Surface modification of Graphene Oxide nanosheets with Magnesium Oxide Nanoparticles as Photocatalyst

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(2017)

Nanotechnology.

Nanomaterials.

Graphene Oxide

X-Ray diffraction.

بسم الله الرحمن الرحيم

إِنَّ مَعَ العُسْرِ يُسْرًا

DEDICATED TO

MY PARENTS AND MY LOVING HUSBAND

INTERNATIONAL ISLAMIC UNIVERSITY, ISLAMABAD

FACULTY OF BASIC AND APPLIED SCIENCES DEPARTMENT OF PHYSICS

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(Registration No. 338-FBAS/MSPHY/S15)

A thesis submitted to

Department of Physics for
the award of the degree of
MS Physics

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FINAL APPROVAL

It is certified that the work presented in this thesis entitle“**Surface modification of the Graphene Oxide nanosheets with Magnesium Oxide nanoparticles as Photocatalyst**”by **Mehreen Iftikhar**bearing **Registration No. 338-FBAS/MSPHY/S15** in partial fulfilment for the award of degree of MS Physics from International Islamic University, Islamabad has been approved.

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ABSTRACT

Co-precipitation and modified hummer's method were utilized for the synthesis for magnesium oxide and graphene oxide respectively. Surface of graphene oxide is then modified with the magnesium oxide nanoparticles through dispersion method for the application of the photo-catalysis. Photocatalytic activity of synthesized samples was contemplated by watching their behavior in degrading dye like methylene blue. To investigate the prepared samples, XRD, SEM and FTIR techniques were utilized. SEM characterization revealed that the average 62.18 nm sized MgO nanoparticles were dispersed uniformly and separately on the GO nanosheets. Few layers of these thin graphene oxide nanosheets were transparent, which affirmed the existence of 2-D nanosheets. XRD technique confirmed the formation of MgO nanoparticles and GO nanosheets. The oxidation process during the synthesis of GO nanosheets and Mg-O bonding in MgO nanoparticles were confirmed through the FTIR examination. No clear degradation happens when GO utilized as photocatalyst showing no photocatalytic activity. Whereas dispersion of MgO on GO displays upgraded photocatalytic execution contrasted with that of GO.

Chapter 1

1 Introduction

1.1 Nanoscience:

Nanoscience is the examination of structures and materials on the extent of nanometers. To give you a considered to what degree a nanometer is, this printed page is around 75,000 nanometers wide. Right when structures are made pretty smaller particularly in the nanometer range, they can go up against entrancing and significant properties. Nanoscience has authoritatively influenced our lives with progressions, for instance, recolor safe surfaces moved by nanoscale highlights found on lotus plants and PC hard drives, which store information on appealing strips that are just 20 nanometers thick. Scientists and experts from a couple orders including material science, science uses nanoscience norms for forefront applications in essentialness, solution, information stockpiling, enrolling and elsewhere.

