

FRP Composites Nano-Modified with Graphene Derivatives for Enhanced Mechanical Properties

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STRUCTURAL ENGINEERING

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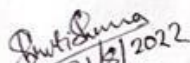
Declaration

I hereby declare that the research work entitled “FRP composites nano-modified with graphene derivatives for enhanced mechanical properties”, is an authentic record of my work carried out as requirements for the partial fulfilment for the award of degree of **Master of Engineering** in Structural Engineering at **Thapar Institute of Engineering & Technology, Patiala** under the supervision of **Dr. Shruti Sharma**, Professor, Civil Engineering Department, Thapar Institute of Engineering & Technology, Patiala & **Dr. Sandeep Kumar Sharma**, Associate Professor, Mechanical Engineering Department, Thapar Institute of Engineering & Technology, Patiala. No part of the matter embodied in this report has been submitted elsewhere.

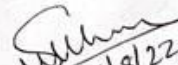
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ABSTRACT

The study influence of the graphene oxide (GO) inclusion on the mechanical properties of carbon fiber reinforced polymer (CFRP) hybrid composite and glass fiber reinforced polymer (GFRP) is reported. Different wt.% content of GO used for development of epoxy matrix and GFRP/CFRP hybrid composite was prepared using VARIM. GO was synthesized from graphite powder and was mixed with epoxy resin. Three different GO/rGO concentration of 0.1, 0.2, and 0.3 phr were incorporated in the GFRP and CFRP laminates. The overall performance of GFRP/CFRP laminates in which the epoxy is nano reinforced with graphene derivatives is to be assessed and the results have significantly shown that inclusion of graphene derivatives improves the mechanical properties of the composites. The performance includes tensile testing, flexural strength, and impact strength.

The epoxy used in these laminates for repairing and retrofitting of the structure is of very brittle nature hence, to make the epoxy ductile nano particles, nano clay, graphene derivatives were nano reinforced on the composites. In our study, we have primarily focused on graphene derivatives i.e., graphene oxide (GO) and reduced graphene oxide (rGO) fabricated on glass and carbon fibers. It was observed that GO is improving and enhancing the strength better than rGO because of more functional groups present in their composition.

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1

INTRODUCTION

1.1 BACKGROUND AND MOTIVATION

Advanced composite materials have excellent properties like high strength, stiffness and durability, appropriate strength-to-weight ratio, the main reason why they are actively used in the industrial fields like aerospace, marine and automotive industries. The various use of FRP polymers in various applications is represented in Table 1.1. These materials possess low thermal coefficient and act as solid resistance to corrosion. As, many difficulties are associated with the failure of civil buildings or infrastructure, engineers and the industry are moving forward with these advanced composites as a material for repairing, strengthening, or retrofitting of the structures. Various studies have shown the strengthening of RC structures using the Externally Bonded Reinforcement (EBR) technique. The bonding of Fiber reinforced polymer (FRP) with the concrete structures have improved the flexural and shear capacity of the structural elements, hence improving the performance of structure. The main reason for the deteriorated structural performance is the combined effect of various service loads and environmental attacks. The damage accumulation to infrastructure majorly occurs due to these physical attacks. FRP reinforcement has great strength and the ability to repair the concrete structures for both seismic and corrosion protection. Recently, Glass fiber reinforced polymer (GFRP) and Carbon fiber reinforced polymer (CFRP) has been used for structure repair and retrofitting and maintenance of the structures.

Table 1.1. Applications of fiber reinforced composites in various industries.

S.no	Industry	Applications
1.	Aircraft and Military	Baggage doors, Vertical fins, Vertical and Horizontal stabilizers, Rotor blades, Outer flaps, Radom, Tail cone etc.

2.	Space Industry	Payload bay door, Remote manipulator arm, Support structures for smaller components(solar arrays, antennas, mirror substrates) etc.
3.	Automotive Industry	Hood or door panels, Radiator support, Bumper beams, Roof and door frames, Engine valve cover, Timing chain covers etc.
4.	Marine Industry	Boat hulls, Decks, Frames, Masts, Bulkheads, Propulsion shafts etc.
5.	Sporting Goods Industry	Bicycle frames for racing, Tennis rackets, Hockey sticks, Arrows, Javelins, Athletic shoe soles and heels etc.

FRP in the combination of glass or carbon fibers bonded together in the matrix of epoxy, polyester, and vinyl ester. These fibers uphold the major load in FRP. FRP products can be in various forms such as sheets, laminates for structure rehabilitation. Although they are used in various applications, there is obstacle that hinders their strength and that is the brittle nature of epoxy.

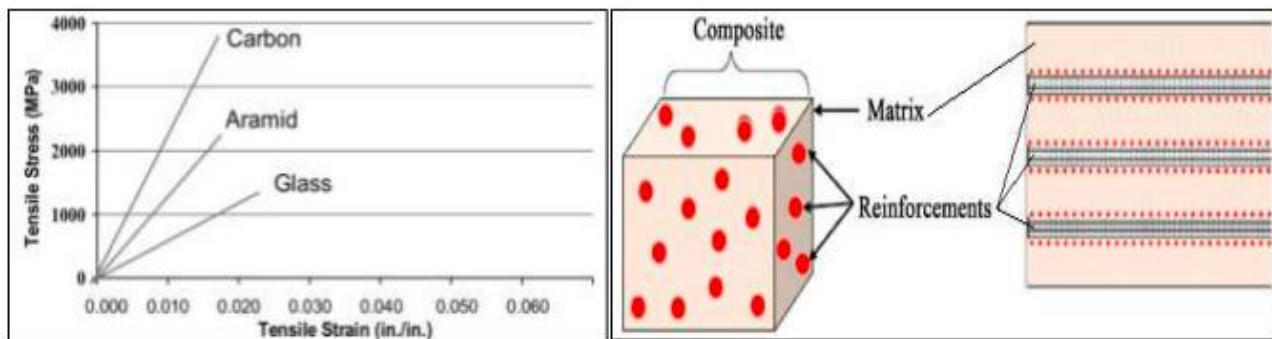


Figure 1.1: The matrix (Ashik et al., 2015)

Several researchers have experimented with the use of fiber reinforced polymer (FRP) composite materials that are externally applied to strengthen strengthened concrete buildings like beams, slabs, and columns. Because of its high strength-to-weight ratio and corrosion resistance, retrofitting or strengthening reinforced concrete columns with FRP composites is chosen over other materials like

steel. For many years, several investigations on FRP-equipped or strengthened concrete columns have been conducted. Carbon fiber reinforced polymers (CFRP) are used as laminates to improve the strength of the structural elements like beams, and columns in the buildings. Due to its low thermal expansion carbon fiber, they are preferred in steel road bridges. The foundations of civil engineering structures transmit loads from the upper levels to the lower levels and then to the soil via a column, which is one of the basic elements. Environmental effects or fatigue of its constituent materials may cause deterioration in a column during its service life, reducing the column's strength. In place of destruction and replacing of columns, retrofitting, or strengthening may be used to preserve the columns. GFRP/CFRP plays a vital role in engineering material as they are used in various applications. The mechanical property for these composites depends on positioning of fibers, fiber volume content and interaction between the fiber and matrix. These fibers exhibit various mechanical properties as well as physical properties that includes high strength-to-weight ratio, manufacturing flexibility etc.

Amongst various strengthening techniques FRP bonded to the external faces of damaged concrete elements have emerged as the most popular strengthening technique. (*Masoud et al. 2001, Bakis et al. 2002, B. Taljsten 2002, Balaguru et al. 2009*). It is primarily due to the various advantages of FRP like they can be easily bonded to concrete with minimal increase in dimensions and weight, their flexibility in wrapping, ease in handling, durability and improved mechanical properties over the conventional methods. Another advantage is their high durability in adverse environments like corrosive effect of acids and alkalis. Their high strength to weight ratio in addition to their ease of application makes them the most preferred material of repair.. The effectiveness of FRP in improving bending and shear capacities in flexural elements and confinement in compression elements is well established in literature. (*Mukherjee et al. 2004,2005,2009*). Another added advantage is that they create a barrier layer against ingress of moisture and other aggressive ions and hence have been used effectively to impede corrosion and offer protection to RC structures. It has been well documented in literature (*Badawi et al. 2005, Tamer et al. 2005, Gadve et al. 2009*). GFRP has been used as a passive protection since it applies confinement pressure to RC member affected by corrosion and prevents the dislodging of the concrete cover (*Sharma et al. 2015*). In addition to such passive protection, CFRP has been recommended against active protection due to its electrically conductive nature and can be used as anode for cathodic protection (*Sharma et al . 2021*).

Additionally, Carbon fiber reinforced polymers (CFRP) are used as laminates to improve the strength of the structural elements like beams, and columns in the buildings. Due to its low thermal

expansion carbon fibers are preferred in steel road bridges. Few investigations have checked the performance of columns under eccentric stresses, with the majority looking at column behavior under concentric stresses (*Li and Hadi 2003; Hadi 2006; Hadi 2007; Kumutha et al. 2007; Wang and Wu 2008*). *Dai et al. 2008* considered the performance of concrete structures confined with FRP composites of huge deformability formed by fibers made from recycled materials. *Wei et al. 2012* offered an investigational study on concrete columns with moderately depreciated strength confined with CFRP to evaluate the effect of a partial confinement. *Ashik et al. 2015* observed that when natural fibers reinforced in polymer composites depends upon orientation of fibers and characterization.

But for structural applications, FRP laminates also require high impact strength in addition to good static mechanical properties for safe operations. Epoxy, on polymerization, acquires a cross-linked structure. This typical structure obtains high modulus/strength, etc. in the material but causes increased brittleness, thereby reducing impact performance of FRP composites. Another main challenge in these FRP composites is the epoxy used in them as epoxy are very brittle in nature. They have relatively high curing time and handling difficulties. Hence to overcome this problem nano-fillers are introduced such as nano clay, carbon nanotubes (CNT) and graphene derivatives. FRP are reinforced with different nano-fillers to enhance their mechanical strength like tensile strength, flexural strength, and impact strength. This fiber reinforced polymer (FRP) is used frequently for repair and rehabilitated of reinforced structures (RC) that are affected by corrosion of steel bars.

Nanocomposites have shown that they may improve the qualities of the base material. Because of their numerous benefits including stronger mechanical strength and filling nanoscale space voids, the use of nanomaterials has become a trend. The mechanical, thermal, properties of these laminates have shown improvement when nanomaterials such as CNTs, graphene, GO, and graphene-based nanomaterials were used. *Sharma et al. 2015* studied the effect of ultrasonic guided waves for monitoring corrosion of FRP wrapped concrete structures. The ultrasonic guided waves were used to check the beginning and development of corrosion in reinforcing bars in concrete after they were wrapped with FRP laminates. The observations showed that there was significant protection with CFRP and GFRP laminates. The experiment was carried out at different days of exposure and result concluded than GFRP provided better protection than CFRP. *Sharma et al. 2021* studied the electric wave-based evaluation of CFRP protected RC structures exposed to corrosion. Concrete elements such as cylinders were wrapped with CGRP and GFRP laminates and the corrosion damaged elements were protected passively and actively. When the reinforced concrete was enfolded

by CFRP laminates it was known as passive protection and when cathodic protection with the application of wraps was used, it was known as active protection. The experiment of RC cylinders corroded was carried out in different intervals of time i.e., after 4,8,12 and 20 days of exposure and four different levels of damage were noted down. Further these specimens were examined using ultrasonic guided waves. From the results obtained with the variation in signal amplitudes it was concluded that active protection was the better option. The pull-out strength was also higher for the active protection as compared to the passive after 4,8,12 and 20 days of exposure. There was an increase of 15-20% in the pull-out strength in active protection with reference to the passive protection. *Garg et al. (2016)* studied the Glass fiber reinforced polymer (GFRP) modified with carbon nanotubes (CNT) of two types i.e., pristine nanotubes (PCNT) and amino-functionalized (ACNT) where it was observed that these nano tubes when reinforced in the composites has significant improvement in the mechanical properties. *Garg et al. (2016)* investigated the role of curing conditions and silanization of glass fibers on carbon nanotubes (CNTs) reinforced glass fiber epoxy composites. *Nanda et al. 2019* studied the enhancement in multi-scale filler reinforced epoxy nanocomposites for improved mechanical strength. These modified composites possess great improvement in tensile and flexural strength. The impact strength for these modified nanocomposites was enhanced by the addition of thermoplastic fibers as an additional filler. *Shelly et al. (2018)* studied the effect of nanomer clays to GFRPs for enhanced mechanical properties. Two different types of clays were nano reinforced in the GFRP laminates i.e., un-modified clay (Nanomer PGV) and an organically modified clay (Nanomer 1.28E) to improve the impact strength and fracture toughness. *Shelly et al. (2021)* investigated the addition of nano clay and ultra-high molecular weight polyethylene (UHMWPE) fibers to epoxy based GFRPs for improving the mechanical strength.

It is proposed in this work to enhance the properties of epoxy used to prepare GFRP/CFRP composites with graphene derivatives of GO and rGO for better mechanical strength and improving the ductility and toughness of these FRP composites (both GFRP and CFRP).

1.3 GRAPHENE DERIVATIVES

Nano-fillers were reinforced in the GFRP/CFRP laminates to enhance their mechanical properties. In our study we used graphene derivatives nano reinforced in glass and carbon sheets to improve their ductility and toughness. The graphene derivatives were incorporated in the epoxy based GFRP and CFRP sheets in a small amount (concentration incrementing from 0.1 to 0.3 phr) and sheets were prepared using Vacuum Assisted Resin Infusion Molding (VARIM).

The derivatives used in our study were:

- **Graphene oxide (GO)**. Graphene derivative achieved through a process called exfoliation (shaking or sonication).
- **Reduced graphene oxide (rGO)**. Graphene derivative produced by eliminating the oxygen content from the graphene oxide structure using chemical or thermal processes.

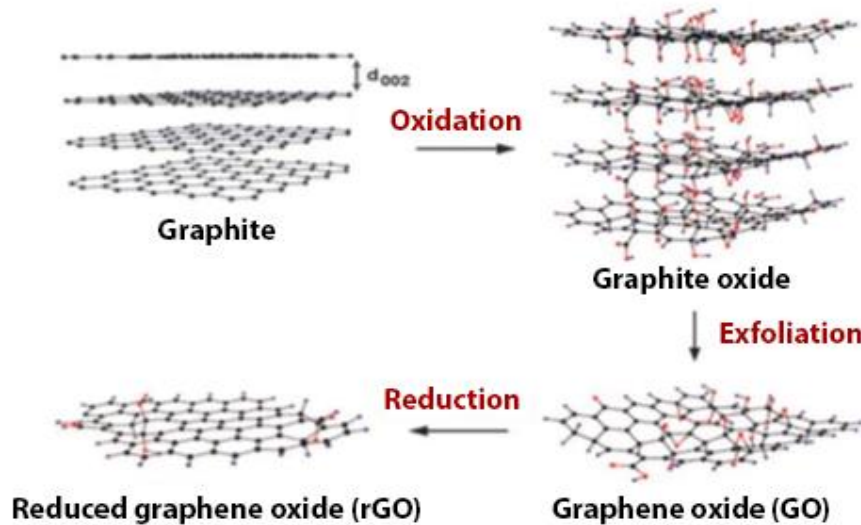


Figure 1.2: Extraction of Graphene Oxide from Graphite (Umer et al. 2015)

The main difference between these graphene derivatives lies essentially in chemical aspects, such as the level of oxygen: while the content of oxygen in graphene oxide ranges from 40 to 50% by weight, in the reduced graphene oxide that content is circa 40%.

GO is graphene with carbon-oxygen bonds and has functions such as hydroxyl, carbonyl, and carboxyl groups. Therefore, GO is a highly hydrophilic molecule and more important for electronics and energy-harvesting applications, this process provides a band gap (E.g.). Recently, the giant optical nonlinearities of graphene oxide (GO) have proven useful for several applications. Due to the presence of these functional groups, it has better bonding with the epoxy molecules.



Figure 1.2: (a) Graphene Oxide (b) Reduced graphene oxide used for the experiment

Graphene Oxide (GO) have better oxidative properties which helps to disperse in the epoxy resin whereas other nanomaterials do not possess these dispersive properties. *Chuah et al. 2012* have discussed that the degree of sonication was not dependent for GO dispersion in water but tend to deagglomerate as the repulsive forces provided by oxygenated functional groups at GO sheets was overwhelmed by van der Waals forces between the GO nanosheets. Addition of GO/rGO in appropriate amounts in the GFRP and CFRP composites have shown significant improvement in the mechanical strength. adverse effect on the workability due to its large surface area that tend to absorb more water molecule to get wet and a bulky lateral size with high capacity for water retention created due to cluster of GO nanosheets. The incorporation of small content of GO of about 1% by weight have improved the compressive strength. The functional groups present in the composition of GO and rGO helps in improving the fiber-matrix interactions. *Pathak et al. (2019)* also discussed in the research paper concluding that GO has more functionalities and OH groups present in their structure which enhances the bonding thereby improving the mechanical strength of the composites. GO bonds with the epoxy resin via H-bonding. The epoxy group of GO is converted to ether bond by CF hydroxyl group via substitution reaction.

The development of techniques for using graphene oxide in producing continuous, strong, tough fibers is motivated by their extraordinary properties. The toughness of these fibers has been

demonstrated to be higher than that of previously known materials in previous research. We can further increase the toughness from combining carbon nanotubes and reduced graphene oxide flakes in solution-spun polymer fibers. During solution spinning, this toughness improvement is consistent with the development of a network of partially aligned reduced graphene oxide flakes and carbon nanotubes that deflect cracks and let polymer deformation occur more easily. The degree of graphene oxidation and the volume ratio of reduced graphene oxide flakes to carbon nanotubes in the spinning solution are both important factors for toughness. Hybrid fibers could be formed into high-modulus helical springs and were sew able and waivable. The chemical bonding between the matrix and GO makes stronger bond due to the presence of functional groups which helps in improving the mechanical strength of composites.

1.4 OBJECTIVES OF WORK

- Assess the overall performance of GO reinforced GFRP/CFRP laminates for improving the strength for enhancing the repair and retrofitting of RC structures.
- Assess the overall performance of rGO reinforced GFRP/CFRP laminates for improved performance.
- Investigate the tensile strength, flexural strength, and impact strength of nano reinforced GFRP/CFRP sheets.

1.5 LAYOUT OF THESIS

The thesis covers 5 chapters, and each chapter has its own importance in the thesis.

- **Chapter 1:** This chapter throws light on the basic background of GFRP/CFRP laminates modified with graphene derivatives and their properties
- **Chapter 2:** Throws light on literature review of different nano materials reinforced in GFRP/CFRP
- **Chapter 3:** Discusses about the experimental program and details of GFRP/CFRP laminates prepared
- **Chapter 4:** This chapter discuss the experimental results and analyzes the results with appropriate discussions
- **Chapter 5:** This chapter reports the major findings from the results with recommendations for future work.

2

REVIEW OF LITERATURE

2.1 GENERAL

This section presents the literature review on polymer-based nanocomposites processed for improved mechanical properties. The review mainly focuses on addition of fiber and/or graphene derivatives to epoxy based composites and epoxy based GFRP nanocomposite systems. The literature review has been used to discuss the effect of different constituents, methods of preparation, surface modification of fibers etc. on the mechanical properties of epoxy-based nanocomposite systems.

2.2 EFFECTS OF NANO FILLERS

Nano fillers such as nano clay, CNT, graphene derivatives are nano reinforced in the GFRP/CFRP to make the epoxy ductile to enhance the mechanical properties of these sheets. Graphene Oxide has great mechanical and durability properties due to which it has a huge impact in construction industry. Due to its oxygenated functionalities attached on the aromatic structure, it has improved dispersibility property than any other graphene-based.

2.2.1 NANO-CLAY

Kumar et al. (2010) investigated the outcome of clay addition on the tensile and flexural properties of epoxy-based glass fiber reinforced composites. Tensile strength and modulus have shown significant improvement by 133% and 58% respectively in epoxy-clay nanocomposites (at 5 wt.% of clay) as compared to pristine epoxy. In epoxy/clay/glass fiber nanocomposites, tensile properties increased till 5 wt.% of clay; beyond this clay loading, properties showed a decreasing trend. Flexural properties of epoxy–clay nanocomposites also increased till 5 wt.% of clay. In case of glass fiber reinforced nanocomposites, flexural strength and modulus improved by 56.8% and 103.6% respectively (at 5 wt.% of clay) as compared to glass fiber reinforced composite without clay.

