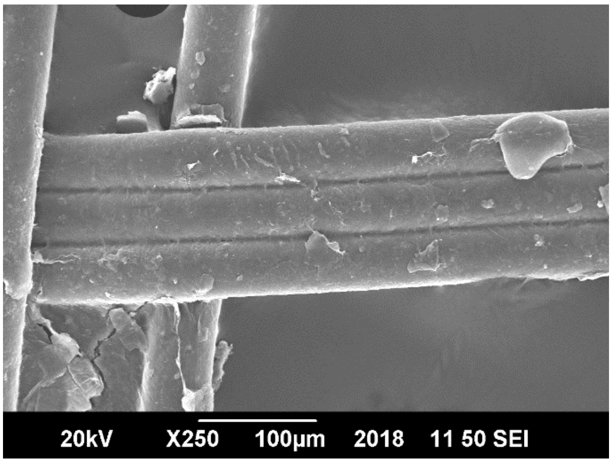


Figure 5.18: SEM micrograph for GO coated Nylon fabric at 250X

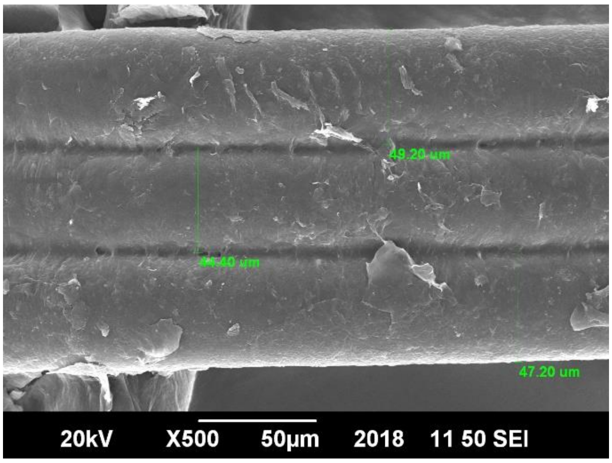


Figure 5.19: SEM micrograph for GO coated Nylon fabric at 500X

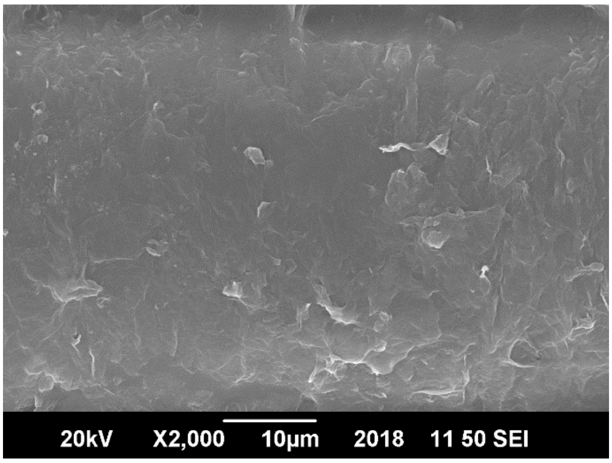


Figure 5.20: SEM micrograph for GO coated Nylon fabric at 2000X

SEM micrographs after reduction of GO cladded Nylon fabric show activity of reducing agent on the surface of Nylon strands in Figures 5.21, 5.22, 5.23 and 5.24. Upper layer of GO coating is erdoded at some places. The abraded upper layer of rGO sheets at the surface is quite visible.