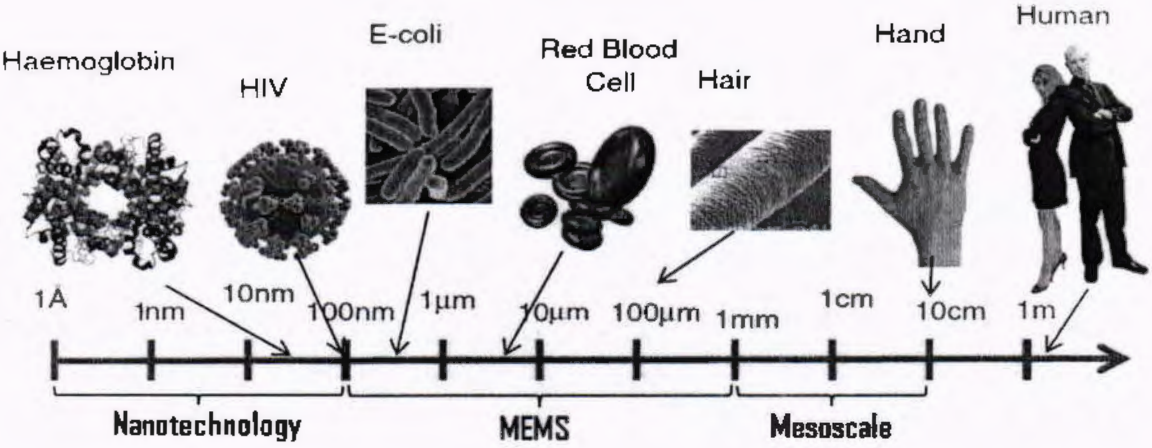

Figure 1: Concept of nanoscience

1.2 Nanotechnology:

The blueprint, depiction, era, and utilization of structures, contraptions, and systems by controlled control of size and shape at the nanometer scale (atomic, sub-nuclear, and macromolecular scale) that produces structures, devices, and systems with no short of what one novel/common trademark or property.

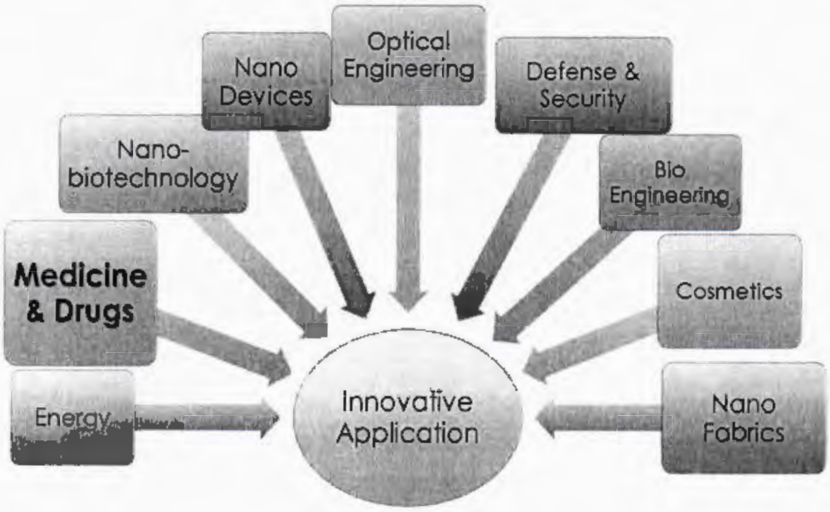

Figure 2: Applications of nanotechnology

1.3 Applications of nanotechnology:

In spite of the fact that the advancement and use of nanotechnology is essentially still in the examination stage, some designed nanomaterials are created and utilized as a part of business applications today. Cases of items that are created right now utilizing nanotechnologies include:

- **Medicines:**

  Investigators are making nanoparticles in which the degree of molecules changed that can pass on quiets particularly to sickly cells in your body. Right when it's culminated, this system should fundamentally lessen the damage treatment, for instance, chemotherapy does to a patient's sound cells.

- **Electronics**:

  Nanotechnology holds a few responses for how we may build the abilities of electronics while we diminish their weight and power utilization.

- **Food:**

  Nanotechnology is influencing a couple parts of sustenance science, from how food is produced to how it is packaged. Associations are making nanomaterials that will have any sort of impact in the substance of support, and additionally in sustenance prosperity, and the therapeutic points of interest that food passes on.

- **Solar cells:**

  Associations have made nanotech sun fueled cells that can be delivered at basically cut down cost than conventional sun arranged cells.

- **Batteries:**

  Associations are at present making batteries using nanomaterials. One such battery will be a better than average as new in the wake of sitting on the rack for a significant long time. Another battery can be invigorated essentially speedier than customary batteries.

- **Fuel cells:**

  Nanotechnology is being utilized to diminish the cost of impetuses utilized as a part of energy units to create hydrogen particles from fuel, for example, methanol and to enhance the proficiency of layers utilized as a part of power modules to separate hydrogen particles from different gasses, for example, oxygen.

- **Space:**

  Nanotechnology may hold the way to making space-flight more viable. Progressions in nanomaterials make lightweight rocket and a link for the space lift conceivable. By altogether diminishing the measure of rocket fuel required, these advances could bring down the cost of achieving circle and going in space.

- **Fabric:**

  Making composite texture with nano-sized particles or strands permits change of texture properties without a huge increment in weight, thickness, or firmness as might have been the situation with already utilized procedures.