Sharma et al. (2016) investigated the effect of surface treatment of nano clay on the mechanical properties of hybrid composites. The study was based on the Cloisite 15A nano clay treated with a method of silane treatment. Different coupling agents were used i.e., 3-glycidyloxypropyltrimethoxy silane and 3-aminopropyltriethoxy silane. These silane coupling agents were incorporated with different concentration such as 10,50, 200 and 400 wt.% of clay. These modified clays were incorporated in the GFRP laminates to enhance the mechanical properties. The hybrid composites were prepared using vacuum bagging technique. The results concluded that there was an improvement in the mechanical strength with the addition of these modified clays. The optimum concentration for these modified clays was 200 wt.%. The optimum concentration showed an increase in the elongation at break which showed improvement in mechanical strength.

Sharma et al. (2017) studied the curing studies and mechanical properties of GFRP composites based on silanized clay minerals. The phenomena's that clay minerals offer a great opportunity to achieve good dispersion in polymer matrix. Various studies have been done to modify the clay minerals using different techniques (*Bergaya et al., 2006; de Paiva et al., 2008; He et al., 2013; Kango et al., 2013; Kotal and Bhowmick, 2015; Varadwaj et al., 2016*). The improved reinforcement effect of clay minerals results in improvement of mechanical properties in the polymers even when a small amount of clay is added. The study aimed at complete curing of nano reinforced composites which includes clay minerals (even in small amount) results in increment of tensile and flexural properties. It is of significant use when used in the controlled concentration as post that it leads to swelling of clay minerals which would ultimately increase the grafting of silanes on the clay mineral. The result concluded that there was an increment in tensile and flexural properties when fiber reinforced nanocomposites modified with clay minerals with high amount of silicanes coupling agents.

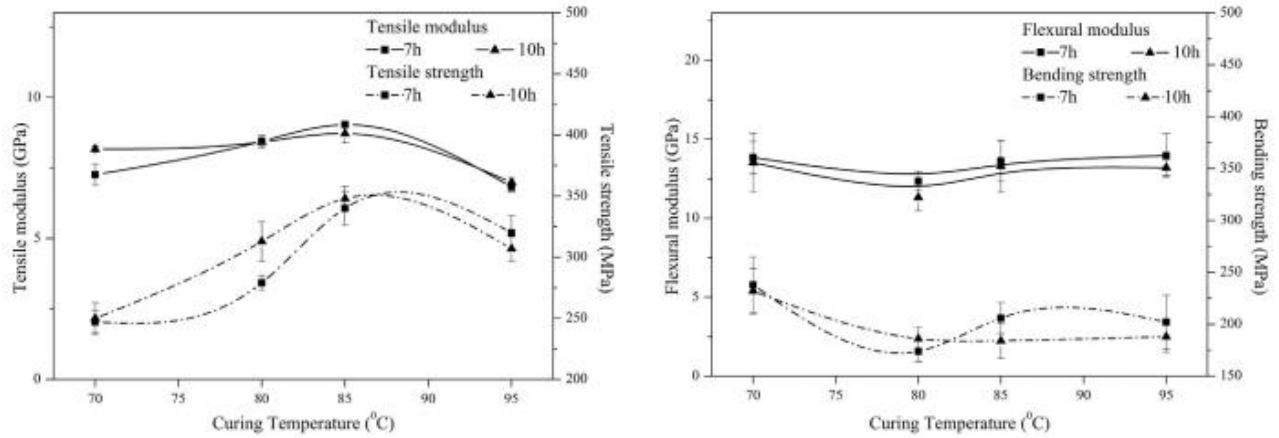


Figure 2.1: Effect of curing time and temperature on mechanical properties (Sharma et al., (2017))

Nanda et al. (2019) investigated the epoxy clay-based composites modified with clay for their mechanical properties such as tensile strength and impact strength. The concentration of nano clay varies with 0-4 wt.% The nano clay epoxy-based matrix was dispersed uniformly with the homogenizer and ultrasonication. Morphology is done using XRD and TEM analysis. Both the epoxy and nano clay have significant properties as epoxy has low shrinkage upon curing. The study concluded that an enhancement is observed in the properties only at the optimum clay loading level. Beyond the optimum level, it accumulates the clay, making the clusters or phase separated morphology the concern for deterioration in mechanical properties of epoxy-based polymer matrix composites. The silicate layers in these polymers behaves as a resistance to the cracks acting as a stopper, resulting in a path with less cracks that helps in improving the properties. The inclusion of 1.5 wt.% nano-clay shows an increase in tensile strength of the nanocomposites followed by a drop beyond that concentration. As with the optimum concentration an increase of 48% was observed with reference to the neat epoxy. The property of high aspect ratio in nano clay helps in filling the contact surface area of nano composites. The clay platelets reinforcing in the epoxy behaves as a stress transfer agent. While the increase in clay loading results in high viscosity of nanocomposites samples. The impact strength also follows the same as of tensile strength i.e., the optimum concentration of 1.5 wt.% of nano clay loading. The maximum increase in the impact strength is of 22% as compared to the neat epoxy.

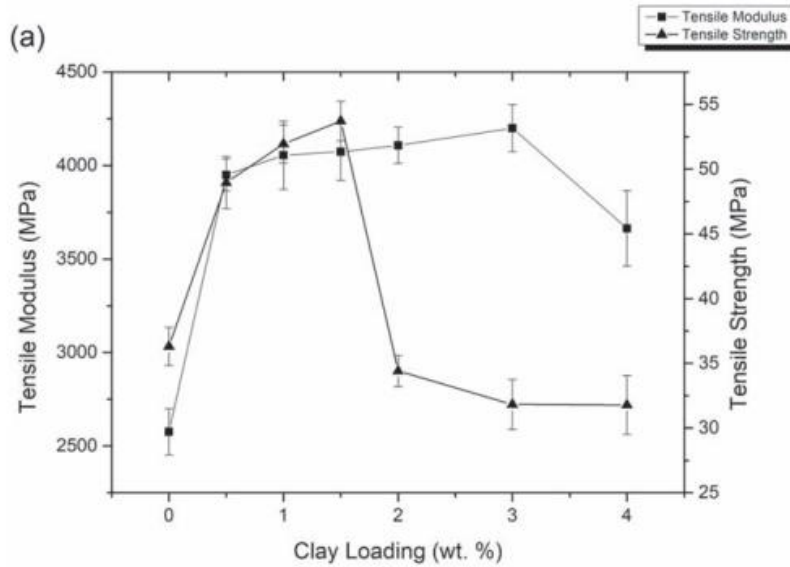


Figure 2.2: Effect of nano clay loading on tensile strength (Nanda et al., 2019)

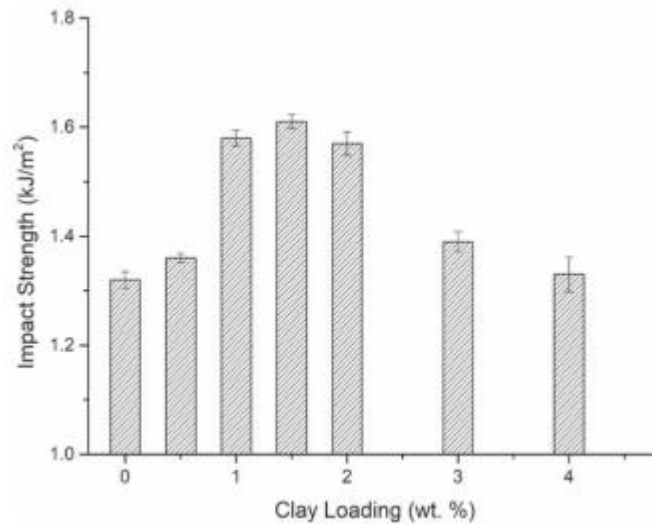


Figure 2.3: Effect of nano clay loading on impact strength (Nanda et al., 2019)

Shelly et al. (2018) studied the effect of nanomer clays to GFRPs for enhanced mechanical properties. Two different types of clays were nano reinforced in the GFRP laminates i.e., un-modified clay (Nanomer PGV) and an organically modified clay (Nanomer 1.28E) to improve the impact strength and fracture toughness. These hybrid composites were fabricated using VARIM. The results concluded that the un-modified clay had better impact in improving the mechanical strength than the modified clay. There was an increase of 22%, 31% and 73% in impact strength, flexural strength and fracture toughness respectively with the reference to the GFRP with no clay reinforced in them.

Shelly et al. (2021) investigated the addition of nano clay and ultra-high molecular weight polyethylene (UHMWPE) fibers to epoxy based GFRPs for improving the mechanical strength. These

GFRPs were fabricated using VARIM. The XRD spectra or X-ray diffraction electron microscopy confirmed the uniform or nano level dispersion of nano clay. The main objective for this study the effect of nano clay incorporated in GFRPs along with the UHMWPE fibers. These fibers possess great properties as they have low density, high toughness, and strength etc. The epoxy based GFRPs have great improvement with the inclusion of nano clay (nano-filler) and the UHMPWE fibers i.e., a micro-filler. An important factor was the compatibility along different constituents to improve the mechanical properties. The concentration of nano clay was at 2 phr where 3-aminopropyltriethoxy silane agent was used for salinization of nano clay. There was a variation in the addition of UHMWPE that varied in the range of 0.125-0.5 phr. The nano fillers are a great substitute as they carry high aspect ratio and large active surface area. It was observed that the optimum concentration was at 2 phr nano-filler and 0.25 micro-filler. These modified composites depict the improvement in mechanical properties where 23.5% increase is seen in impact strength and 4% in tensile strength. The improvement in these propertied is because of the better bonding between the epoxy molecules and nano clay.

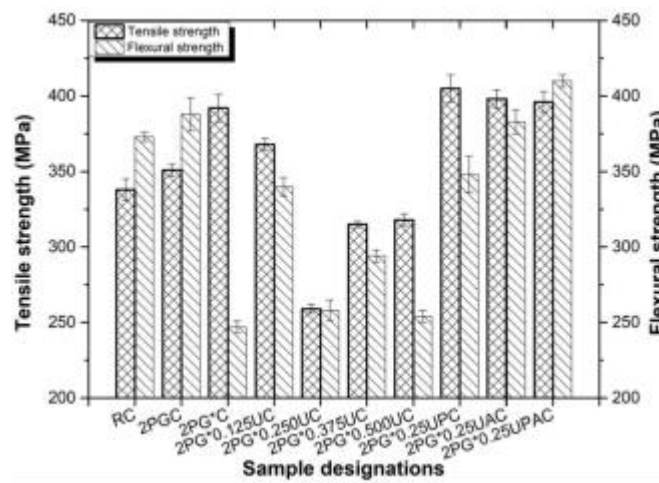


Figure 2.4: Effect of nano clay loading and UHMWPE fibers on tensile and flexural strength (Shelly et al., 2021)

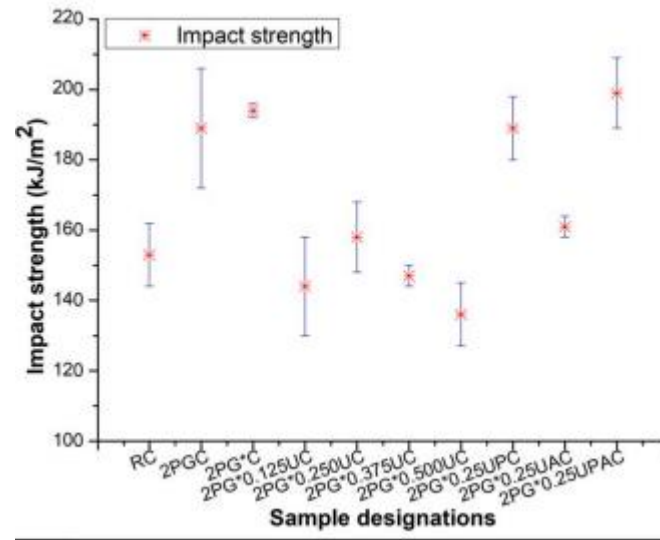


Figure 2.5: Effect of nano clay loading and UHMWPE fibers on impact strength (Shelly et al., 2021)

2.2.2 CNT modified

Siegfried et al. (2014) studied the consequence of carbon nanotubes (CNTs) on the impact and post impact performance of woven carbon fiber/epoxy composites. Three nano-reinforced epoxy systems that differ in CNT dispersion and functionalization are used as a matrix material. The composites are verified for the resistance to an out-of-plane low velocity impact, the residual compressive strength, the mode II interlaminar fracture toughness and the interlaminar shear strength. The composite with the highest mode II fracture toughness is found to be the best performing system in compression test. The resin in this composite contains a network-like structure of CNTs. These nanotubes significantly improve the mode II interlaminar fracture toughness and because of that damage can be controlled of a composite. Furthermore, composites are more persuaded to the onset of matrix cracks leading to a larger delamination area post impact. The conclusion was that the material's sensitivity to matrix failure under in-plane compressive stresses is increased when CNTs are present in the matrix. Because of the toughening processes of CNTs, such as bridging cracks and CNT pull out, CNTS may enhance the characteristics of composites. During the impact, these matrix cracks serve as delaminating initiation sites. As a result, it's no surprise that the delaminating parts of the CNT-containing materials were larger, as there were also more matrix cracks present. The findings of this research show that the dispersion state of CNTs affects material mechanical properties. The results concluded that addition of CNT has significantly improved the mechanical properties. The results were also proved by the morphology of these hybrid composites.

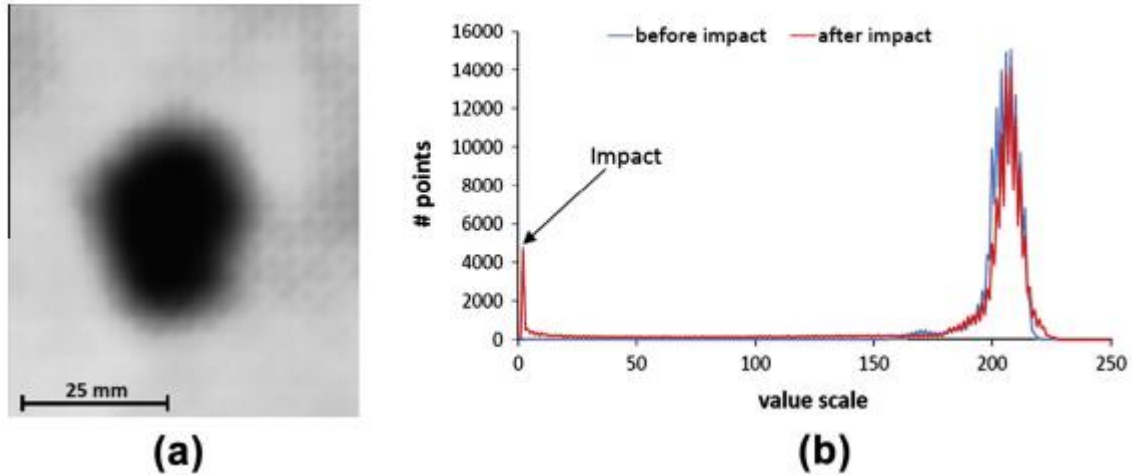


Figure 2.6: (a) The c-scan image of a damaged area after impact, (b) the c-scan histogram of a sample before and after impact (Siegfried et al., 2014).

	CF/epoxy/CNT	CF/epoxy/CNTa	CF/epoxy/CNTf
CNT dispersion			
Changes in CNT dispersion	as received	through masterbatch aging	through CNT functionalization
Impact test			
Delaminated area, mm ²	Increased, more matrix cracks	Increased, more matrix cracks	Increased, more matrix cracks
Compression test (after impact)			
Residual strength, MPa	Decreased	No change	Decreased
Mode II interlaminar fracture toughness			
GIIc, J/m ²	No change	Increased	No change
SBS test			
ILSS, MPa	No change	No change	No change
Failure modes	multiple shear + compression	multiple shear + compression	Single shear + compression

Figure 2.7: Overview of the results in comparison with the reference CF/epoxy composite (Siegfried et al., 2014).

Garg et al. (2016) studied the Glass fiber reinforced polymer (GFRP) modified with carbon nanotubes (CNT) of two types i.e., pristine nanotubes (PCNT) and amino-functionalized (ACNT). The main objective of this research was to compare the effect of two different liquids i.e., Sea water

(SW) and Distilled water (DW) on the mechanical properties and durability in GFRP and hybrid composite laminates. The experiment for both GFRP and hybrid composites was processed under the similar conditions i.e., at room temperature of 25⁰ C. This study also focused on the kinetics of energy absorption for modified GFRPs and unmodified GFRPs. The graph was plotted that depicted the water absorbed along with the increase in weight % versus time for modified and unmodified GFRPs. The trend for the diffusion curves is similar for both over the aging time. During the span of 18 days, increasing trend was observed for GFRP specimens and at that time, their maximum water content level was noted down. Many studies have seen the similar kind of results for polymer matrix composites. The trend of increasing in initial stages fails when the aging time increases as it could be due to the immediate drop in water content once the maximum level is breached. It could happen due to various reasons such as debonding, delamination or due to development of cracks etc. inside the matrix composites. The primary reason is the excess water intake that caused inflammation of epoxy matrix. The other reason for the same could be the chemical interaction of glass fibers surrounded by water molecules. Other justification for this could be the stress relaxation that occurs due to the swelling after water absorption. The composited modified with CNTs showed decrease in water uptake content as compared to unmodified GFRPs. The covalent bonding of CNTs and epoxy matrix also helps in slowing down the movement of water and these nano fillers occupied any free voids in the composites. The mechanical properties for modified GFRPs as compared to the other.

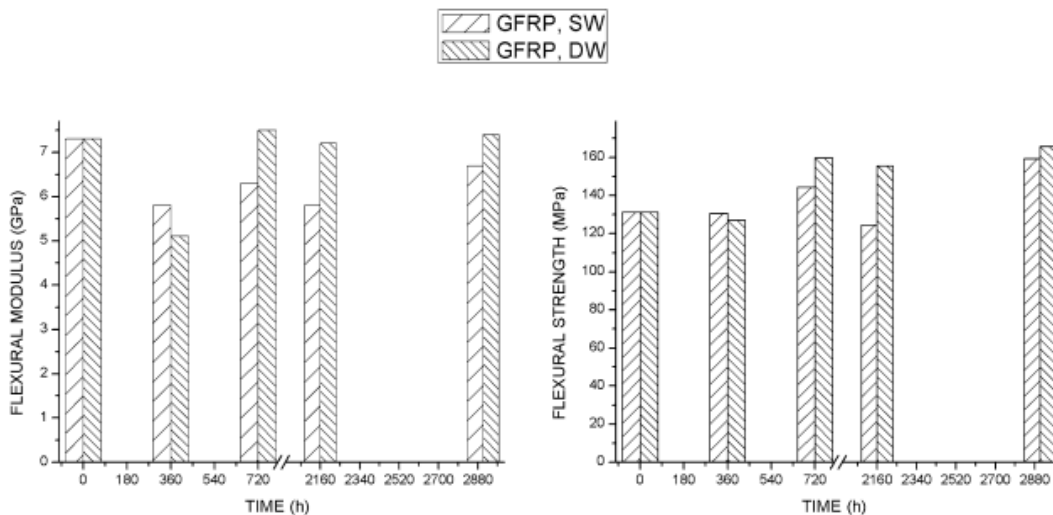
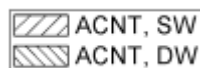


Figure 2.8: Flexural properties of GFRP immersed in SW and DW for inc. aging time (Garg et al., (2016))



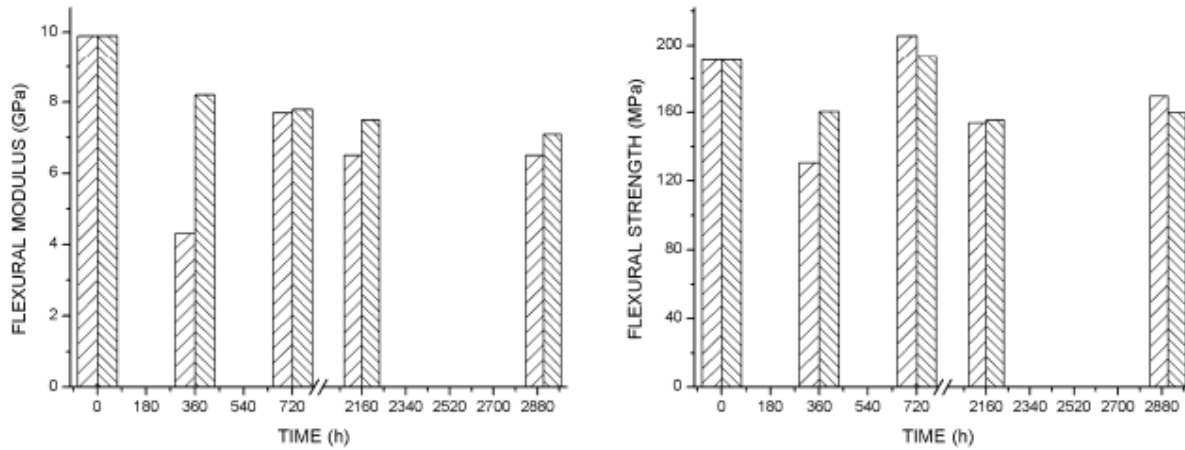


Figure 2.9: Flexural properties of CNT GFRP immersed in SW and DW for inc. aging time

(Garg et al., (2016))

Garg et al. (2016) investigated the role of curing conditions and silanization of glass fibers on carbon nanotubes (CNTs) reinforced glass fiber epoxy composites. The amino functionalized nanotubes (ACNT) were reinforced in the GFRP laminates and were analyzed using thermal analysis. The results showed that modification in curing can enhance the thermomechanical properties. The strong fiber matrix interfacial interactions due to the addition of CNTs has significantly improved the properties. The silane treatment of GFRPs when reinforced with ACNT has improved the tensile and flexural strength of the composites whereas when PCNT GFRP composites were reinforced it showed improvement in the other mechanical properties. This improvement is seen in the composites because of the presence of strong covalent bonding between fiber-matrix interactions.