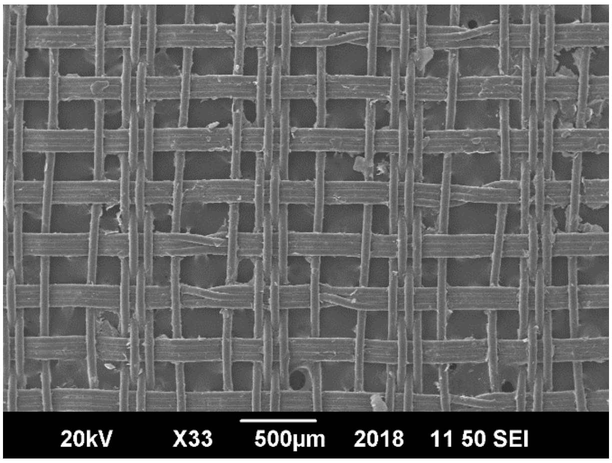


Figure 5.21: SEM micrograph for rGO coated Nylon fabric at X33

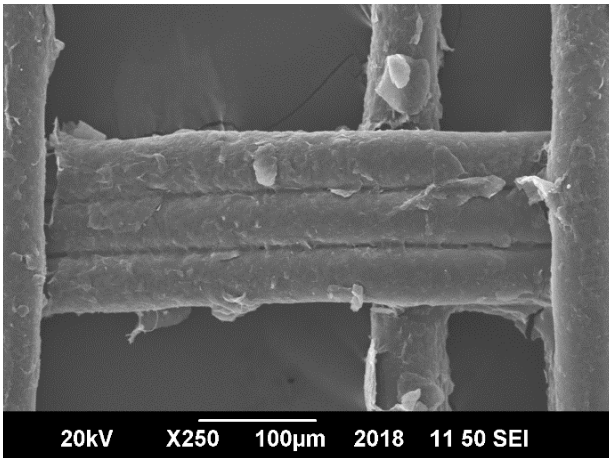


Figure 5.22: SEM micrograph for rGO coated Nylon fabric at X250

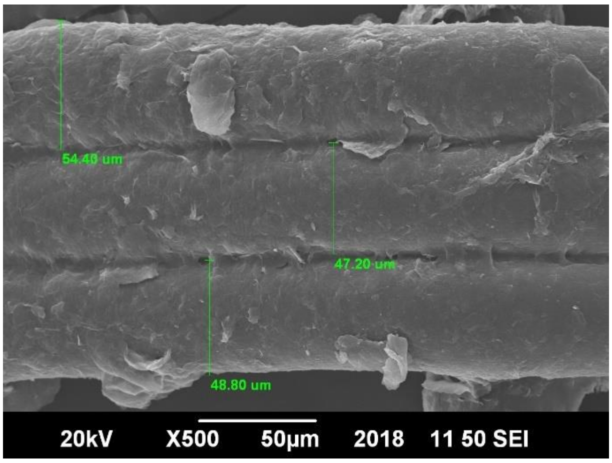


Figure 5.23: SEM for rGO coated Nylon fabric at X500

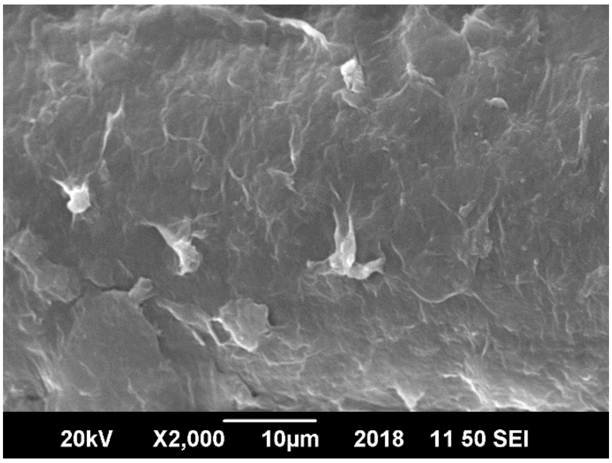


Figure 5.24: SEM for rGO coated Nylon fabric at X2000

5.3 Fourier Transform Infrared Spectroscopy [FT-IR]

FT-IR examination provides information about functional groups attached in Fig. 5.25. The broad peaks in the range from 3700 to 3000 cm^{-1} are representative of stretching vibrations of hydroxyl group, where the hydroxyl groups might be from retained water particles or phenolic OH or OH from carboxylic group. Due to oxidation of Graphite particles large number of oxygen functional groups are present. Accordingly the amount of water retained will decrease because of less polar character of rGO. The two little peaks close to ~2927 cm^{-1} and ~2868 cm^{-1} can be seen in GO quite easily which are present due to the OH groups of dimeric COOH groups and intra-sub-atomic O-H stretching of alcohols individually. The peak near 1603 cm^{-1} in GO and 1614 cm^{-1} in rGO is owing to O-H bending vibrations of water. The difference in the peak position for the two is accepted because of the difference in the level of hydrogen holding and association. Peak at 1711 cm^{-1} is related to the C=O stretching vibrations. Other peaks corresponding to C-OH (1375 cm^{-1}), and C-O (1039 cm^{-1}) can be clearly seen.