- **Sporting Goods:**

  In case you're a tennis or golf fan, you'll be cheerful to hear that despite wearing items has wandered into the nano area. Current nanotechnology applications in the games field incorporate expanding the quality of tennis racquets, filling any flaws in club shaft materials and diminishing the rate at which air spills from tennis balls.

1.4 Introduction to Nanomaterials:

The material world can be divided in to five categories like ceramic, polymer, metal, semiconductor and composites. Among them Nanomaterial are one of the category which is the study of significant relationships between physical properties and dimensions of the materials on the nanometer scale. Nanomaterial are considered to those materials that exhibits in the order of a billionth of a meter on the nanometer scale or in other words, we can say that nanomaterials referred as those materials whose size ranges from 1-100nm[1].Nanomaterials are considered to be the cornerstones of the nanoscience and nanotechnology. Same composition of a particular nanomaterial as known material in bulk form may have different physical as well as chemical properties[2] than the same material in the bulk form and when they entered the body their behavior is quite different. By various groups[3-12] have been reported that in the past few decades different nanostructures such as Nanorods, Nanotubes Nanoribbons, Nanoflakes, Nanoflowes, Nanocubes, Nanobelts, Nanoclusters, Nanofibers and Nanoporous materials.

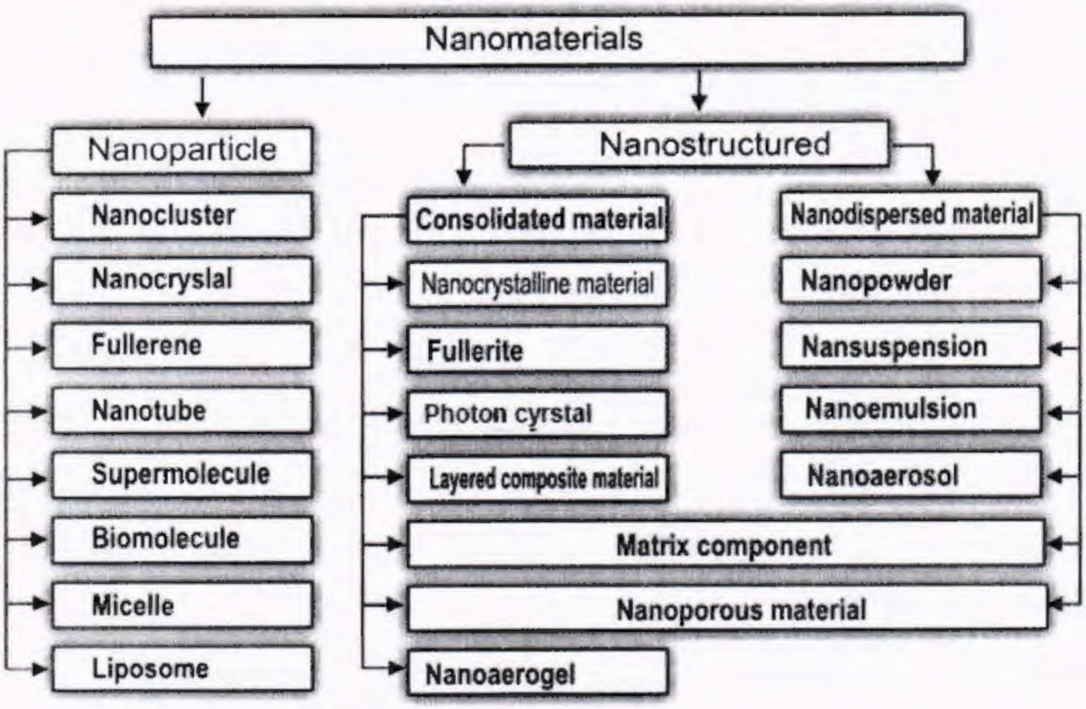

Figure 3: Classification of nanomaterials

1.4.1 Method of preparation of nanomaterials:

1.4.1.1 Physical methods:

Following physical methods are used for the preparation of the nanomaterials.

- ➢ High energy ball milling
- ➢ Flame spray pyrolysis
- ➢ Laser ablation

1.4.1.2 Chemical methods:

Following chemical methods are used for the preparation of the nanomaterials.