2.2.3 Graphene Derivatives modified

Pathak et al. (2012) investigated to progress the mechanical properties of polymer composites; graphene oxide was preferred as one of the fillers for the growth of CFRP/GO hybrid composites. Initially, 0.1 to 0.6 weight% of graphene oxide was used to modify epoxy resin by incorporating various weight%. To create hybrid composites by compression molding, the required size of carbon fiber cloth was impregnated with modified epoxy resin. FTIR, XPS, NMR, XRD, and Raman Spectroscopy were used to characterize the graphene oxide produced. The figure 2.10 shows the XRD spectra of GO where we see a sharp peak at $2\theta = 11^\circ$. Different kind of functional groups are seen to exist in graphene oxide produced, which is responsible for binding to epoxy resin and Carbon fibers. With 0.3 weight% of graphene oxide included in the carbon fiber reinforced polymer hybrids,

the hybrid composite flexural strength improves by 66%, flexural modulus increases by 72%, and interlaminar shear strength improves by 25%. Hydrogen type bonding and mechanical interlocking of graphene oxide with carbon fibers and epoxy resin are responsible for the improvements in composite properties at the percolation threshold. One of the ways to increase the characteristics of carbon fiber polymer composites is through graphene oxide usage.

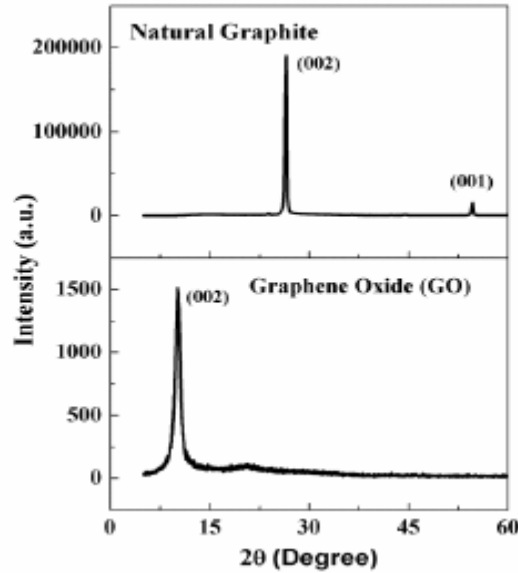


Figure 2.10: XRD plot of Graphene oxide (GO) relative to natural graphite flakes (Pathak et al., 2012).

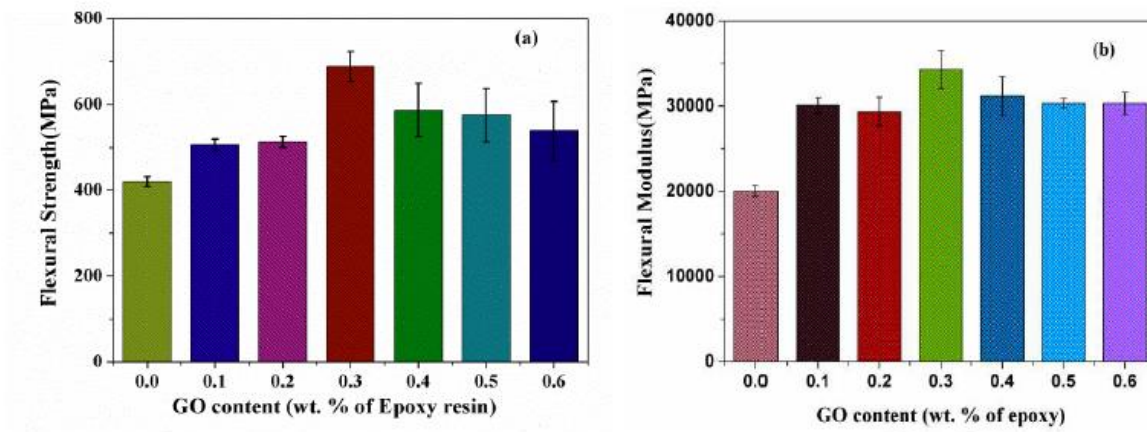


Figure 2.11: Variation in Flexural strength and flexural Modulus with increasing GO content in epoxy resin (Pathak et al., 2012).

The mechanical properties of CFRP composites also depend upon the interfacial interaction of fiber and epoxy matrix. Interlaminar shear strength (ILSS) is the most required measurements for calculating out the interfacial interactions. The figure 2.12 is presenting a 23% enhancement in the ILSS of 0.3 weight% GO epoxy/carbon fiber composite to pure epoxy/carbon fiber composites. This improved ILSS attributes in fact, that GO contains the oxygenase function group which exerts polarity onto the surface and edge of GO because of this it exerts good dispersion which led effective bonding between GO and matrix. Epoxy (Bisphenol A epoxy resin) includes epoxy and hydroxyl as functional group, so hydrogen bonding involved among GO molecules and epoxy as well with carbon fibers.

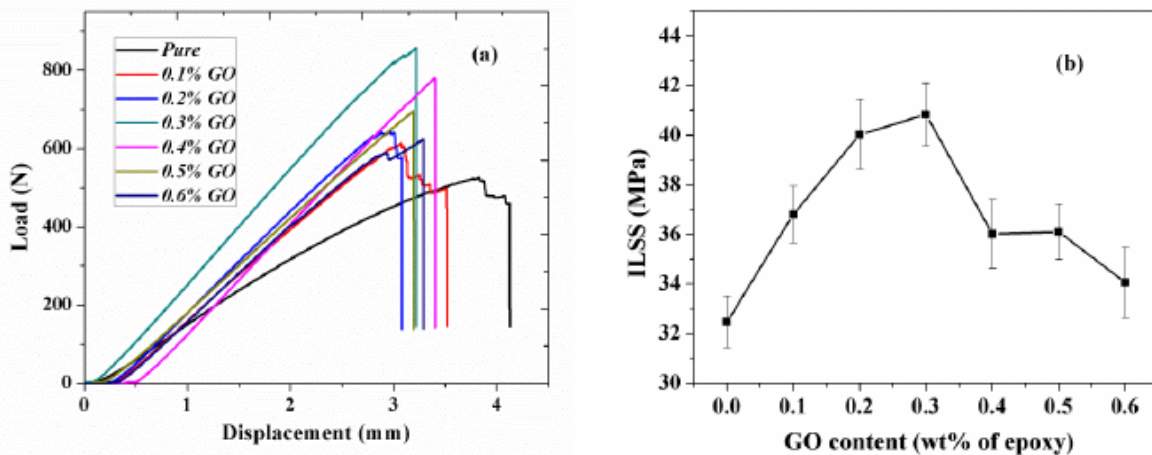


Figure 2.12: (a) Maximum load vs. extension with GO weight % variation and (b) ILSS difference with GO wt. % of epoxy (Pathak et al., 2012).

Umer et al. (2015) studied the properties of graphene oxide (GO) nanoparticles on glass fiber composite processing by incorporating them into epoxy resin. GO was synthesized from graphite powder and was mixed with epoxy resin to make the epoxy ductile. Three different GO contents of 0.05, 0.1, and 0.2 wt% were used. To see the impacts of GO content on important resin infusion processes, epoxy/GO samples were investigated for rheology and cure kinetics. The findings demonstrate that increasing the viscosity of neat epoxy resin and shortening the gel time resulted in improved results. The resin infusion method was then used to make glass fiber composites. The key processing parameters used in the production of composite structures using liquid molding methods have been shown to be affected by GO nanoparticles. The GO-modified epoxy has a noticeable catalytic response that influences total cure rate, as evidenced by the results of cure kinetics. In

contrast to the pure epoxy system. Despite the reduction of processing window for resin infusion, the improved curing behavior of GO-modified epoxy resin has the potential to improve curing cycles in terms of time and energy savings.

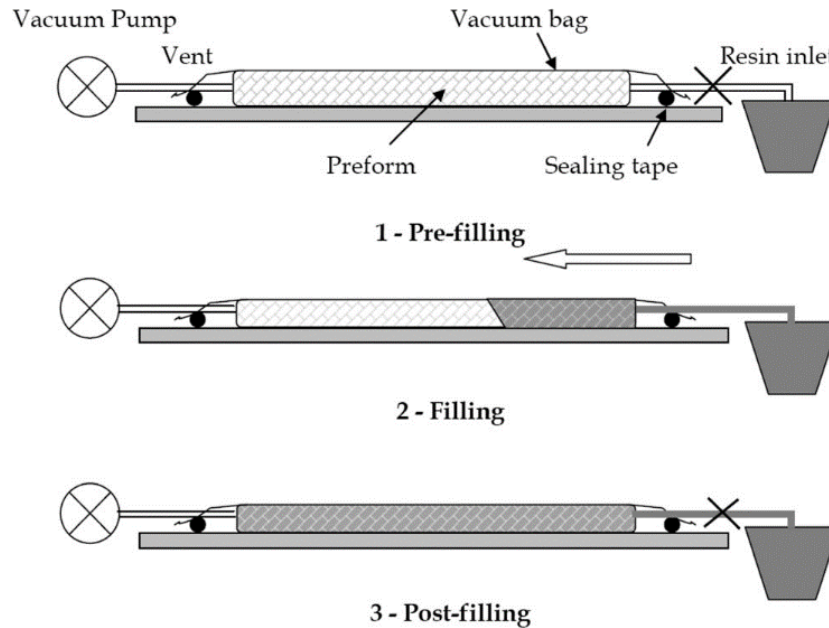


Figure 2.13: Representation of a typical resin infusion process (Umer et al., 2015)

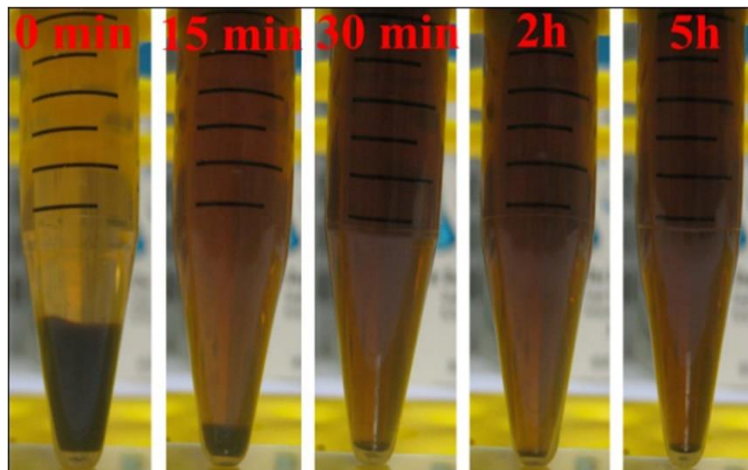


Figure 2.14: Optical images of GO–water solution in a tube, treated at different ultrasonication times (photos taken after 7 days) (Umer et al., 2015)

Yao et al. (2017) designed the fiber interface and discusses that the behavior of the carbon fiber-reinforced composite can be rehabilitated. The research shows that carboxylic-functionalized carbon nanotubes (CNTs) and graphene oxide (GO) were individually deposited on the carbon fiber (CF)

surface to enhance the interfacial properties of composites. The properties of fibers that includes the surface topography, surface chemical composition and surface energy, and the interfacial shear strength between the fibers and the matrix was examined using a microdroplet test. Furthermore, GO-coated, CNT-coated, and pristine unidirectional carbon fiber fabrics were considered to make CF/epoxy composites via the vacuum-assisted resin transfer molding (VARIM) method, and their moisture absorption and interlaminar shear strength were calculated. The conclusion from the research revealed that both CNTs and GO have improved the interfacial properties of composites. The CNT/CF-reinforced composites have higher shear strength, and the GO/CF-reinforced composites have better humidity resistance. The interfacial reinforcing mechanisms of the composites with CNTs and GO were analyzed and compared. The bonding strength among the matrix and the studied fibers was assessed by probing the IFSS, and the findings are depicted in the figure 2.15 and 2.16 respectively. As the results proved that the GO and CNTs have improved the interfacial bonding strength of the mixtures.

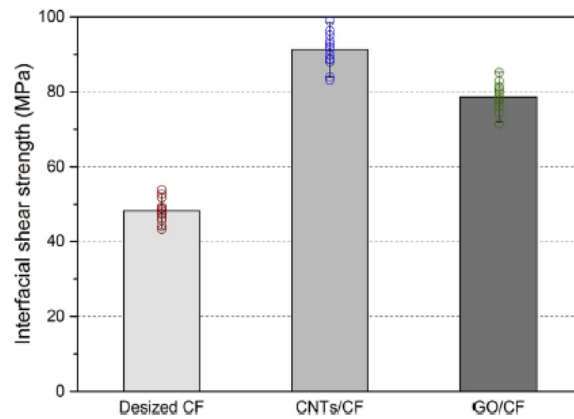


Figure 2.15: Interfacial shear strength results of the compounds. (Yao et al., 2017).

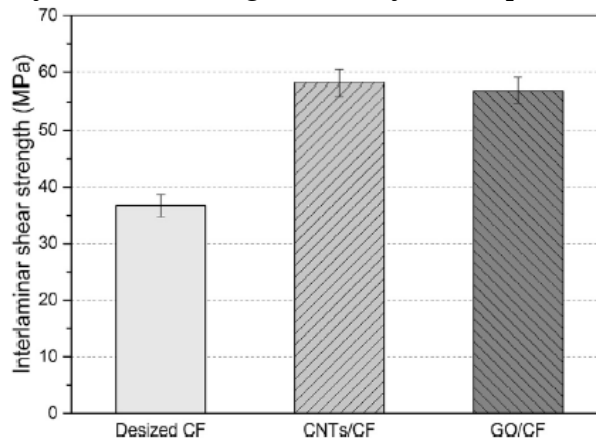


Figure 2.16: Interlaminar shear strength of the composites. (Yao et al., 2017).

The electrophoretic deposition approach is an effective method to deposit carboxylic functionalized CNTs and GO onto the surface of carbon fibers, as shown in this research. The fiber morphologies and chemical compositions were investigated and compared with each other. The interfacial shear strength of CNTs/CF increased by 89.4% relative to the desized CF, according to microdroplet testing. Carbon-fiber-reinforced epoxy composites' interfacial features are improved when nanomaterials are incorporated. The moisture absorption of the GO/CF/epoxy composite was reduced by 63.6%, while the CNTs ILSS increased by 58.6%. The creation of distinct interphases in the CF/epoxy composites is due to differences in the dimensions and structure of GO and CNTs. The interphase in the CF/epoxy composite was a nanocomposite of CNTs and epoxy, whereas in the GO/CF/epoxy composite it was a CF-GO-epoxy structure.

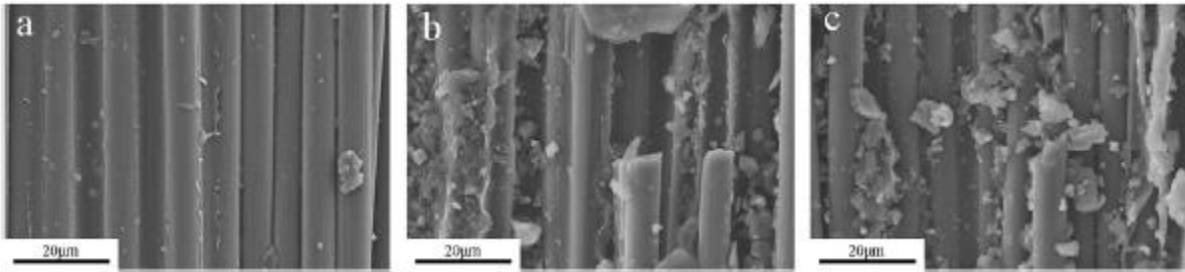


Figure 2.17: Fractured descriptions of the (a) desized CF, (b) CNTs/CF, and (c) GO/CF (Yao et al., 2017).

Prusty et al. (2017) investigated the unique and exceptional properties of graphene based nanofillers in fibrous polymeric composites. The mechanical response of glass fiber/epoxy (GE) composite loaded with a variety of graphene oxide (GO) concentrations was studied in this investigation. In the results, flexural strength was improved by 21.1% when 0.5 wt.% GO was incorporated in the material. The impact of interface (GO/epoxy and glass fiber/epoxy) on flexural behavior at various testing temperatures has been investigated, as well as the state of interface. At a temperature range of 40 to 200°C, the viscoelastic features of all the materials were further investigated. The effect of GO content and testing temperature on the Weibull design parameters has been examined. To better understand different interfacial strengthening and failure micro-mechanisms, scanning electron microscope (SEM) analysis of fractured surfaces was carried out. The effect of GO content on the flexural stress strain response of GE composites at various temperatures has been shown in the figure 2.14. The dependence of flexural response of the material

on environmental temperature is more pronounced in case of GO-GE composite than GE composite.

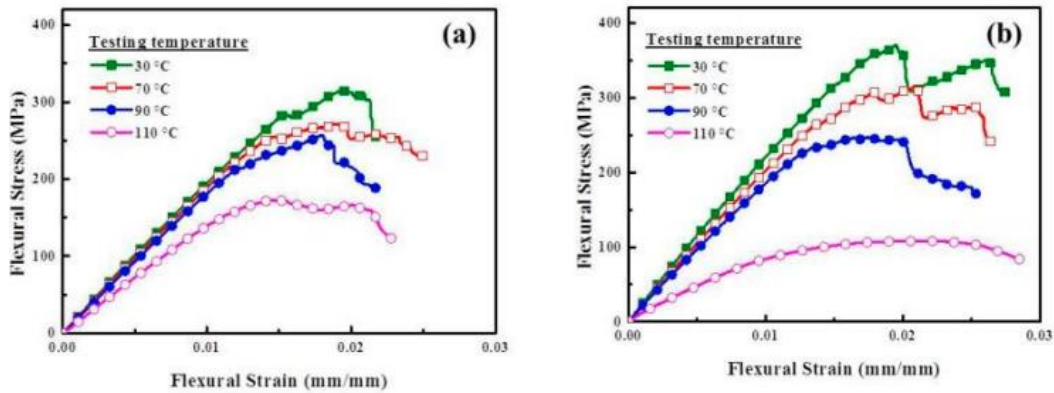


Figure 2.18: Variation in flexural strength with (a) 0 wt.% GO and (b) 0.1 wt.% GO (Prusty et al., 2017).

The flexural properties, i.e., strength and modulus of the materials at various testing temperatures are shown in the figure 2.18. The addition of 0.1 wt.% of GO has significantly improved the properties (18.5% in strength and 15.6% in modulus).

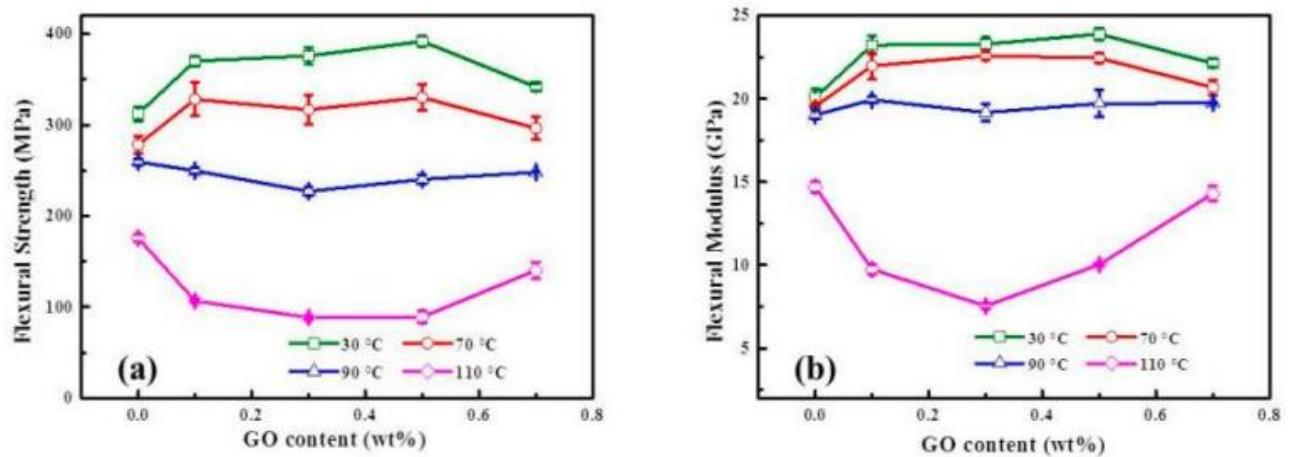


Figure 2.19: Variation in (a) flexural strength, (b) flexural modulus and (c) strain at peak stress (Prusty et al., 2017).