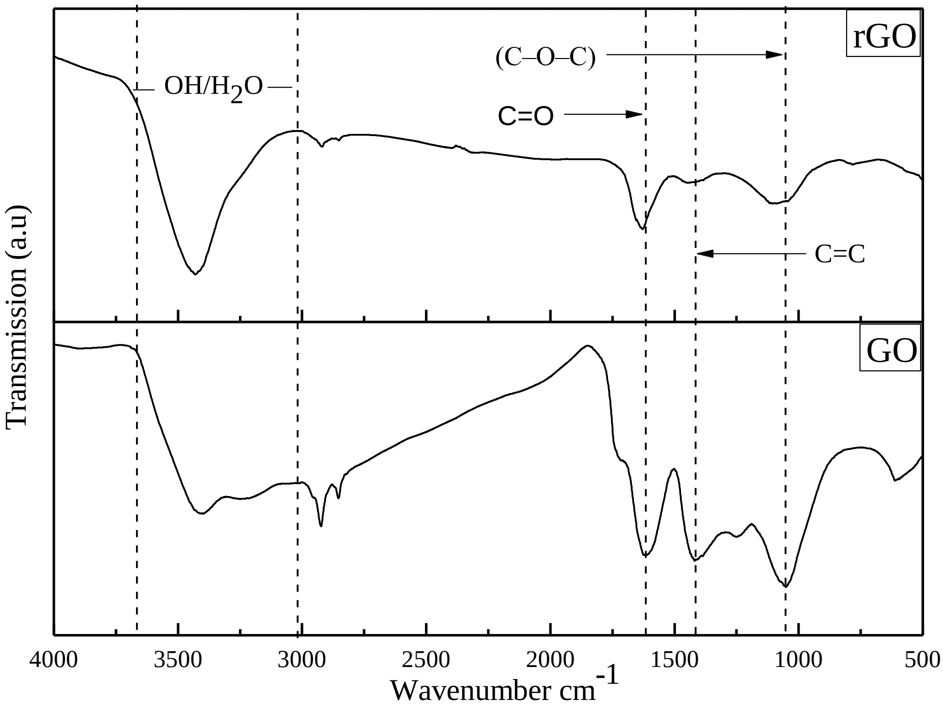


Figure 5.25: FTIR spectrograph for GO and rGO

5.4 UV-Visible range spectroscopy

The spectra for GO maintains a big absorption peak between 225nm-233 nm with a shoulder peak between 270nm-316 nm as visible from Fig. 5.26. The absorption peak at ~229nm, relates to π-π* exchange of aromatic C-C bonds, and the shoulder peak is because of n-π* exchange of C=O [48].