- ➢ Co-precipitation method
- ➢ Sol–gel method
- ➢ Hydrothermal method

1.4.2 Importance of Nanomaterials:

As compared to the bulk substances nanomaterials exhibits unique properties such as

- Mechanical strength
- Electrical conductivity
- Thermal stability
- Magnetic properties
- Catalytic activity
- Optical properties

As Nanomaterials exhibits unique properties so they can be utilized in various applications.

1.4.3 Classification of the Nanomaterials:

There are different methods by which we categorized the nanomaterials including origin, dimensions and their structural configuration[13].

I. Classification based on origin:

On the basis upon origin, nanomaterials can be classified in to two main categories such as:

1-Natural nanomaterials

2-Artificial Nanomaterials

II. **Classification based on dimensions:**

On the basis upon dimensions, nanomaterials can be classified in to four main categories such as:

1-Zero dimensional (0-D) Nanomaterials

2-One dimensional (1-D) Nanomaterials

3-Two dimensional (2-D) Nanomaterials

4-Four dimensional (3-D) Nanomaterials

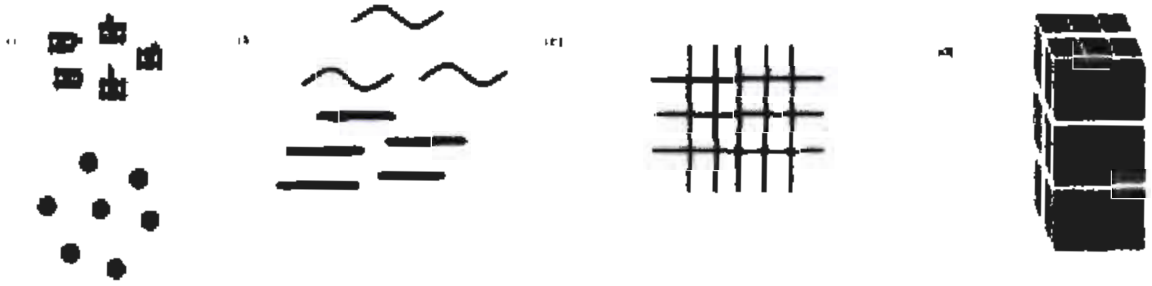

Figure 4: Classification based on dimensions

III. **Classification based on structural configuration:**

On the basis upon structural configuration, nanomaterials can be classified in to four main categories such as:

1-Carbon based Nano materials

2-Metal based Materials

3-Dendrimers

4-Composites

1.5 Graphene Oxide:

Graphene oxide is essentially a two-dimensional wrinkly carbon sheets with distinct oxygenated functional groups on its base plane and boundaries having thickness of 1nm that varying between a few nanometers and quite few microns on the sides. In 1859, British chemist B. C. Brodie was the first man who prepare Graphene oxide, and during the last half decade it spread widely in the science communal, just because of the reason that it was supposed to be an prodigious precursors to (a single atomic layer of graphite) graphene. The strong oxidation/embolism of graphite is very significant to prepare Graphene oxide(GO).During the oxidation process, Many oxide-containing groups such as: epoxy, carboxyl, hydroxyl and carbonyl groups on the carbon basal plane are formed [14].