Yuan et al. (2018) studied different surface modifications (specifically four) that have been pragmatic on carbon fiber (CF) before the graphene oxide (GO) electrophoretic deposition (EPD) for preparing GO/CF hybrid fiber reinforced epoxy (GO/CF/EP) composites, that contains acid oxidation, silane coupling coating, sizing coating and electrochemical oxidation. The most important pointed to be noted here that electrochemical oxidation joined with EPD was a viable method so that

it can easily and evenly disperse graphene oxide over CF surface. However, acid modification could increase surface roughness while sizing while coupling coating accountable for chemical activity. In addition, various comparative analyses were performed with diverse modifications, and was shown that surface oxygen-containing functional groups and micromechanical interlocking helped majorly in robust strengthening and electrical conduction of GO/CF/EP composites, as verified by GO/CFs-eco and GO/CFs-acid reinforced composite samples. Based on these results, the four kinds of surface modification were introduced which provided with the proper treatments for the combination of optimizing interfacial properties in GO/CF/EP composites.

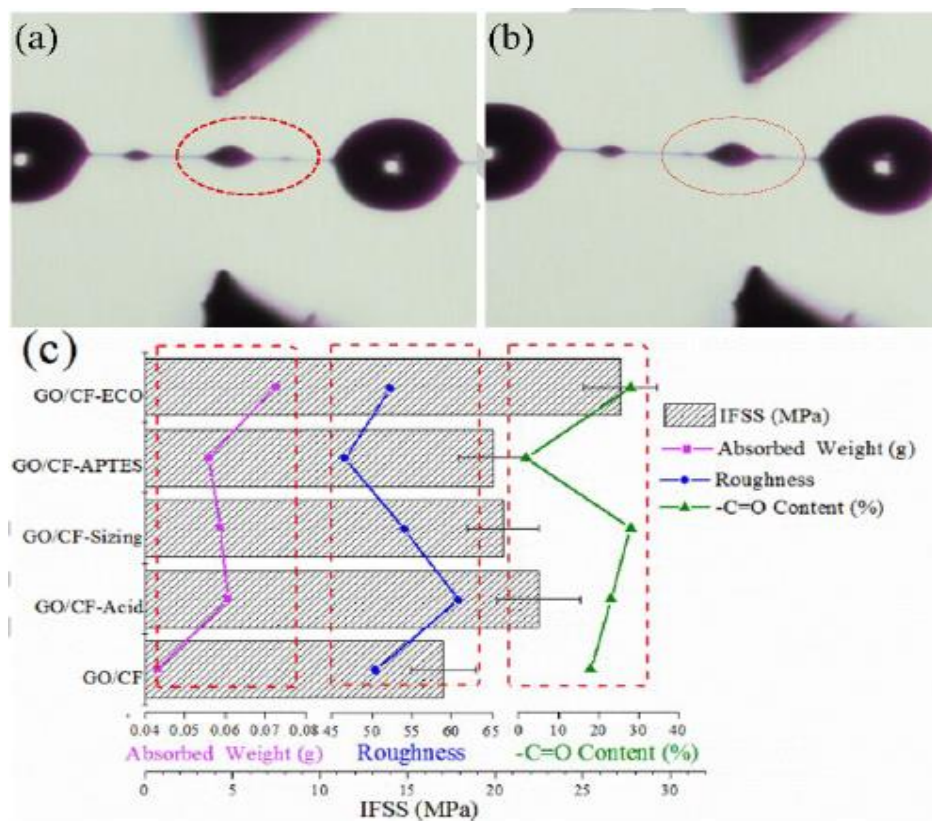


Figure 2.20: Representation of the IFSS gauging procedure (a-b) and effects of various surface modifications before to EPD on the enhancements of IFSS (c) (Yuan et al., 2018).

The reinforcement of GO on the CF surface has improved the mechanical performance of CFRP sheets. When electrochemical oxidation was used to create a homogeneous distribution of GO coating, the SEM and AFM pictures revealed it. GO was also chemically coated on the CF surface through covalent attachments, according to XPS results. The O/C ratio increased by 56.87% and 44.65% in sized coated and electrochemically oxidized GO/CFs, respectively. Increased permeation

of resin in acid-oxidized and coupling coated-GO/CFs was detected because of the improvement in mechanical properties, according to dynamic wettability studies. The in-plane electrical conductivities of GO/CFs-ECO/EP composites increased dramatically due to their uniform nanocapacitor structure and interface polarization. Based on these results, we propose that different surface physiochemical treatments on CFs are directive and purposeful for enhancing mechanical and electrical properties in GO/CF/EP composites. We suggest an applicable utilization of GO/CF-Acid/EP composite for high-strength bulletproof materials, which is beneficial in terms of interfacial adhesion concerns. The electrochemical oxidation of CF can improve the electrical conductivity and mechanical properties.

Refiee et al. (2018) studied the thermal properties of doubly reinforced fiberglass/epoxy composites with graphene nanoplatelets, graphene oxide and reduced-graphene oxide. A novel manufacturing method based on Vacuum Assisted Resin Transfer Molding (VARTM) was devised to incorporate carbon nanoparticles for the enhancement of thermal properties of multiscale laminates. Several graphene-based nanomaterials including graphene oxide (GO), reduced graphene oxide (rGO), graphene nanoplatelets (GNPs) and multi-walled carbon nanotubes (MWCNTs) were used to modify the GFRP laminates. The thermal, rheological, and morphological properties of the resulting glass fiber reinforced multiscale composites were investigated. The thermal properties of the epoxy/nanoparticle composites were studied through thermal conductivity measurements, differential scanning calorimetry (DSC) and thermo-gravimetric analysis (TGA). The thermal characterization results showed that the introduction of GNPs, GO, rGO, and MWCNTs enhanced thermal conductivity. Compared with the neat epoxy/fiberglass composite control results, improvement in thermal conductivity of fiberglass/epoxy modified with MWCNTs 0.3%, GNPs 1%, GO 2% and rGO 0.042% were 8.8%, 12.6%, 8.2% and 4.1%, respectively. The results showed that the addition of these nano particles even in very small quantity has significantly improved the properties.

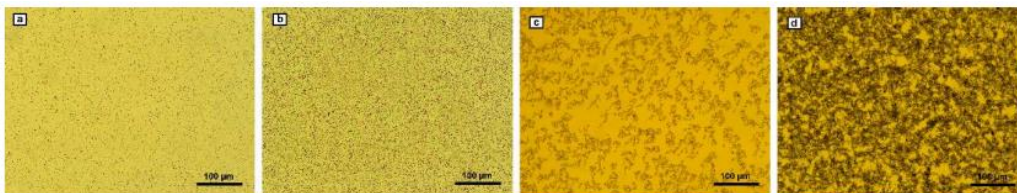


Figure 2.21: Overview of the results in comparison with the reference CF/epoxy composite (Refiee et al., 2018).

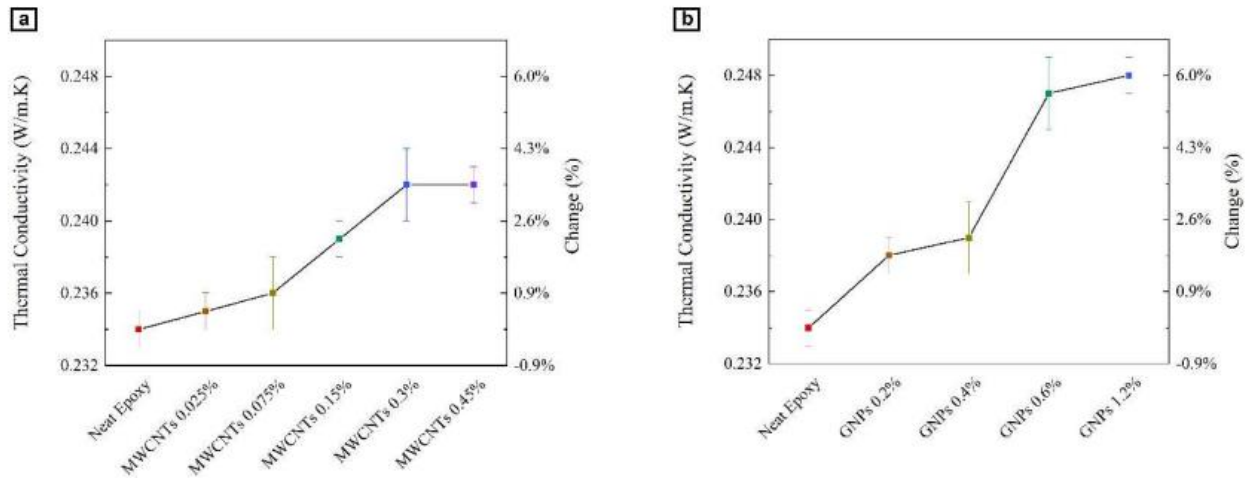


Figure 2.22: Thermal conductivity of epoxy modified with different loading of MWCNTs and GNPs (Refiee et al., 2018).

Pathak et al. (2019) studied the interfacial properties of graphene oxide incorporated in CFRP nanocomposite. This study was done using different concentrations of GO included in the epoxy matrix and the CFRP hybrid composites were prepared using compression molding process. It showed that significant result. The fiber volume content, orientation of fibers and fiber-matrix interactions play a major role in the mechanical properties for the CFRP laminates. The weak interfacial bonding of the laminates and the polymer matrix is a major obstacle as it leads to cracks and failure due to various modes. Hence to overcome the problem nano fillers such as CNT and nano clays are used. These both nano fillers come at a cost and have high complexity due to which graphene derivatives were introduced. GO has oxygenous surface and have functional groups present in its composition that helps in the dispersion filling the empty spaces or voids in composites. Because of the OH groups it is best suited for the reinforcing with the polymer matrix as they have high bonding capability. The characterization and morphology are done using XRD and SEM analysis. FTIR is used to analyze the functional groups present in the graphene oxide (GO). The properties were enhanced with the inclusion of GO in the matrix at the concentration of 0.3 wt.%. The trend of properties was decreasing beyond the optimum concentration due to the steric hinderance in the higher concentration of GO. The figure 2.23 represents the effect of high concentration of GO influences the CF-epoxy composite as the more addition of GO acts as a steric hinderance in the whole system.

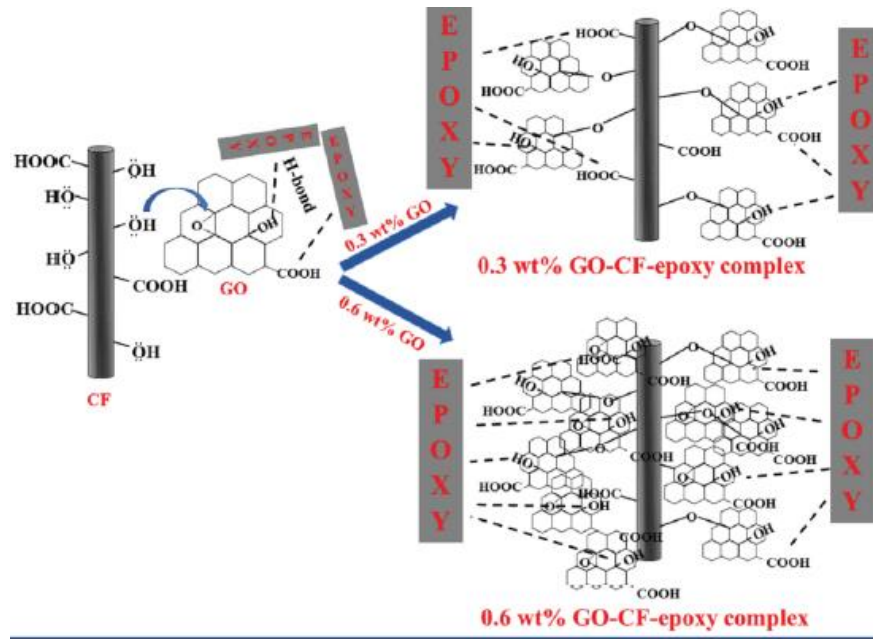


Figure 2.23: Effect of high concentration of GO influences the CF-epoxy composite (Pathak et al., 2019).

2.3 HYBRID COMPOSITES

Reddy et al. (2016) aimed at emerging Carbon and E- glass based hybrid composite laminates for armor applications to get advantages of both the types of fibers. The prominence of thick laminate failures also differentiates with the thickness. All these failure methods are not seen in one single reinforcement. Therefore, to overcome this problem and have a better performance, hybrid composites were involved which is made from two or multiple fibers or resins. It is observed that when multiple fibers are involved in composite laminate, the performance is enhanced by a good margin. The synergy effect thus resulted due to different fibers is called hybrid effect. The increment in hybrid effect helps to attain a composite property that depicts higher value as of single. Hybridization provides an edge in developing lower-areal weight laminates or higher specific energy absorption due to lower density carbon fibers. The other advantage of hybrid composites is that they provide lower density because of low areal density of carbon fibers. The figure 2.24 shows the breaking point of the hybrid composites. The samples have broken through the middle point.

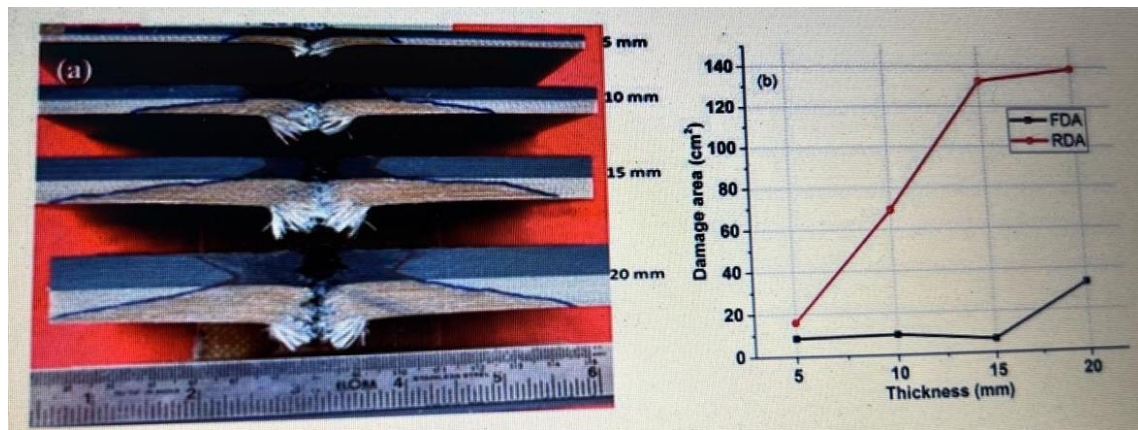


Figure 2.24: (a) Images of cross section of hybrid laminate (b) Damage area of varying thickness laminates (Reddy et al., 2018).

Gokuldass et al. (2019) (a) investigated the effect of modifying the epoxy resin with micro-rubber and nano silica on mechanical properties of woven fabric glass/Kevlar based hybrid composites. Three stacking sequence [GG-KK-KK-GG], [GG-KK-GG-KK] and [GG-GG-KK-KK] were used to prepare nine sets of hybrid composites. Nine sets of hybrid E-glass/Kevlar weave fibers were made with virgin epoxy, epoxy blended micro-rubber (9 wt.%) and nano silica (11 wt.%) and epoxy with micro-rubber (9 wt.%) to form the hybrid resin composites. Mechanical properties of hybrid E-Glass/Kevlar composite with nano-silica and micro-rubber were found to be higher than composites contain only fiber reinforcement. Fracture toughness results showed that combination of E-Glass/Kevlar composite with micro-rubber modified epoxy showed higher value than plain epoxy and epoxy modified with nano-silica/micro-rubber. Stacking sequence of GG/GG/KK/KK provided better mechanical properties than other stacking sequences.

Gokuldass et al. (2019) (b) investigated the effect of modifying the epoxy resin with micro-rubber and nano-silica on mechanical properties of intra-ply and mono-fiber woven glass/Kevlar fabric-based hybrid composites. Epoxy resin modified with rubber and nano-silica and reinforced with intra-ply woven mat showed maximum improvement for fracture toughness (from 9.5 to 48.5 MPa). This improvement was due to intra-ply fibers which offered more barrier layers. Intra-ply woven mat fiber lamina pattern in epoxy matrix provided higher mechanical strength than pure epoxy and mono-fiber of glass and Kevlar woven mat-reinforced epoxy composites. The technique used in the experiment was hand layup method.

Altin et al. 2020 studied the graphene oxide modified carbon fiber reinforced epoxy composites. Three different concentrations of GO were incorporated in the CFRPs to enhance their mechanical strength. This research focused on the graphitic surface of fibers modified with GO nanoparticles with the spray coating technique. This technique was preferred because it was easy, cheap and quick method. The SEM analysis concluded that the derivative GO was coated homogeneously on the surface of fibers. The major component that plays part in the mechanical properties of CFRPs is the interface interactions between the fiber and matrix. These interactions are dependent on the polymer matrix used, the reinforced fiber and the interactions between molecules. With strong bonding it is very efficient to transfer the load onto fiber by decreasing the tension which helps in increasing the strength of laminates. Several modification methods were discussed to increase polarity of CF so that the chemical reactions can bond efficiently. The results shown that when GO was incorporated in CFRP it improved the tensile strength, young's modulus.

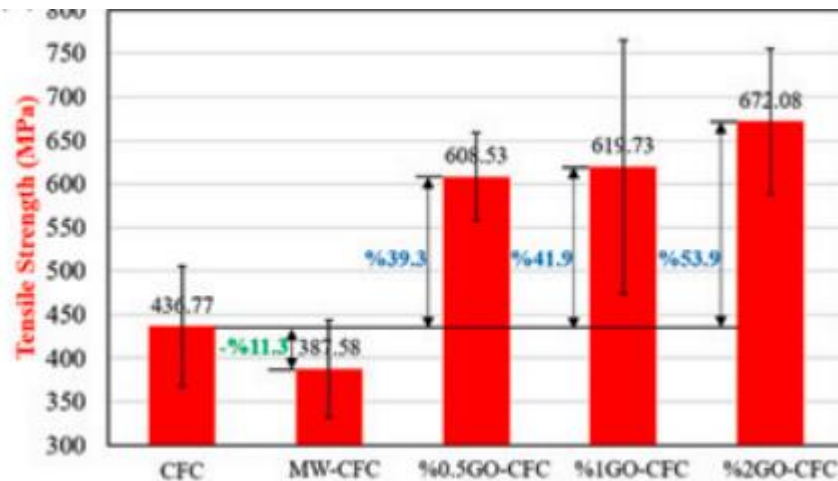


Figure 2.25: Effect of GO modified composites on tensile strength (Altin et al., 2020).

2.3 CLOSING REMARKS

The chapter has discussed several studies reported for the nano fillers or nano reinforcing with fiber polymers and check the overall the performance of the structure.

3

EXPERIMENTAL PROGRAM AND METHODOLOGY

3.1 GENERAL

This chapter outlines the research program followed in this thesis. The aim of the research is to access the overall performance i.e., tensile, flexural and impact strength of GO/rGO nano reinforced GFRP/CFRP laminates for improved repair and retrofitting of reinforced concrete structures. Basically, the epoxy resin that use in the laminates (GFRP/CFRP) is brittle and it leads to its premature failure under various loading conditions such as debonding, matrix failure. Slipping of fibers etc. Hence, to make the epoxy resin ductile different nano particles like nano clay, CNTs and graphene derivatives are used. Therefore, to enhance the strength and the performance of GFRP/CFRP laminates the epoxy is nano reinforced with graphene derivatives. Different GFRP/CFRP laminate sheets were prepared using different concentration of GO/rGO in epoxy and their performance is assessed in tensile, flexural and impact testing.

3.2 TEST METHODOLOGY

The test program for the same involved the following steps:

STEP 1: Weighing of different materials like GO/rGO, epoxy and hardener using weighing scale

STEP 2: Prepare the epoxy and GO based mixture and ensures uniform dispersion of GO in the epoxy matrix using Homogenizer

STEP 3: Probe Sonicator is used for effective processing of the materials that does not allow the particles to settle down.