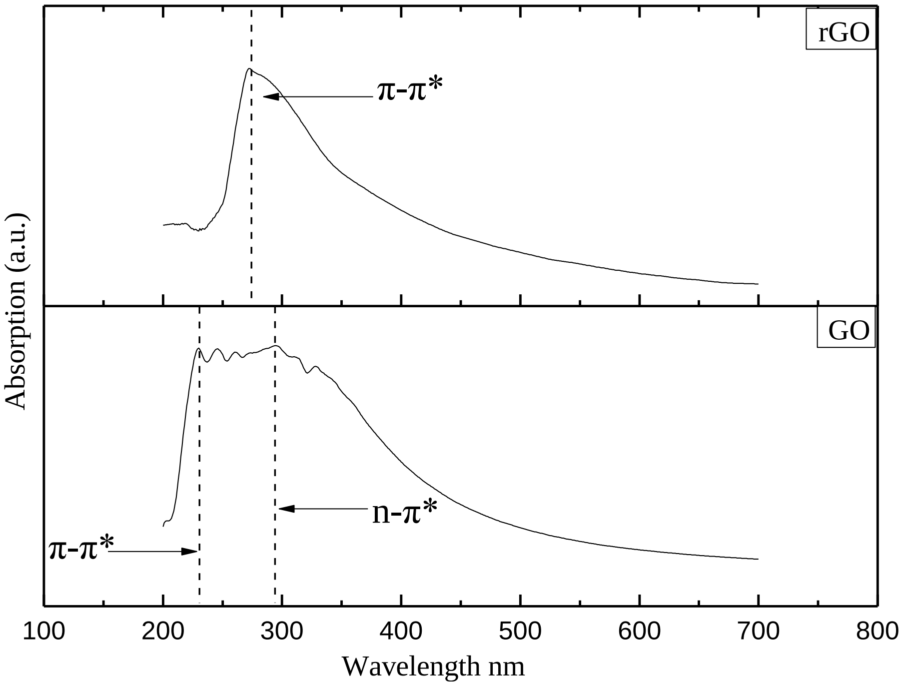


Figure 5.26: UV-Vis spectrograph for GO

5.5 Dielectric testing using Impedance analyzer

The ac conductivity (σac) dependant on frequency is calculated using the following relation from the dielectric data

$$\boldsymbol{\sigma=2\pi f\varepsilon_{\circ}\varepsilon^*}$$

The rise in values of ac conductivity is due to enhanced charge carriers mobility.

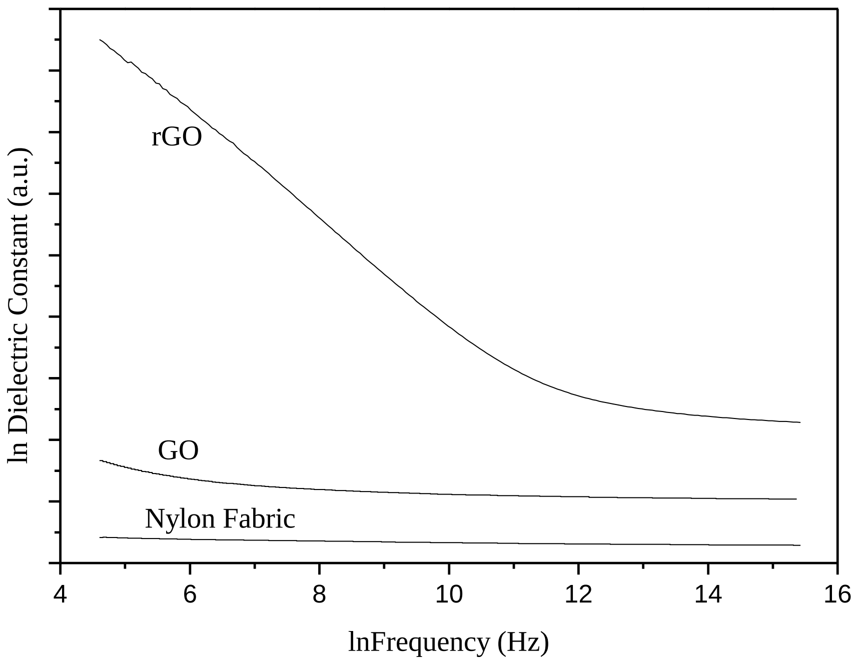


Figure 5.27: ln dielectric constant Vs ln frequency graph for rGO coated, GO coated and pure Nylon Fabric

Maximum value of dielectric constant for uncoated fabric is 1.91 x10^{-6} , for GO cladded fabric it is 5.1 x10^{-6} and for rGO cladded fabric is 5.51 x10^{-1} as shown in Fig.5.27.

Maximum dielectric loss values for Fabric, GO cladded fabric and rGO cladded fabric are noted as 2.04x10^{-7}, 4.69 x10^{-6} and 104 respectively from Fig. 5.28. Clearly, rGO exhibited quite higher values for dielectric loss and Dielectric constant.