STEP 4: Mix the hardener with the mixture using the Mechanical stirring machine

STEP 5: Vacuum Assisted Resin Infusion Molding (VARIM) will be used for fabricating GFRP/CFRP sheets

The methodology used for testing is outlined in Figure 3.1 below:

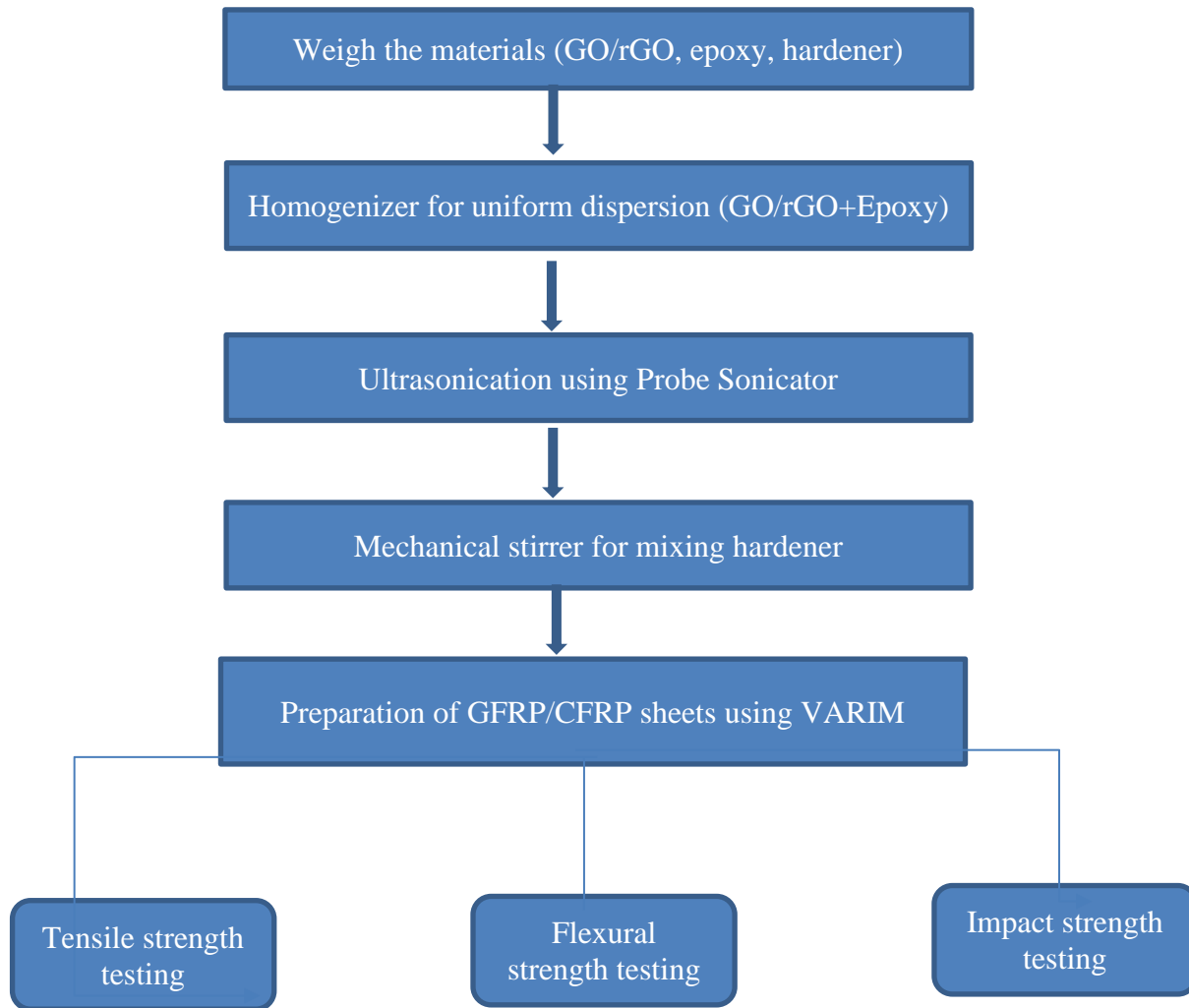


Figure 3.1: Flow chart of working methodology

3.3 MATERIALS USED

Epoxy Resin and Hardener: Epoxy is one of the most important thermosetting resins which is employed as a matrix material for polymer-based composites. The epoxy used in the experiment is Araldite CY 230 and the hardener used is HY 951, sourced from Huntsman Advanced Materials, India. As per supplier's recommendation epoxy and curing agent were mixed in the weight ratio of 10:1. The weight of epoxy and hardener used in the experiment was 170g and 17g respectively.

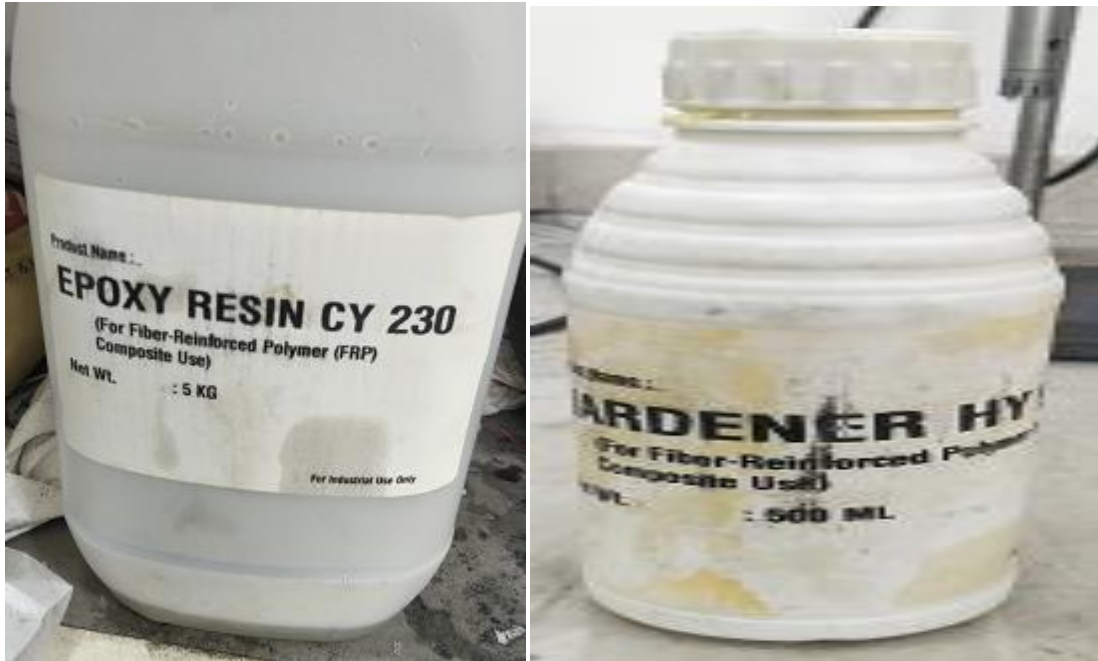


Figure 3.2: Resin and hardener used for making epoxy

Acetone: A clear and colorless liquid used for the dissolving or breaking down the particles present in the equipment so that they are completely dry. The main advantage of this solvent is that it evaporates in the air very quickly thus making it ideal for the process.

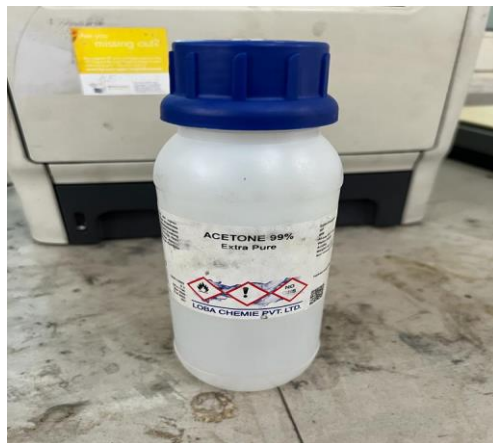


Figure 3.3: Acetone used in the process

Infusion mesh: In the past, this polymer weave was utilized in the advanced composite sector. To guarantee the consistent flow of resin across the mold surface, it is laid flat across the whole mold surface. It may withstand large negative pressures and is used in high vacuums. To better see the flow of resin across the mold surface, it is typically utilized in a green color. The resin is then removed from the molding surface after it has cured. The specifications for the mesh were 160 GSM 1.5 m width. It was obtained from Vactech Composites Private Limited.



Figure 3.4: Infusion mesh for covering the sheet

Mold Cleaner and Releasing Agent: The mold should be rigid and have a high-gloss finish. The VARIM table surface was to be cleaned with a cleaner to have a uniform surface and clean mold. After cleaning, a releasing interface is applied on the surface to prevent other materials from bonding to surfaces. Basically, it acts as a critical barrier between the molding surface and the substrate, to separate the cured part from the mold. The substrate would become bonded to the mold surface if such a barrier was not in place, making clean-up difficult and productivity loss significant. The releasing agent used for the VARIM was Loctite 770-NC and the cleaner was Loctite PMC.



Figure 3.5: Mold Cleanser and Releasing agent for the mold surface

FRP laminates: For civil infrastructure applications, the use of externally bonded fiber-reinforced polymers (FRP) has grown in popularity. By creating a perfect adhesive bond between the concrete and the wrapping material, the GFRP has significantly increased the strength and enhance the mechanical properties. The FRP laminates are used to enhance the mechanical properties that includes tensile, flexural and impact strength. The glass fiber mat (GF), WR310 was obtained from Owens Corning Inc., (India).



(a) *Glass fiber sheet*



(b) *Carbon fiber sheet*

Figure 3.6: Fiber sheets used in the experiment

Laminating sheet: These sheets are used in the experiment to cover the mold surface to avoid any leakage of the air so that the desired temperature is maintained for the experiment. The temperature should be monitored closely to avoid any errors in the result.



Figure 3.7: Laminating sheet used for covering the mold surface

Graphene Derivatives: The graphene derivatives were incorporated in small quantities in the GFRP and CFRP laminates to improve their mechanical properties. The graphene derivatives i.e., GO and rGO were purchased from AdNano Technologies Private Limited, Karnataka, India. These derivatives possess high aspect ratio and is a very thin exfoliated sheet. Due to easily dispersion in the solvent and other matrixes they were nano reinforced in the GFRP/CFRP laminates. GO is an oxidized form of graphene, laced with oxygen-containing groups whereas the rGO is made by the reduction of graphene oxide. As rGO are made by the reduction of GO, so it accounts for the most of oxygen groups removed from its chemical composition. The specifications for both GO and rGO are shown in Table 3.1.

Table 3.1. Specifications for GO and rGO used in the experiment

Parameter	GO	RGO
PURITY	~99%	~99%
THICKNESS (Z)	~0.8 - 2 nm	~0.8 - 2 nm
AVERAGE LATERAL DIMENSION (X&Y)	~5 – 10 μm	~5 – 10 μm
NUMBER OF LAYERS	1-3	1-3
CARBON CONTENT	~60 – 80%	~85 – 92%
HYDROGEN	~1 – 2%	~5 – 10%
NITROGEN CONTENT	~1 – 2%	~1 – 2%
SULPHUR CONTENT	< 1%	< 1%
SURFACE AREA	110 – 250* m^2/g	80 – 200* m^2/g
BULK DENSITY	0.5 g/cm^3	0.03 g/cm^3
CHEMICAL FORMULA	C	C
CAS NO.	7782-42-5	7782-42-5
PHYSICAL FORM	Fluffy, very light powder	Fluffy, very light powder
COLOR	Black powder	Black powder
ODOUR	Odorless	Odorless

3.4 FABRICATION OF GFRP/CFRP NANOCOMPOSITES

The procedure used for preparation of GFRP/CFRP sheets is detailed below:

Step 1: Weigh the epoxy resin, hardener, and graphene oxide in their respective quantities

Step 2: Add GO to the epoxy resin in the beaker and mix it with stirrer

Step 3: Mix the mixture with homogenizer at speed of 10,000 rpm for 10 mins. Homogenizer ensures uniform dispersion of graphene oxide in the epoxy matrix. The dispersed components are broken or subdivided into smaller particles, which are then evenly distributed throughout the mixture is done by homogenizer. Due to immiscibility and precipitation, the action of the homogenizer disrupts large particle formation on a continuous basis.



Figure 3.8: (a) Shear Homogenizer for uniform dispersion

Step 5: After completion of homogenization, it was followed by ultrasonication at 80% amplitude using the probe sonicator equipment. The time for the process of ultrasonication was 10 mins. During ultrasonication, the beaker enclosing epoxy-graphene oxide mixture was placed inside ice bath to avoid excessive increase in temperature. For successful processing of nanomaterials, probe sonication is strongly recommended. The nanoparticles are unable to settle in the mixture due to the

tremendous energy they absorb.



Figure 3.9: (a) Probe Sonicator machine (b) Digital display for the procedure

Step 6: After ultrasonication, hardener was poured to the composition at 40 °C. Then, mechanical stirrer was used for 10 mins at 500 rpm so that hardener is completely mixed leaving no solid particles behind.



Figure 3.10: Mechanical stirrer for mixing the hardener

Step 7: After the above step, the resin system prepared was used for infusion through the Vacuum Assisted Resin Infusion Molding (VARIM) set-up and the final step is to lay up the mixture over fibers. The VARIM (Vacuum Assisted Resin Infusion Molding) technique is a composite

manufacturing technology for producing high-quality large-scale components. A plastic vacuum bag is put on top of the mold and a dry preform fabric is put in it. A resin source and a vacuum pump are connected to the first side of the mold. The vacuum created by the mold pulls the liquid resin into the reinforcing fibers. The impregnation process is completed with curing and de-molding steps.

Step 7: Procedure followed in VARIM (Vacuum Assisted Resin Infusion Molding)

- The very first step is to clean the VARIM table with a cleansing agent and followed by the coating of a release anti-adhesive agent, preventing sticking the molded part to the mold surface.
- After that, breather cloth was placed on the vacuum ports of the VARIM table to build or form a connection with the ports.
- Now, a layer of glass fiber polymer with dimension (45cm*30cm) is placed on the table and the epoxy resin mixture is applied by pouring and brushing. Now, place the required number of plies of glass fiber mat or carbon fiber mat on the VARIM mold. Now, we need to note that 02 plies were taken for the fabrication of tensile samples and a total of 12 plies were used for impact strength and flexural toughness samples. The mixture will be applied to each ply so that a flow medium is created.
- Then separate cloth was lay down on the topmost layer of the fiber followed by the perforated sheet to allow the uniform dispersion of resin and then it was covered by infusion mesh to provide the path for the flow. Post that breather was placed over the wire mesh to form a bridge with the breather connection. Finally, the laminating sheet was placed over the set up and fixed with the help of sealant tape.
- After the setup was completed, the vacuum pump was switched on and the pressure was maintained within 5 millibars. We need to ensure that it was fully vacuumed, and no leakage was there. The resin was then infused with the use of vacuum which was then followed by curing and post curing of 6 hours at 35 °C and 15 hours at 60°C respectively, hence making it total of 21 hours.
- After removing all the layers, a thick fiber mat sheet is prepared.



Figure 3.11: Preparation of nano reinforced fiber sheets on VARIM

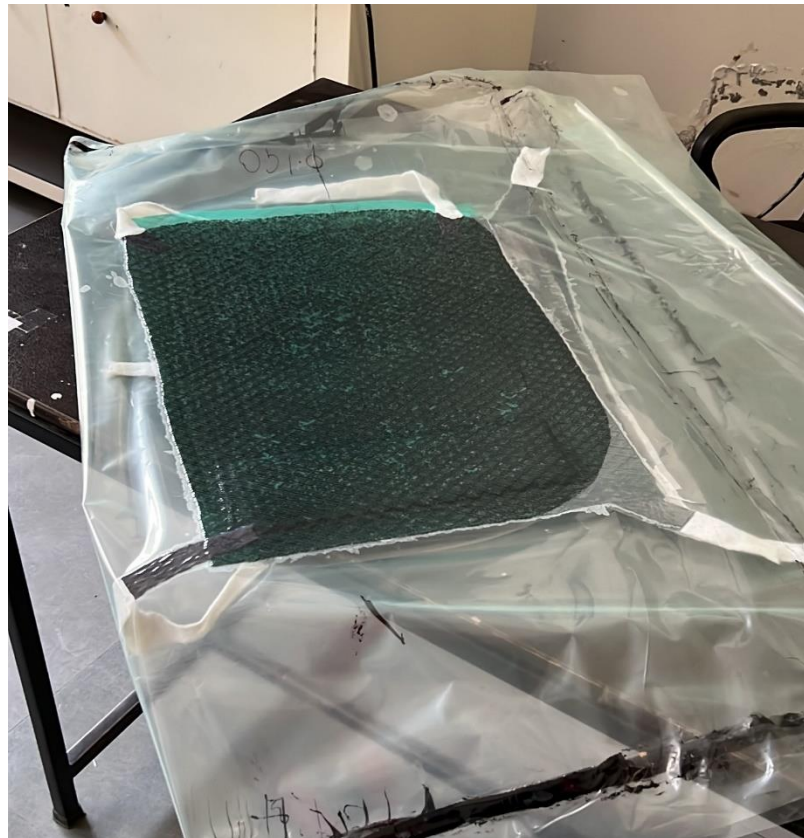


Figure 3.12: Fiber sheets de molded from the surface

3.5 TESTING OF SAMPLES

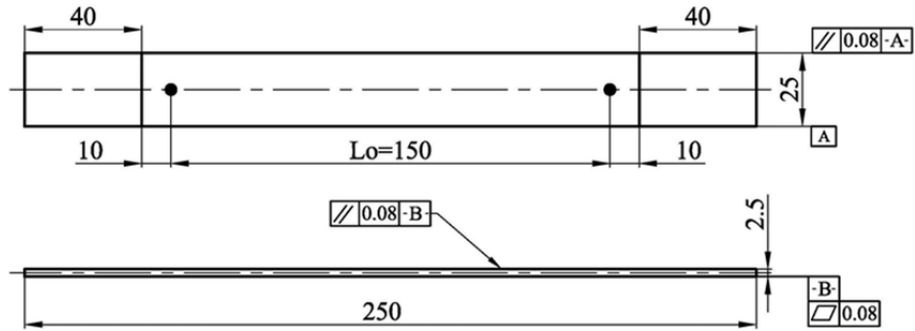
Three types of test samples were prepared to check the overall performance of the graphene derivatives nano reinforced CFRP/GFRP laminates.

- Tensile Strength test
- Flexural Strength test
- Impact Strength test

3.5.1 Tensile Strength test

The tensile strength, yield strength, and ductility of the metallic material are all assessed via tensile testing, which is a destructive test process. It quantifies how much the composite or plastic piece stretches or elongates up until it breaks at a certain point. The tensile testing used in this process is as per specifications of ASTM D3039. ASTM D3039 tensile testing is used to measure the force required to break a polymer composite specimen and the extent to which the specimen stretches or elongates to that breaking point. Tensile tests produce a stress-strain diagram, which is used to determine tensile modulus. In the grips of a mechanical testing machine, a thin flat strip of material is mounted and monotonically loaded in tension while recording load. The maximal weight supported before failure can be used to calculate the material's ultimate strength. The in-plane tensile qualities of polymer matrix composite materials reinforced with high-modulus fibers are evaluated using this test method. Continuous fiber or discontinuous fiber-reinforced composites with a laminate that is balanced and symmetric in terms of the test direction are the only composite material forms allowed. In accordance with ASTM D3039 (2 ply samples) were prepared and tested using Universal Testing machine.

The tensile testing is done with Universal Testing Machine (UTM) for composites in Mechanical Department lab, UIET, PU, Chandigarh. The size of the sample is 250mm in length and 25mm in width and 0.65-0.7mm thickness. The crosshead speed was used as 2mm/min. Five samples of each composition were used to calculate average tensile strength values. The tensile testing specimens' dimension is shown in the Figure 3.13 and 3.14.



*All Dimensions are in mm
According to ASTM D 3039-17 Coupon without Tabs*

Figure 3.13: Sample specifications for tensile testing

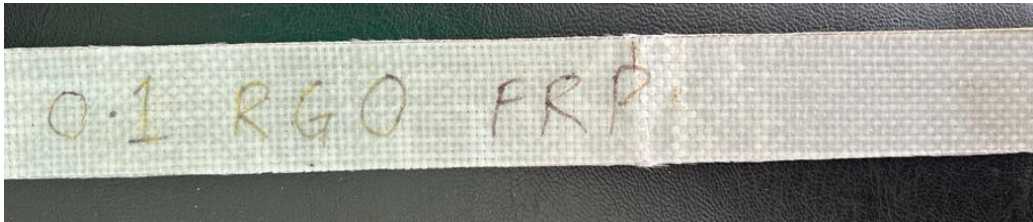


Figure 3.14: Sample before tensile testing



Figure 3.15: Universal Testing machine for tensile testing (PU, Chandigarh)

3.5.2 Flexural testing

The resistance to flexing or stiffness of a material is determined by flexural testing, which measures the force required to bend a beam of plastic material. The flexural testing used in this process is with specifications as ASTM D790. It measures the flexural properties of a material while under a bending strain or deflection. This test is conducted on a universal testing system using a three-point bend fixture at a rate proportional to the depth of the specimen. ASTM D790 testing is used to determine the flexural strength/stress. Flexural properties of unreinforced and reinforced plastics, as well as high-modulus composites and electrical insulating materials in the form of rectangular bars molded directly or cut from sheets, plates, or mold forms are tested using these test methods. Both stiff and semirigid materials may be tested using these test methods. Flexural properties as determined by these test methods are especially useful for quality control and specification purposes.

The flexural properties were evaluated using three-point bend test as per ASTM D790 standards at a constant crosshead speed of 1mm/min. The specimens may be cut from sheets, plates, or molded shapes, or may be molded to the desired finished dimensions.

The Flexural testing was done in the Central Institute of Petrochemicals Engineering and Technology (CIPET), Amritsar. The size of the sample is 127mm in length, 12.7mm in width and 3.2-3.5mm thickness. Five samples of each composition were used to calculate average flexural strength values. The specifications of sample are shown in figure 3.16 and 3.17.

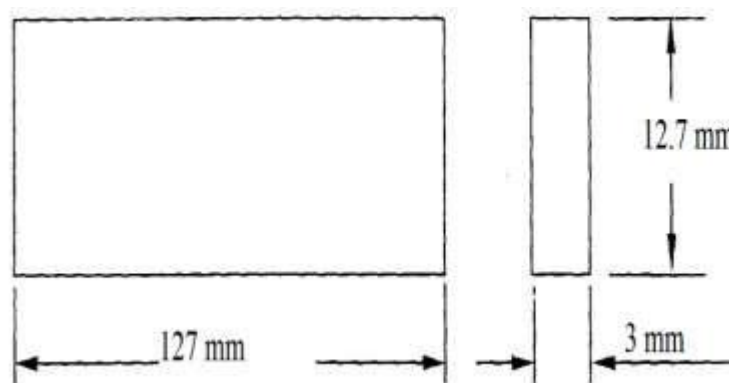


Figure 3.16: Sample specifications for flexural testing



Figure 3.17: Sample for flexural testing



Figure 3.18: Test set up for flexural testing (Cipet, Amritsar)

3.5.3 Impact Testing

The toughness of material is studied using impact tests. The ability of a material to store energy during plastic deformation is known as its toughness. Because of the small amount of plastic deformation that brittle materials can tolerate, they have low toughness. The impact testing used in this process is with specifications as ASTM D256. The notched Izod impact test to ASTM D256 generates characteristic values for the impact strength and notch sensitivity at high strain rates in the form of a thickness-related energy value. A pendulum hammer with defined energy capacity and drop height, which expends a portion of its kinetic energy when it strikes the specimen, is used as the measuring technique. Therefore, following the impact, the pendulum hammer does not return to its original drop height. The energy absorbed is represented by the measured height difference between drop height and peak height. Since the gripping force can affect the result, pneumatic gripping or control of the gripping force is useful. The pendulum impact test indicates the energy to break

standard test specimens of specified size under stipulated parameters of specimen mounting, notching, and pendulum velocity-at-impact.

The impact properties were studied as per ASTM D256 using ATS FAAR Izod set up of 5J capacity (ATS FAAR, Italy). The specimen is positioned horizontally and is broken with one oscillation only of the hammer, the impact line is centered between the two specimen supports. The impact testing was done in the Central Institute of Petrochemicals Engineering and Technology (CIPET), Amritsar. The size of the sample is 63.5mm in length, 12.7mm in width and 3.2-3.5mm thickness. The sample for impact testing is of 12 ply thickness. Five samples were taken for the testing. Five samples of each composition were used to calculate average impact strength values. The specifications of sample are shown in Figure 3.19 and 3.20.

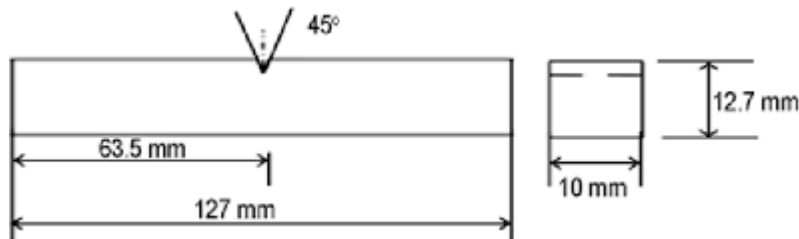


Figure 3.19: Sample dimension for impact testing



Figure 3.20: Sample for impact testing

3.6 CLOSING REMARKS

The chapter discuss about the experimental program and methodology involving nano reinforcement of GO/rGO in Glass and Carbon fiber polymer. The experimental details of various tests and methodology to monitor and evaluate the performance of the structure is discussed.

4

RESULTS AND DISCUSSION

4.1 GENERAL

The objective is to check the overall performance for Glass and Carbon fiber reinforced polymer laminates in which graphene derivatives are reinforced with epoxy. Various tests have been conducted to check the mechanical properties such as tensile strength, flexural strength, and impact strength. The different concentration of graphene derivatives (Go & rGO) in FRP composites have been discussed in this chapter to check their effect on mechanical behavior.

4.2 CHARACTERIZATION OF GRAPHENE DERIVATIVES

Surface morphology and structural characteristics of Graphene oxide and reduced graphene oxide:

Morphology of Graphene Oxide was studied using scanning electron microscopy (SEM). XRD analysis was also conducted to know the crystalline planes existing in the nano-reinforcements to be used. Figure 4.2 and figure 4.4 shows the XRD spectra for GO and rGO. The SEM images of both GO and rGO shows sheet like morphology as shown in figure 4.1 and 4.3. The XRD spectra for GO show $2\theta = 11^\circ$ and for rGO a sharp peak emerges at $2\theta = 25.2^\circ$. Both of the sharp peaks shows that sample is quite crystalline in nature.

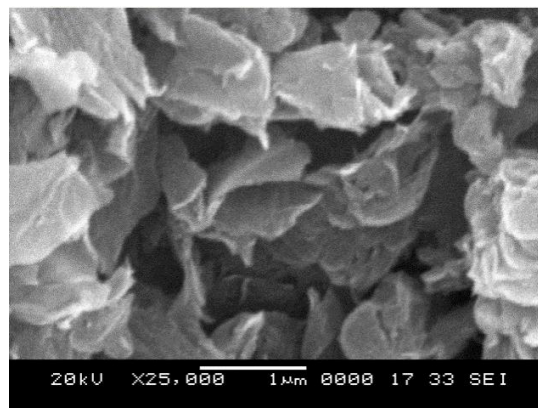


Figure 4.1: SEM images of graphene oxide

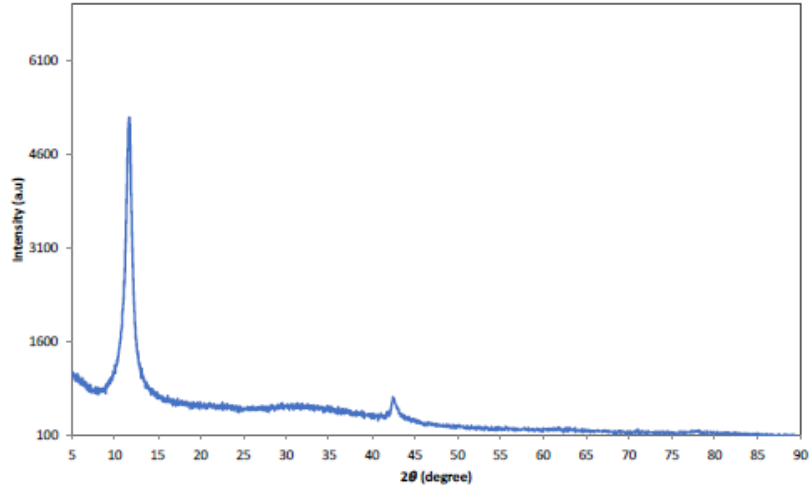


Figure 4.2: XRD images of graphene oxide

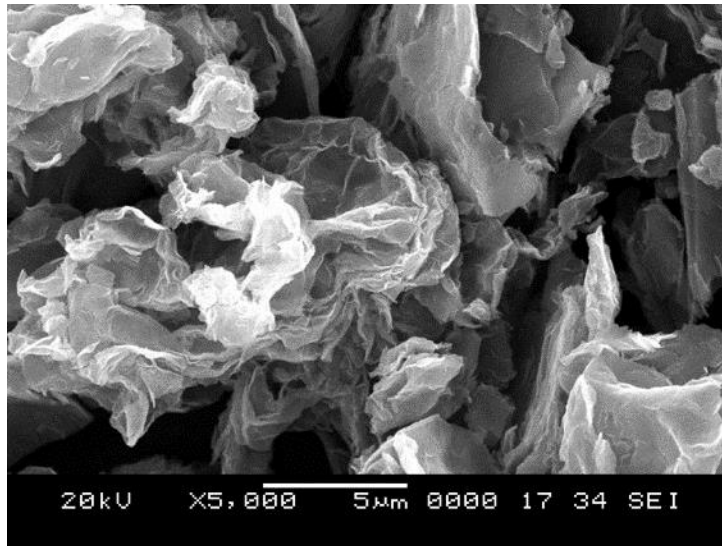


Figure 4.3: SEM images of reduced graphene oxide

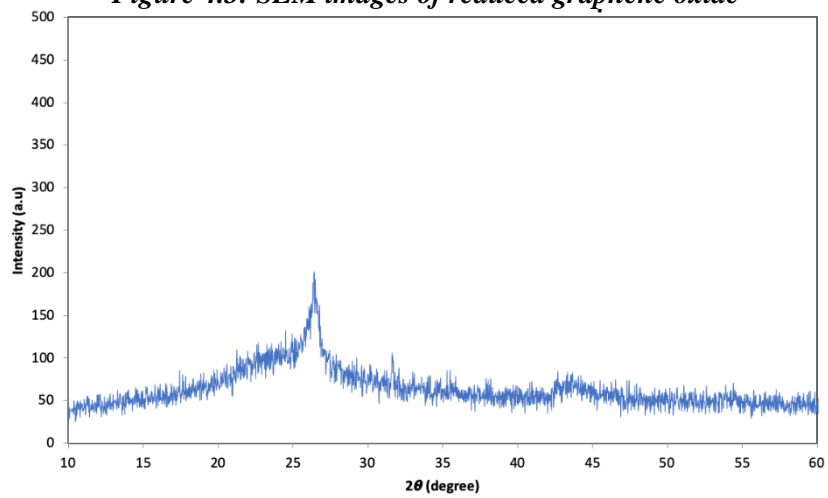


Figure 4.4: XRD images of reduced graphene oxide

4.3 NANO-MODIFIED GFRP POLYMERS

Glass fiber sheets were prepared in which the epoxy resin was nano reinforced with graphene derivatives i.e., graphene oxide (GO) and reduced graphene oxide (rGO) with different concentration in the range 0-0.3 (parts per hundred resin), in increments of 0.1 phr. In the table 4.1, the weight of graphene derivatives is shown corresponding to the different concentrations taken in phr.

Table 4.1. Test matrix for GO/rGO samples

S.no	CONCENTRATION (phr)	GO/rGO (By Weight in grams)
1.	0.3	0.56
2.	0.2	0.37
3.	0.1	0.19
4.	Without GO/rGO	-

4.3.1 GFRP/GO modified

Graphene oxide was nano reinforced in the glass fiber reinforced polymer composite.

TENSILE TESTING

Tensile testing provides us the tensile strength and tensile stress. Tensile strength measures the maximum force required to break any specimen whereas the tensile stress means the extent to which the specimen can stretch to that breaking point. The tensile strength is not the ideal parameter as it can vary with the change in cross section area whereas stress is irrespective of the cross-section area. In the figure 4.6, the sample breaks from the middle after testing and it is the maximum force the sample can withstand. We carried out tensile strength for different concentrations and five specimens for each concentration were taken. A graph is plotted to find out the optimum concentration where the maximum tensile strength is obtained.

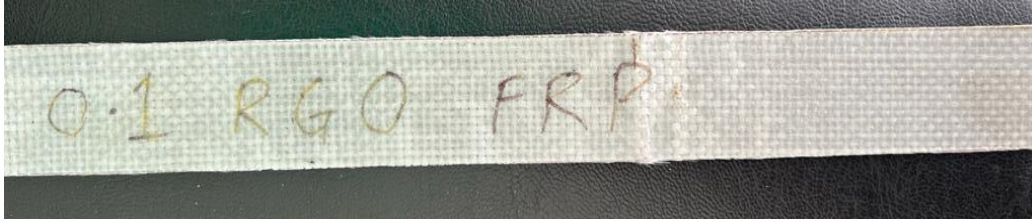


Figure 4.5: Glass fiber sample before tensile testing

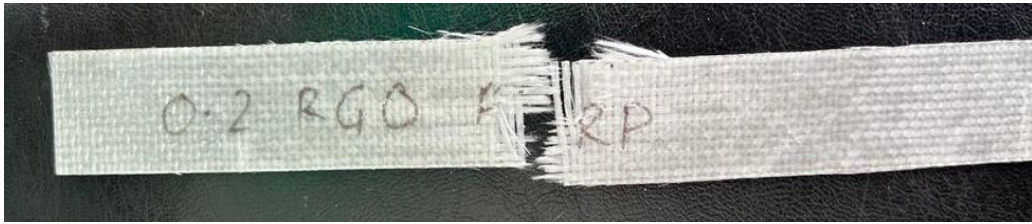


Figure 4.6: Glass fiber after the tensile testing

The weight of graphene oxide was 0.19g and the epoxy hardener were in ration 10:1 with weight as 170g and 17g respectively (Epoxy: Hardener is recommended by supplier)

Table 4.2. Variation in Tensile strength with different concentration of GO in GFRP laminates

	Samples	Tensile Load (kN)	Tensile Strength (N/mm²)
GFRP	-	-	1.39
GFRP/0.1 GO	S1	7.60	1.56
	S2	8.20	
	S3	8.04	
	S4	8.05	
	S5	7.10	
	Average	7.81	
GFRP/0.2 GO	S1	7.66	1.48
	S2	7.47	
	S3	7.03	
	S4	7.52	
	S5	7.37	
	Average	7.41	
GFRP/0.3 GO	S1	7.08	1.40
	S2	6.77	
	S3	6.75	
	S4	6.9	
	S5	7.43	
	Average	6.99	

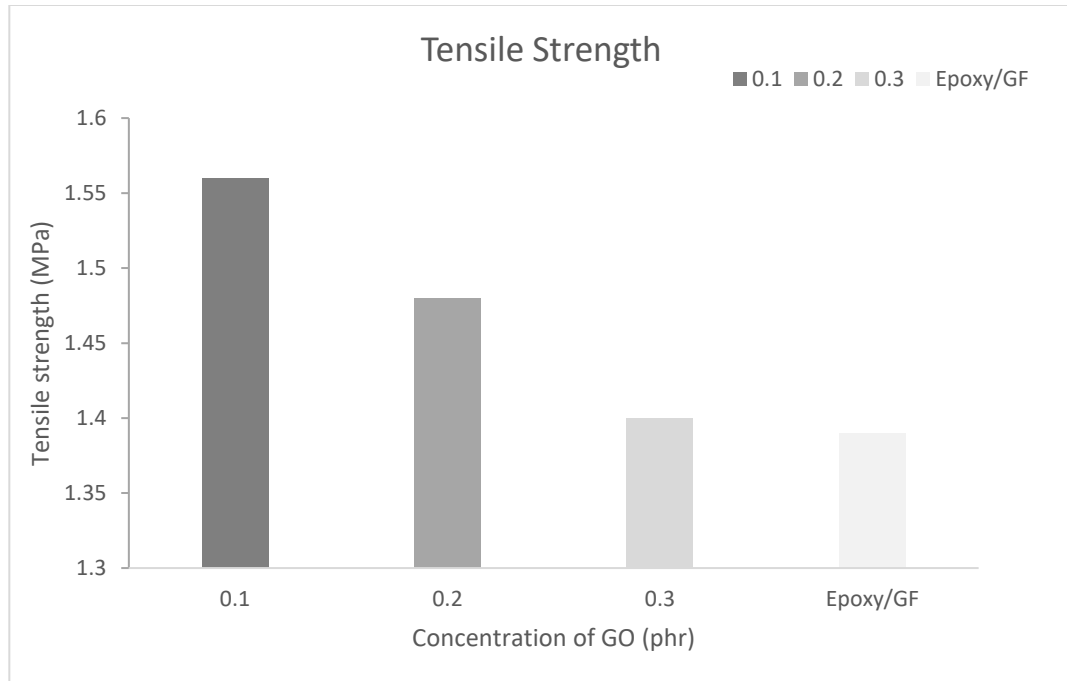


Figure 4.7: Tensile strength results of GO reinforced glass fiber composites

From the above results, we observed that we have the maximum tensile strength with GO concentration 0.1 phr i.e., 1.56 N/mm² whereas with concentration 0.2 phr and 0.3 phr and epoxy/GF have tensile strength as 1.48 N/mm², 1.40 N/mm² and 1.39 N/mm² respectively. We observed an increase of 12% when the GO at concentration at 0.1 phr was included in the matrix as compared to the epoxy/GF matrix.

FLEXURAL STRENGTH

Flexural strength is considered as the material's ability to resist the breakage and cracking under the influence of bending stress. Basically, it is the capacity of materials to resist bending deflection when an energy is applied to the structure. These test measures how strong or stiff the material is. With high flexural strength, the composite or material has high capability to resist the deformation whenever the force is applied whether in tension or compression. When the material has low flexural strength, the material may not be appropriate for long term use. We carried out flexural strength for different concentrations and five specimens for each concentration were taken. A graph is plotted to find out the suitable concentration where we achieve the maximum flexural strength.

Table 4.3. Variation in Flexural strength with different concentration of GO in GFRP laminates

	Samples	Flexural Strength (MPa)	Average Flexural Strength (MPa)
GFRP	-	210	210
GFRP/0.1 GO	S1	248.76	280
	S2	271.54	
	S3	308.36	
	S4	298.69	
	S5	271.16	
GFRP/0.2 GO	S1	271.67	265
	S2	266.44	
	S3	252.11	
	S4	278.41	
	S5	257.38	
GFRP/0.3 GO	S1	244.49	253
	S2	270.26	
	S3	294.13	
	S4	250.67	
	S5	205.75	

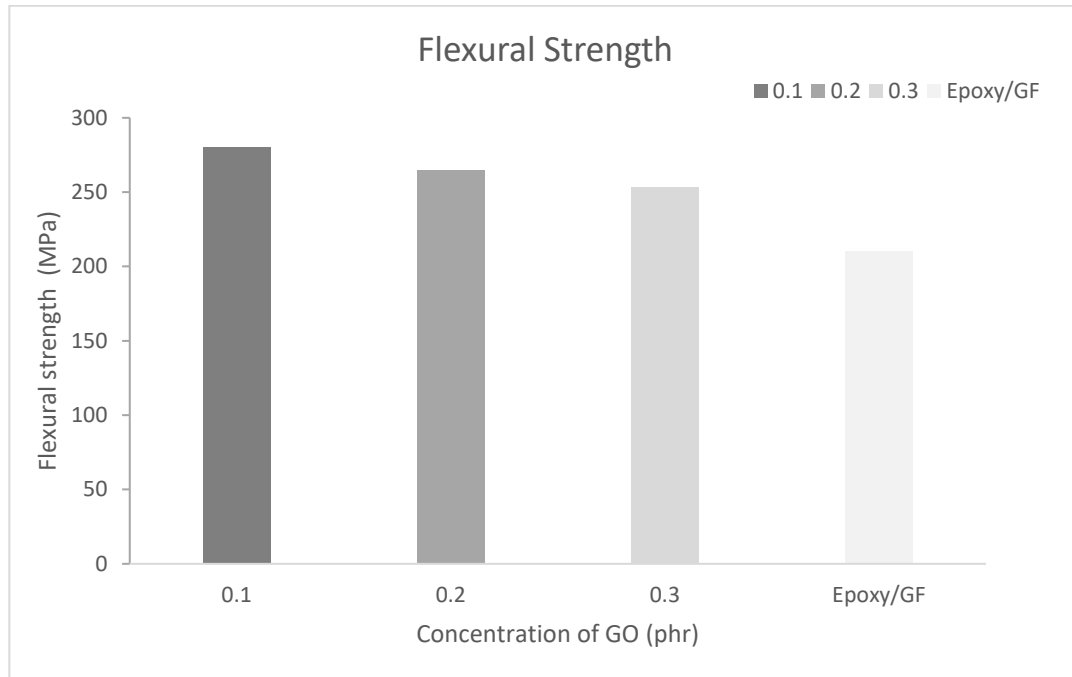


Figure 4.8: Flexural strength results of GO reinforced glass fiber composites

From the above results, we observed that we have the maximum flexural strength with GO reinforcement at concentration at 0.1 phr i.e., 280 MPa whereas with concentration 0.2 phr and 0.3 phr and epoxy/GF have flexural strength as 265 MPa, 253 MPa and 210 MPa respectively. We compute that 0.1 phr has an increase of 33% as compared to epoxy/GF matrix.