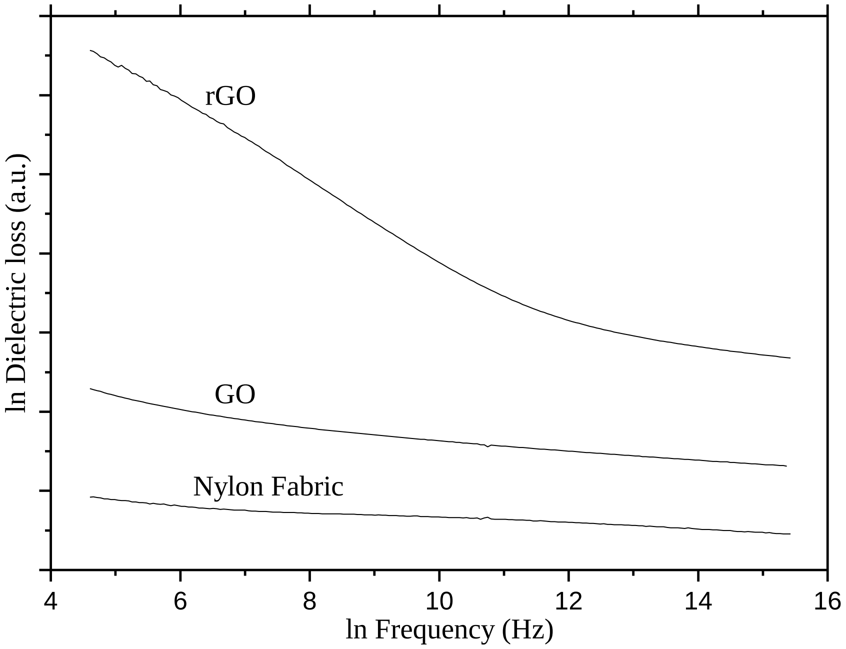


Figure 5.28: ln dielectric loss Vs ln frequency graph for rGO coated, GO coated and Pure Nylon Fabric

Maximum capacitance values for Fabric, GO and rGO cladded fabric are 1.79 x 10^{-11}F, 5.03 x10^{-11}F and 5.06 x10^{-6}F respectively. Capacitance is improved 10^5 times in rGO cladded fabric. Fig. 5.29 show the trend for capacitance.

Conductivity values for PMMA are in order of 10^{-14}Scm^{-1}. Maximum AC conductivity for uncoated nylon fabric, for GO coated fabric and rGO cladded fabric is noted as 1.13 x 10^{-13}Scm^{-1}, 9.34 x 10^{-10}Scm^{-1} and 5.84 x 10^{-4} Scm^{-1} respectively. Fig. 5.30 elaborate comparative graphical trends for conductivity.

All the dielectric values Dielectric constant, Dielectric loss, Capacitance and AC conductivity were enhanced for rGO coated Nylon fabric due to higher electron mobility of rGO sheets.

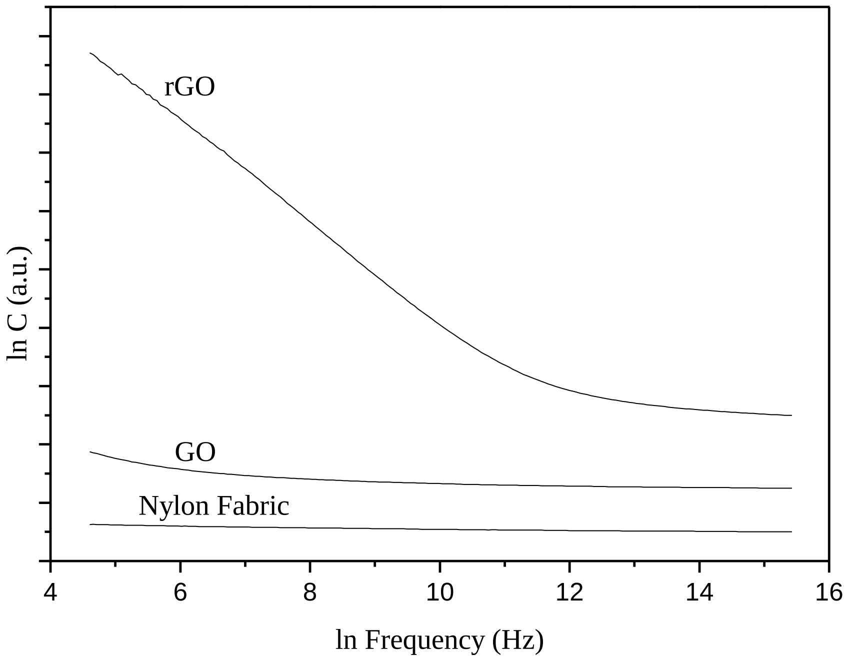


Figure 5.29: ln Capacitance Vs ln Frequency graph for rGO coated, GO coated and Pure Nylon Fabric