IMPACT TESTING

Impact testing is the capability of the material to overcome any type of impact, it can be impact at flaws, notches, or cracks. The kinetic energy required to start fracture and maintain it until the object breaks is known as Izod impact. To prevent deformity of the specimen after impact, Izod specimens are notched. This test might be used to check whether a material possesses specified impact qualities or to compare materials for overall toughness. Generally, brittle materials have low toughness as they can endure a small amount of deformation for that reason, we utilized the nano particles like GO/rGO.

We carried out impact strength for different concentrations and five specimens for each concentration were taken. A graph is plotted to find out the optimum concentration to get the maximum impact strength.

Table 4.4. Variation in Impact strength with different concentration of GO in GFRP laminates

	Samples	Impact Strength (kJ/m ²)	Average Impact Strength (kJ/m ²)
GFRP	-	145	145
GFRP/0.1 GO	S1	170.43	170
	S2	156.47	
	S3	186.04	
	S4	172.97	
	S5	163.45	
GFRP/0.2 GO	S1	112.91	118
	S2	120.47	
	S3	114.76	
	S4	118.11	
	S5	121.65	
GFRP/0.3 GO	S1	124.7	112
	S2	105.67	
	S3	112.96	
	S4	99.43	
	S5	120.84	

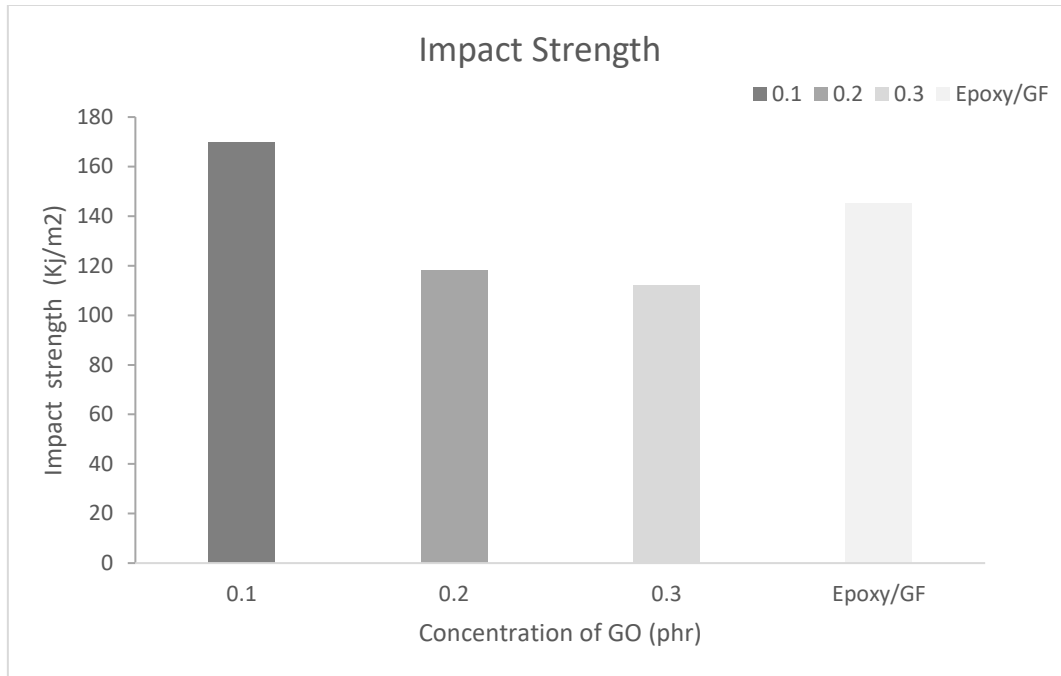


Figure 4.9: Impact strength results of GO reinforced glass fiber composites

From the above results, we observed that we have the maximum impact strength with GO concentration 0.1 phr i.e., 170 kJ/m² whereas with concentration 0.2 phr and 0.3 phr and epoxy/GF have impact strength as 117 kJ/m², 112 kJ/m² and 145 kJ/m² respectively. We observed that 0.1 phr has an increase of 17% as compared to epoxy/GF matrix.

4.3.2 GFRP/rGO Modified

Reduced Graphene oxide (rGO) was nano reinforced in the glass fiber-based polymer composites.

TENSILE TESTING

For tensile testing, various specimens were prepared with different concentrations of rGO, and five specimens were taken from each sample of different rGO based compositions.

Table 4.5. Variation in Tensile strength with different concentration of rGO in GFRP laminates

	Samples	Tensile Load (kN)	Tensile Strength (N/mm ²)
GFRP	-	-	1.39
GFRP/0.1 rGO	S1	7.54	1.65
	S2	8.49	
	S3	8.54	

	S4	8.32	
	S5	8.45	
	Average	8.27	
GFRP/0.2 rGO	S1	7.15	1.41
	S2	7.15	
	S3	7.07	
	S4	6.94	
	S5	7.10	
	Average	7.06	
GFRP/0.3 rGO	S1	6.97	1.42
	S2	7.21	
	S3	7.35	
	S4	6.84	
	S5	7.22	
	Average	7.12	

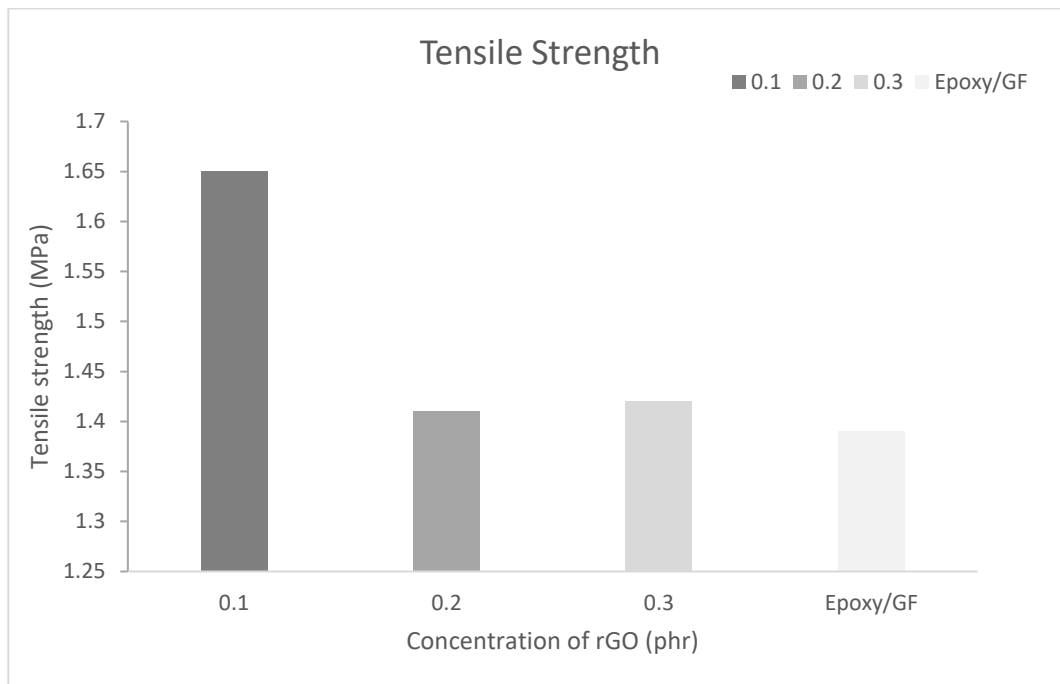


Figure 4.10: Tensile strength results of rGO reinforced glass fiber composites

From the above results, we observed that 0.1 phr has the maximum tensile strength of 1.65 N/mm² as compared to 0.3 phr and 0.2 phr that have tensile strength of 1.41 N/mm², 1.42 N/mm². The epoxy/GF matrix has tensile strength of 1.39 N/mm². We observed that 0.1 phr has an increase of 18% as compared to epoxy/GF matrix.

FLEXURAL TESTING

For flexural testing, various specimens were prepared with different concentrations of rGO, and five samples were taken from each specimen.

Table 4.6. Variation in Flexural strength with different concentration of rGO in GFRP laminates

	Samples	Flexural Strength (MPa)	Average Flexural Strength (MPa)
GFRP	-	210	210
GFRP/0.1 rGO	S1	250.69	254
	S2	260.56	
	S3	259.93	
	S4	249.91	
	S5	247.1	
GFRP/0.2 rGO	S1	259.93	246
	S2	250.69	
	S3	217.71	
	S4	244.49	
	S5	257.38	
GFRP/0.3 rGO	S1	261.17	238
	S2	197.71	
	S3	256.54	
	S4	256.19	
	S5	219.8	

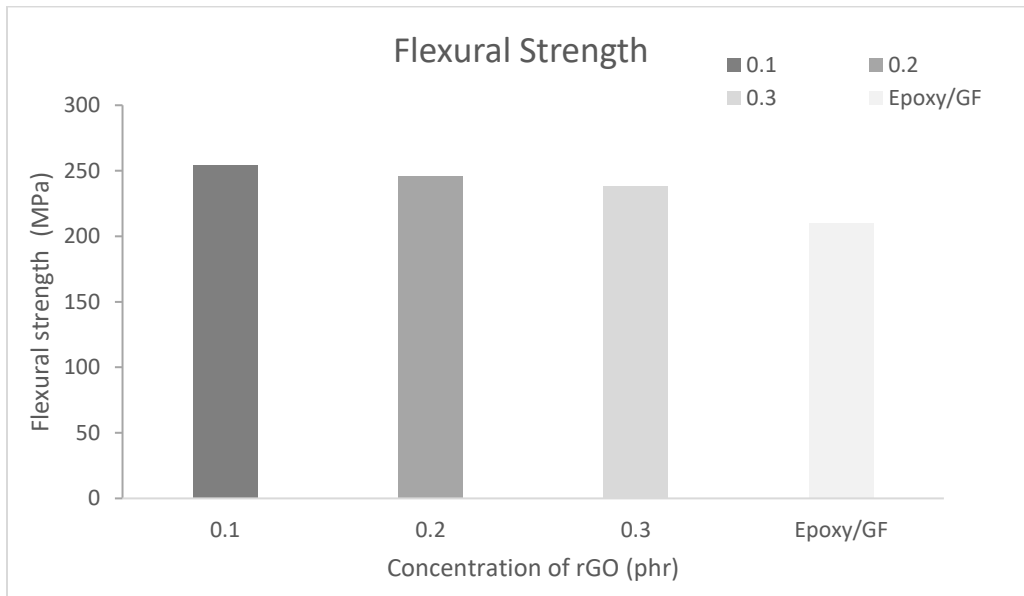


Figure 4.11: Flexural strength results of rGO reinforced glass fiber composites

From the above results, we observed that we have the maximum tensile strength with rGO concentration 0.1 phr i.e., 254 MPa whereas with concentration 0.2 phr and 0.3 phr and epoxy/GF have tensile strength as 238 MPa, 246 MPa and 210 MPa respectively. We observed an increase of 20% when the rGO at concentration at 0.1 phr was included in the matrix as compared to the epoxy/GF matrix.

IMPACT TESTING

For impact testing, various specimens were prepared with different concentrations of rGO, and five samples were taken from each specimen.

Table 4.7. Variation in Impact strength with different concentration of rGO in GFRP laminates

	Samples	Impact Strength (kJ/m ²)	Average Impact Strength (kJ/m ²)
GFRP	-	145	145
GFRP/0.1 rGO	S1	162.47	155
	S2	170.05	
	S3	158.21	
	S4	124.43	
	S5	161.34	
GFRP/0.2 rGO	S1	134.07	129
	S2	118.53	
	S3	120.6	
	S4	135.97	
	S5	130.1	
GFRP/0.3 rGO	S1	112.97	120
	S2	116.47	
	S3	120.1	
	S4	121.98	
	S5	129.11	

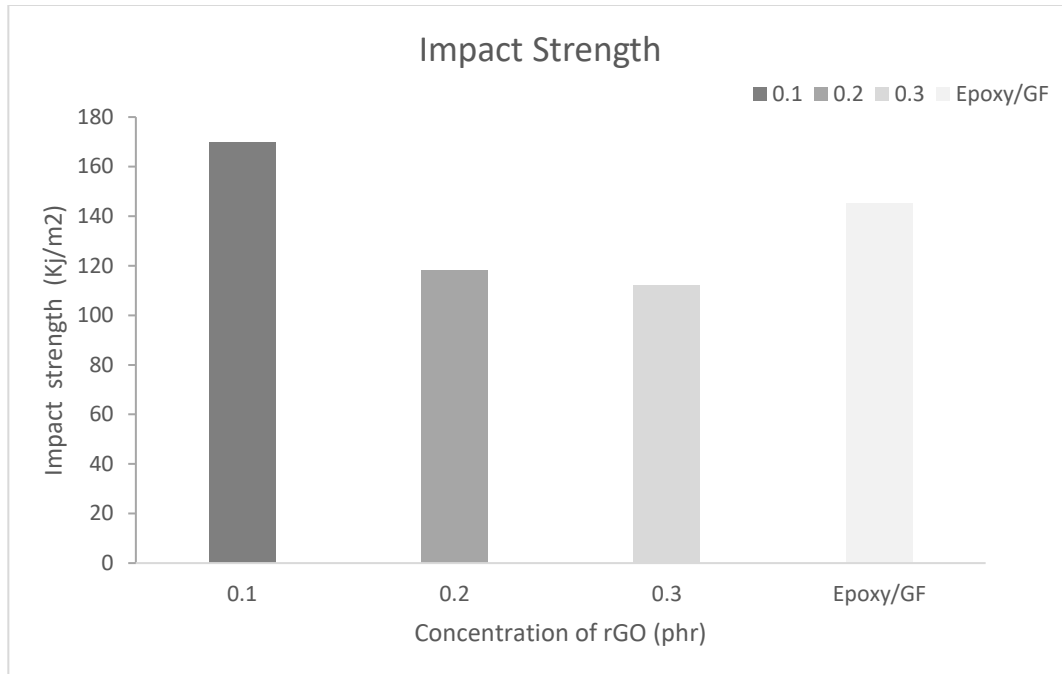


Figure 4.12: Impact strength results of rGO reinforced glass fiber composites

From the above results, we observed that we have the maximum impact strength with rGO concentration 0.1 phr i.e., 155 kJ/m² whereas with concentration 0.2 phr and 0.3 phr and epoxy/GF have impact strength as 129 kJ/m², 120 kJ/m² and 145 kJ/m² respectively. We observed that 0.1 phr has an increase of 6% as compared to epoxy/GF matrix.

4.4 NANO-MODIFIED CFRP POLYMERS

Carbon fiber reinforced polymer (CFRP) wrap is made to repair and strengthen structural concrete components, and CFRP wrap is a high-strength unidirectional carbon fiber wrap pre-saturated. Carbon fiber sheets were prepared in which the epoxy resin nano reinforced with graphene derivatives i.e., graphene oxide (GO) and reduced graphene oxide (rGO) with different concentration in the range 0-0.3 (parts per hundred resin), in increments of 0.1 phr.

4.4.1 CFRP/GO modified

Graphene oxide (GO) was nano reinforced in the carbon fiber reinforce polymer to enhance their properties. The GO were incorporated in the small amounts in the CFRP laminates.

TENSILE TESTING

Carbon fiber sheets were prepared and were to check for the tensile testing. For testing the sample were cut from the sheet with dimension 250mm length, 25mm width and 2mm thickness. Five samples for each concentration and then were analyzed by plotting the graph with the maximum tensile strength.



Figure 4.13: Carbon fiber sample before the tensile testing



Figure 4.14: Carbon fiber sample after the tensile testing

Table 4.8. Variation in Tensile strength with different concentration of GO in CFRP laminates

	Samples	Tensile Load (kN)	Tensile Strength (N/mm ²)
CFRP	S1	17.10	3.77
	S2	20.48	
	S3	19.49	
	S4	19.80	
	S5	17.26	
	Average	18.83	
CFRP/0.1 GO	S1	20.66	3.69
	S2	16.95	
	S3	17.10	
	S4	18.86	
	S5	18.53	
	Average	18.42	

CFRP/0.2 GO	S1	19.68	3.95
	S2	18.94	
	S3	20.71	
	S4	20.19	
	S5	16.75	
	Average	19.25	
CFRP/0.3 GO	S1	18.60	3.57
	S2	18.58	
	S3	16.70	
	S4	19.80	
	S5	15.68	
	Average	17.87	

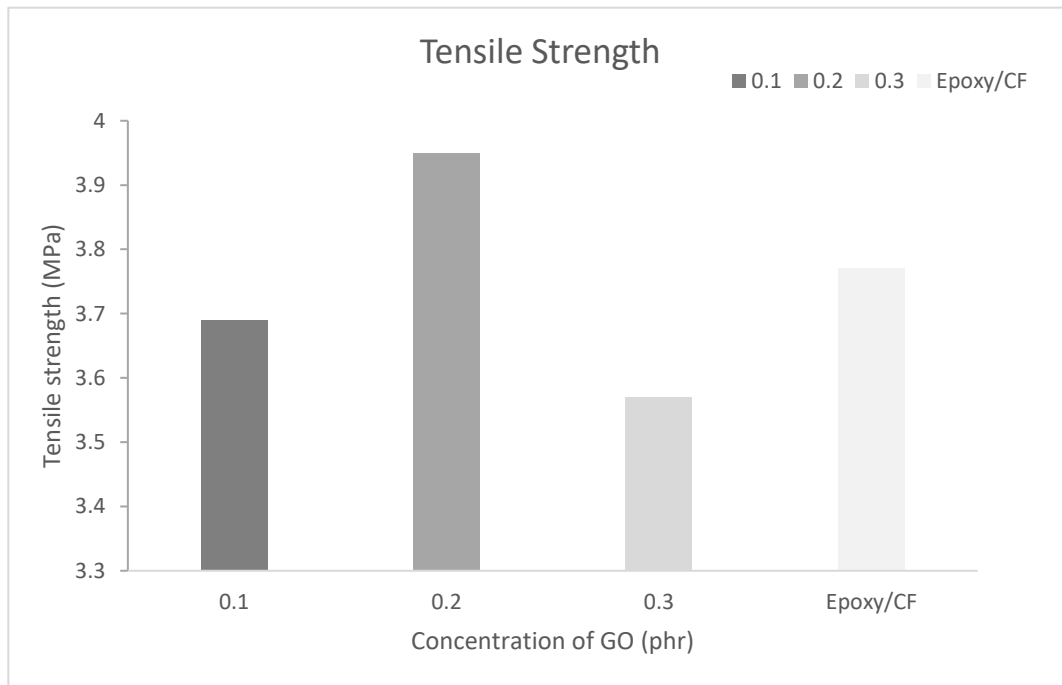


Figure 4.15: Tensile strength results of GO reinforced carbon fiber composites

From the above results, we observed that 0.2 phr has the maximum tensile strength of 3.85 N/mm² as compared to 0.1 phr and 0.3 phr that have tensile strength of 3.69 N/mm², 3.57 N/mm². The epoxy/CF matrix has tensile strength of 3.77 N/mm². We observed that 0.2 phr has an increase of 5% with reference to epoxy/CF matrix.

FLEXURAL TESTING

Carbon fiber sheets were prepared and were to check for the flexural testing. For testing, the sample were cut from the sheet with dimension 127mm length, 13mm width and 3.4mm thickness. Five samples for each concentration were taken and then plotted the graph for flexural strength.

Table 4.9. Variation in Flexural strength with different concentration of GO in CFRP laminates

	Samples	Flexural Strength (MPa)	Average Flexural Strength (MPa)
CFRP	S1	361.21	499
	S2	585.68	
	S3	506.38	
	S4	496.27	
	S5	544.79	
CFRP/0.1 GO	S1	457.99	477
	S2	491.29	
	S3	377.38	
	S4	525.02	
	S5	536.33	
CFRP/0.2 GO	S1	576.46	602
	S2	631.47	
	S3	667.65	
	S4	658.01	
	S5	477.77	
CFRP/0.3 GO	S1	406.79	450
	S2	432.26	
	S3	452.8	
	S4	493.63	
	S5	457.34	

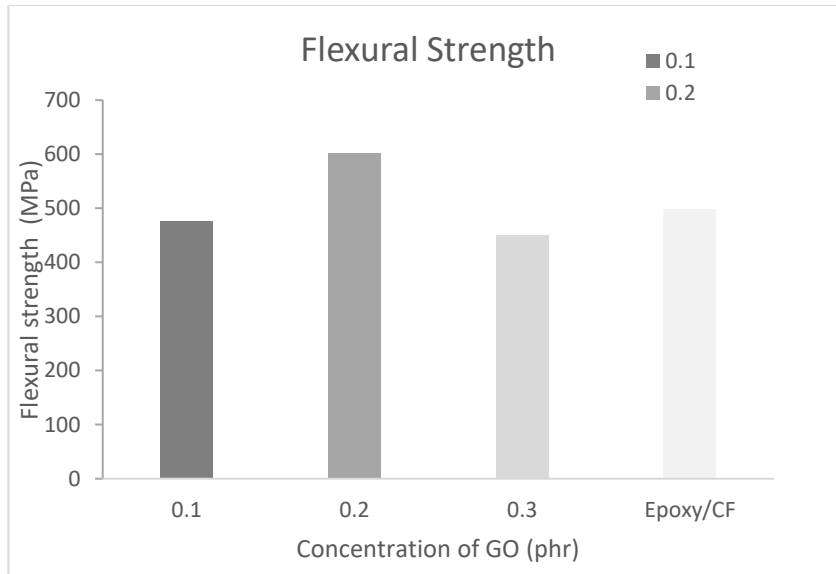


Figure 4.16: Flexural strength results of GO reinforced carbon fiber composites

From the above results, we observed that we have the maximum tensile strength with GO concentration 0.2 phr i.e., 602 MPa whereas with concentration 0.1 phr and 0.3 phr and epoxy/CF have tensile strength as 477 MPa, 450 MPa and 499 MPa respectively. We observed an increase of 20% when the GO at concentration at 0.2 phr was included in the matrix as compared to the epoxy/CF matrix.

Impact Strength

Carbon fiber sheets were prepared and were to check for the impact testing. For testing the sample were cut from the sheet with dimension 64mm length, 13mm width and 3.4mm thickness. Five samples for each concentration were taken and plotted the graph for the impact strength.

Table 4.10. Variation in Impact strength with different concentration of GO in CFRP laminates

	Samples	Impact Strength (kJ/m ²)	Impact Flexural Strength (kJ/m ²)
CFRP	S1	84.93	87
	S2	104.79	
	S3	95.99	
	S4	66.49	
	S5	78.31	
CFRP/0.1 GO	S1	44.79	62
	S2	67.4	
	S3	75.11	

	S4	59.65	
	S5	59.22	
CFRP/0.2 GO	S1	146.69	144
	S2	141.86	
	S3	149.96	
	S4	141.87	
	S5	140.15	
CFRP/0.3 GO	S1	92.79	120
	S2	134.16	
	S3	176.09	
	S4	98.99	
	S5	97.31	

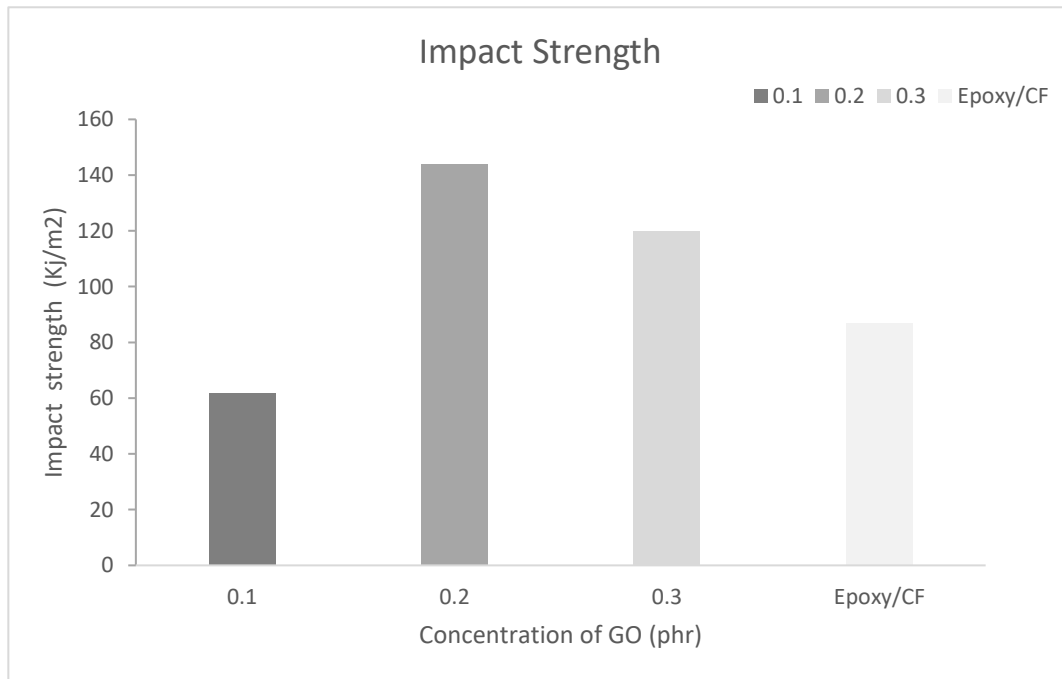


Figure 4.17: Impact strength results of GO reinforced carbon fiber composites

From the above results, we observed that we have the maximum impact strength with GO concentration 0.2 phr i.e., 144 kJ/m² whereas with concentration 0.1phr and 0.3 phr and epoxy/CF have impact strength as 62 kJ/m², 120 kJ/m² and 87 kJ/m² respectively. We observed that 0.1 phr has an increase of 65% as compared to epoxy/CF matrix.

4.4.2 CFRP/rGO modified

Reduced Graphene oxide (rGO) was nano reinforced in the carbon fiber reinforce polymer.

TENSILE TESTING

For tensile testing, various specimens were prepared with different concentrations of rGO, and five samples were taken from each specimen. The dimension was 250mm length, 25mm width and 0.5mm thickness.

Table 4.11. Variation in Tensile strength with different concentration of rGO in CFRP laminates

	Samples	Tensile Load (kN)	Tensile Strength (N/mm ²)
CFRP	S1	17.10	3.77
	S2	20.48	
	S3	19.49	
	S4	19.80	
	S5	17.26	
	Average	18.83	
CFRP/0.1 rGO	S1	17.6	3.44
	S2	17.67	
	S3	19.35	
	S4	13.62	
	S5	17.85	
	Average	17.22	
CFRP/0.2 rGO	S1	18.69	3.78
	S2	20.96	
	S3	20.11	
	S4	16.86	
	S5	17.9	
	Average	18.90	
CFRP/0.3 rGO	S1	19.69	4.30
	S2	22.34	
	S3	20.67	
	S4	22.44	
	S5	22.27	
	Average	21.48	

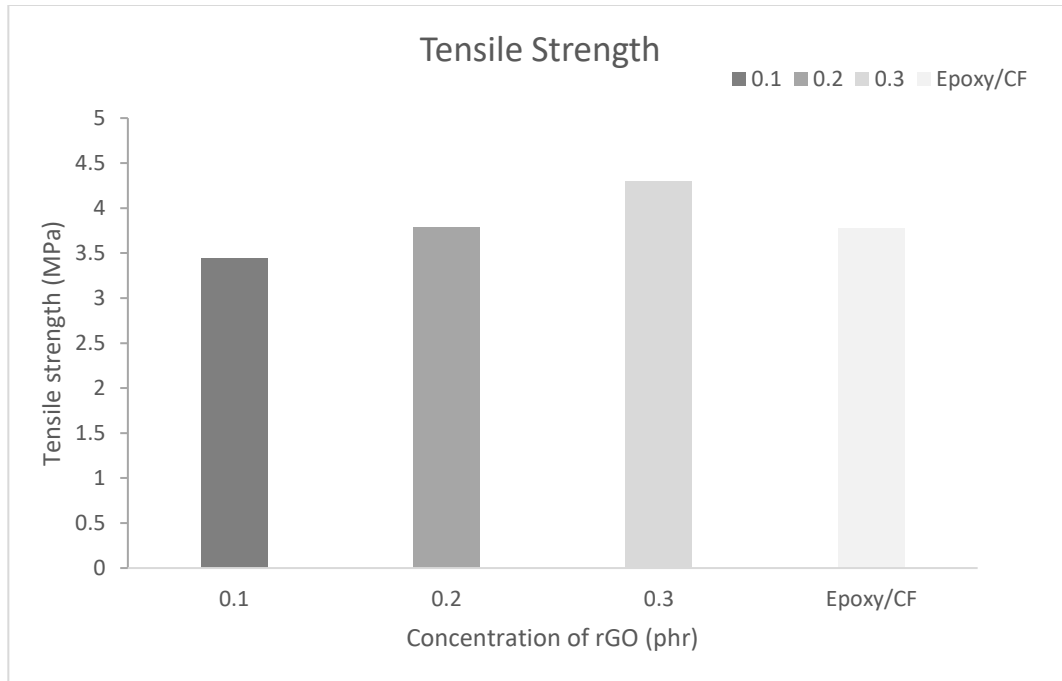


Figure 4.15: Tensile strength results of rGO reinforced carbon fiber composites

From the above results, we observed that 0.3 phr has the maximum tensile strength of 4.30 N/mm² as compared to 0.1 phr and 0.2 phr that have tensile strength of 3.44 N/mm², 3.78 N/mm². The epoxy/CF matrix has tensile strength of 3.77 N/mm². We observed that 0.2 phr has an increase of 14% with reference to epoxy/CF matrix.

FLEXURAL TESTING

For flexural testing, various specimens were prepared with different concentrations of rGO, and five samples were taken from each specimen.

Table 4.12. Variation in Flexural strength with different concentration of rGO in CFRP laminates

	Samples	Flexural Strength (MPa)	Average Flexural Strength (MPa)
CFRP	S1	361.21	499
	S2	585.68	
	S3	506.38	
	S4	496.27	
	S5	544.79	
CFRP/0.1 rGO	S1	461.81	520
	S2	568.25	
	S3	426.82	

	S4	537.93	
	S5	608.12	
CFRP/0.2 rGO	S1	498.86	530
	S2	510.05	
	S3	487.65	
	S4	585.82	
	S5	568.86	
CFRP/0.3 rGO	S1	563.05	562
	S2	528.92	
	S3	538.84	
	S4	619.15	
	S5	563.6	

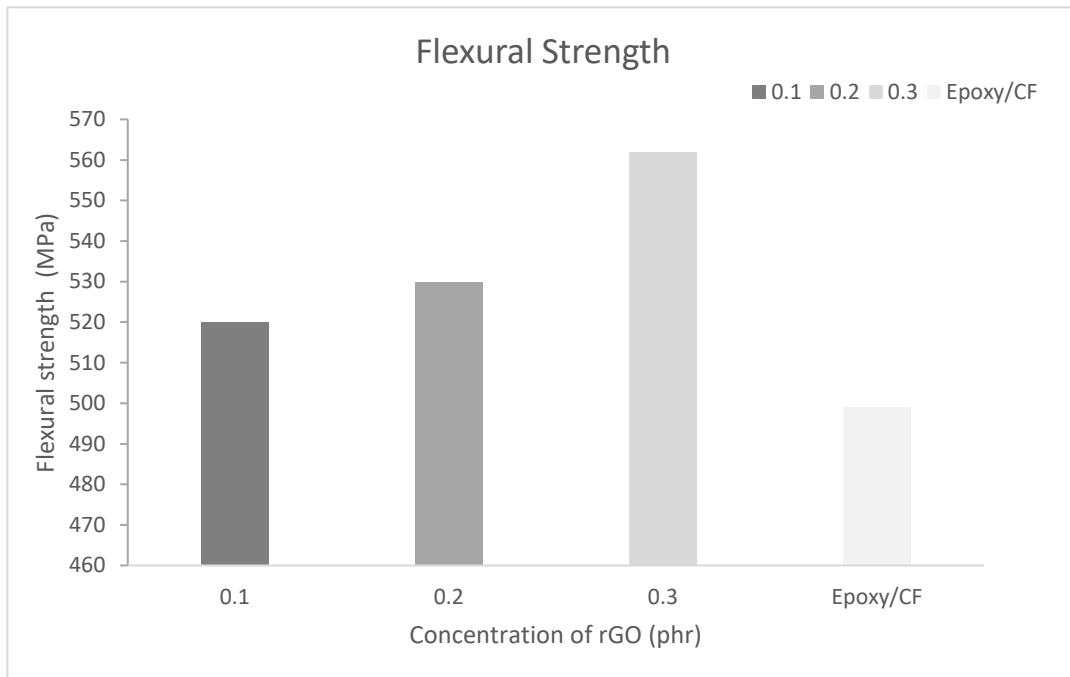


Figure 4.16: Flexural strength results of rGO reinforced carbon fiber composites

From the above results, we observed that we have the maximum tensile strength with GO concentration 0.3 phr i.e., 562 MPa whereas with concentration 0.1 phr and 0.2 phr and epoxy/CF have tensile strength as 520 MPa, 530 MPa and 499 MPa respectively. We observed an increase of 12% when the GO at concentration at 0.3 phr was included in the matrix as compared to the epoxy/CF matrix.

IMPACT STRENGTH

For tensile testing, various specimens were prepared with different concentrations of rGO, and five specimens were taken from each sample.

Table 4.13. Variation in Impact strength with different concentration of GO in CFRP laminates

	Samples	Impact Strength (kJ/m ²)	Impact Flexural Strength (kJ/m ²)
CFRP	S1	84.93	87
	S2	104.79	
	S3	95.99	
	S4	66.49	
	S5	78.31	
CFRP/0.1 GO	S1	77.34	68
	S2	66.08	
	S3	62.92	
	S4	73.12	
	S5	61.98	
CFRP/0.2 GO	S1	82.08	75
	S2	62.38	
	S3	77.69	
	S4	75.15	
	S5	78.28	
CFRP/0.3 GO	S1	92.84	97
	S2	100.83	
	S3	103.95	
	S4	85.73	
	S5	99.41	

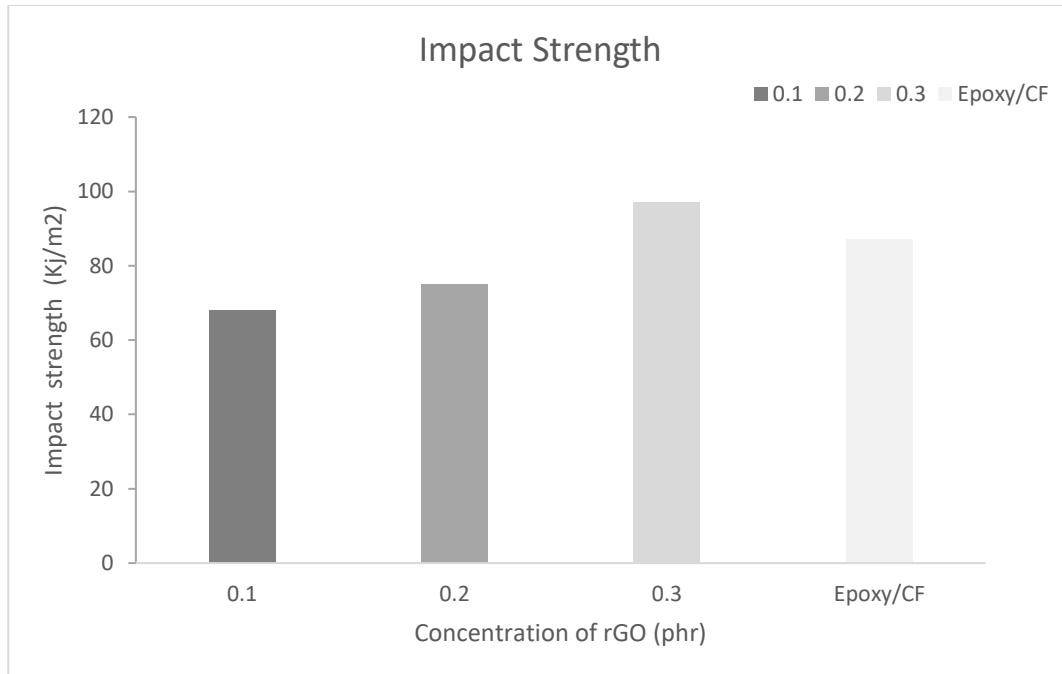


Figure 4.17: Impact strength results of GO reinforced carbon fiber composites

From the above results, we observed that we have the maximum impact strength with rGO concentration 0.3 phr i.e., 97 kJ/m² whereas with concentration 0.1phr and 0.2 phr and epoxy/CF have impact strength as 68 kJ/m², 75 kJ/m² and 87 kJ/m² respectively. We observed that 0.3 phr has an increase of 12% as compared to epoxy/CF matrix.

4.5 CLOSING REMARKS

The chapter discusses the results attained from the modified composites having different concentrations of GO and rGO. The results have shown that GO was more effective in increasing the flexural and impact strength of GFRP/CFRP composites. Pathak et al. (2019) also discussed in their study that GO has more functionalities and OH groups in their structure which enhances the bonding among constituents thereby improving the mechanical strength of the composites. GO bonds with the epoxy resin via H-bonding. The epoxy group of GO is converted to ether bond by CF hydroxyl group via substitution reaction. When we increase the weight of GO in GFRPs/CFRPs, agglomeration of GO weakens the bonding between the GF/CF and GO and due to the steric hindrance, causes nano defects in the composite which is accountable for deterioration of properties. The chemical bonding of GF and GO is important for the interfacial bonding.

The results also proved that GO is behaving as the interface modifying agent which helps in

enhancing the properties of GFRP/CFRP composites. For GFRPs the ideal composition of GO and rGO is 0.1 phr for accomplishing the maximum mechanical properties of composites. The inclusion of GO into composites increases the stability which is because of the layered structure of GO that builds a path, behaving as a barrier to prevent any permeation of oxygen. The increase in weight of GO can cause restacking of GO which will decrease the interfacial properties of composites.

5

CONCLUSION AND FINDINGS

5.1 GENERAL

Graphene derivatives have very high potential for implementation in construction industry. Use of these materials extends the life of structures since it provides the structure more strength and repair of the structures. In the present study, GFRP/CFRP laminates or sheets were modified with graphene derivatives to enhance their mechanical behavior. The mechanical properties tested include the tensile strength, flexural strength, and impact strength.

5.2 GFRP NANO-MODIFIED

5.2.1 With graphene oxide (GO)

- There has been a significant improvement in the mechanical properties of GFRP laminates with the inclusion of GO in the matrix.
- The inclusion of GO in the matrix increases the tensile strength with 12% as compared to the epoxy/GFRP laminates without any nano-reinforcement.
- The maximum flexural strength increased by 33% with reference to the epoxy/GFRP laminates.
- For impact strength, the incorporation of GO increases the strength by 17% as compared to epoxy/GFRP laminates.
- All the prepared GFRP laminates modified with GO showed enhancement at 0.1 phr concentration of GO.

5.2.2 With reduced graphene oxide (rGO)

- The inclusion of rGO in the matrix increases the tensile strength with 18% as compared to the epoxy/GFRP laminates without any nano reinforcement.

- The maximum flexural strength increased by 21% with reference to the epoxy/GFRP laminates.
- For impact strength, the incorporation of rGO increases the strength by 6% as compared to epoxy/GFRP laminates.
- All the prepared GFRP laminates modified with rGO, showed enhancement at 0.1 phr concentration of rGO.

5.3 CFRP NANOMODIFIED

5.3.1 With graphene oxide (GO)

- There has been a significant improvement in the mechanical properties of CFRP laminates with the incorporation of GO in the matrix.
- The inclusion of GO in the matrix increases the tensile strength with 5% as compared to the epoxy/CFRP laminates.
- The maximum flexural strength increased by 20% with reference to the epoxy/CFRP laminates.
- For impact strength, the incorporation of GO increases the strength by 65% as compared to epoxy/CFRP laminates.
- All the prepared CFRP laminates modified with GO, showed enhancement at 0.2 phr concentration of GO.

5.3.2 With reduced graphene oxide (rGO)

- The inclusion of rGO in the matrix increases the tensile strength with 12% as compared to the epoxy/CFRP laminates.
- The maximum flexural strength increased by 12% with reference to the epoxy/CFRP laminates.
- For impact strength, the incorporation of rGO increases the strength by 14% as compared to epoxy/CFRP laminates.
- All the prepared CFRP laminates modified with rGO, showed enhancement at 0.3 phr concentration of rGO.

Based on the concentration of graphene derivatives, it improves the mechanical strength of composites. Hence the results proved that GO acts as better reinforcing material as it possesses high functional groups in its structure (carboxyl, hydroxyl, and epoxy moieties) which can easily bond with the epoxy molecules of polymer matrix, thereby increasing the properties of composites. The more addition of graphene-x acts as steric hinderance in the whole system creating an interference for bonding of constituents, hence decreasing the strength of composites.

5.4 RECOMMENDATIONS FOR FUTURE WORK

- In proposed fibers, the hybrid model can be considered where both glass and carbon fibers are layered up together which can find potential applications in ballistics.
- Carbon fibers have high cost as compared to glass fiber polymer so any other alternative which is cheaper than these materials can be studied.
- Graphene derivatives can also be combined with other nano-fillers such as carbon nanotubes (CNTs), clay to further enhance the mechanical properties.

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