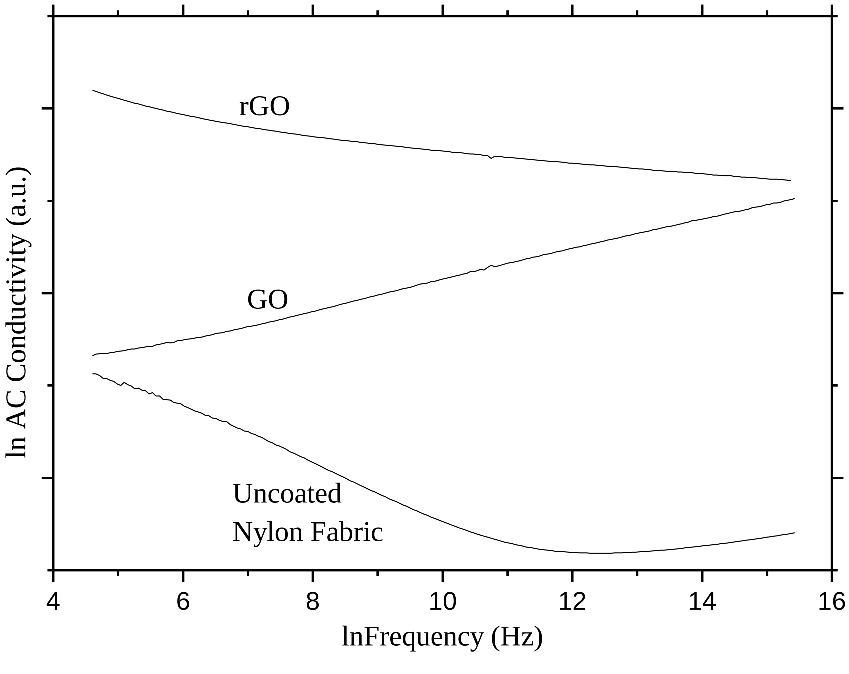


Figure 5.30: ln AC conductivity Vs ln Frequency graph for rGO coated, GO coated and Pure Nylon Fabric

Conclusions

Intrinsicallay conductive and transparent film of Graphene coated Nylon fabric reinforced PMMA matrix nano-composite was made. GO and rGO were synthesized from graphite via improved hummers method. Materials Characterization was done by XRD, FTIR and UV-Vis spectroscopy and supported the analysis with SEM. Then Nylon fabric was coated with GO and was reduced chemically with hydrazine. Morphological, electrical and chemical changes were verified by SEM, FTIR and impedance analysis. Average Fiber size of Nylon fabric was 46µm. Which was increased to 46.8µm after GO coating analysed by SEM. The final GO and rGO coated samples were tested for Dielectric constant, Dielectric loss, capacitance and AC conductivity values. Maximum dielectric loss values for Fabric, GO cladded fabric and rGO cladded fabric are noted as 2.04x10^{-7}, 4.69 x10^{-6} and 104 respectively. Dielectric constant value for Fabric is 1.91 x10^{-6} , for GO cladded fabric is 5.1 x10^{-6} and for rGO cladded fabric it is 5.51 x10^{-1}. Clearly, rGO exhibited quite higher values for dielectric loss and Dielectric constant. Capacitance values for Fabric, GO and rGO cladded fabric are 1.79 x 10^{-11}F, 5.03 x10^{-11}F and 5.06 x10^{-6}F respectively. Capacitance is improved 100000 times in rGO cladded fabric. The AC conductivity for rGO coated Nylon fabric was 5.87x10^{-2} Scm^{-1} which is 10^{9} folds higher than pure Nylon and PMMA AC conductivity. Finally, Nylon fabric was embedded in PMMA matrix using solution casting method, resulting in noval type of nano-composite of PMMA.

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