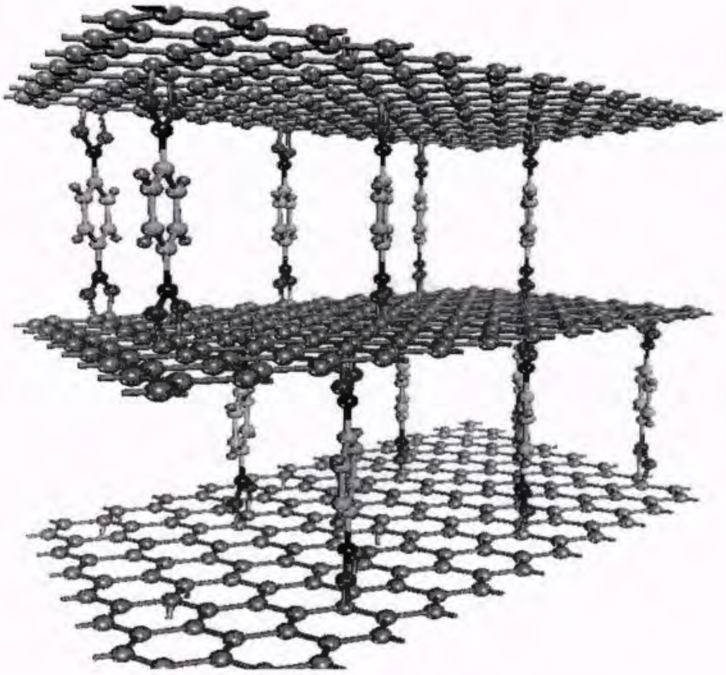

Figure 5: Graphene Oxide

1.5.1 Sp^3 Bonding and Chemical Structure Of GO:

The carbon arrangement of the graphene oxide is disarranged by a variety of oxygen functional groups (transferring an appreciable degree of sp^3 hybridization), who's structure will be explained more incisively below. Such type of the functionalization has a very deep impression on the properties of the material, mentioned above all when compared to its parent graphene. Various structural models are used to explain graphene oxide by many scientists. But accurate structure is still under discussion. The main reason behind this is due to the modification in the fabrication processes, use of different (light and strong) oxidizing agents, fluctuation in the precursor that utilizes in the synthesis of Graphene Oxide etc.

In 1939, Hofmann and Holst for the very first time gave his first structural model of the graphene oxide. In this structure the base plane of GO sheet have equal ratio of carbon and oxygen and they also consist of epoxy groups spreading from the corner to corner[15].

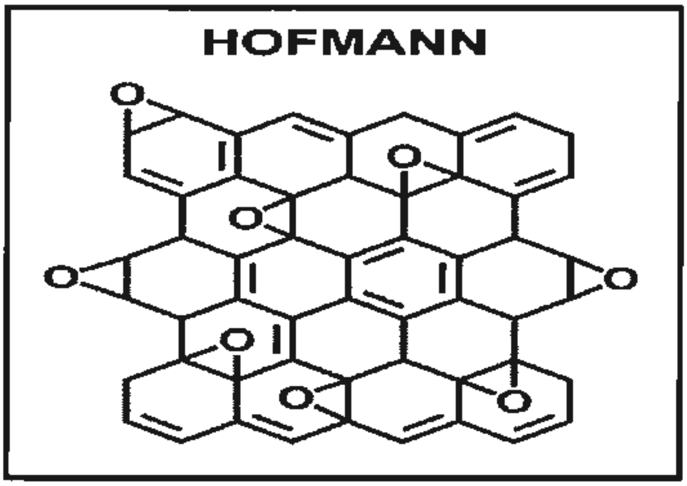

Figure 6: Structure proposed by Hofmann

After the Hofmann and Holst, the modification of lattice structure is done by introduction of the hydroxyl groups on the basal plane, the idea given by Ruess. For the first time this introduction of hydroxyl groups in the structure was an indication of the existence of the hydrogen content in the GO.

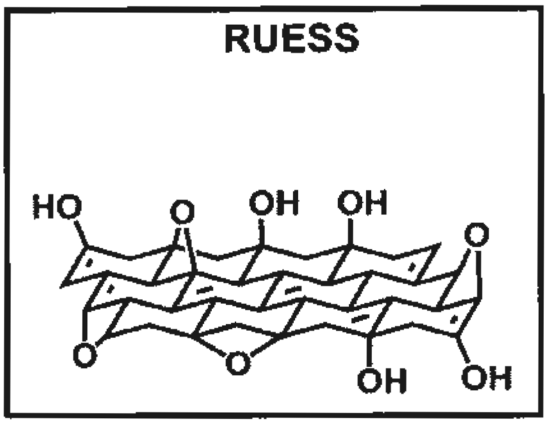

Figure 7: Structure proposed by Ruess

After these proposed structures, Clauss and Boehm give his enhancing contribution by exhibiting C=C bonds (Ketone and enolic groups) with carboxylic groups on the boundaries. After some time Schloz and Boehm taking the review of the chemistry of the structural model and developed it into uneven carbon layers that consists of interchangeably linked ribbons of quinoidal structure and rings of cyclohexane are opened in the chair formation[16].

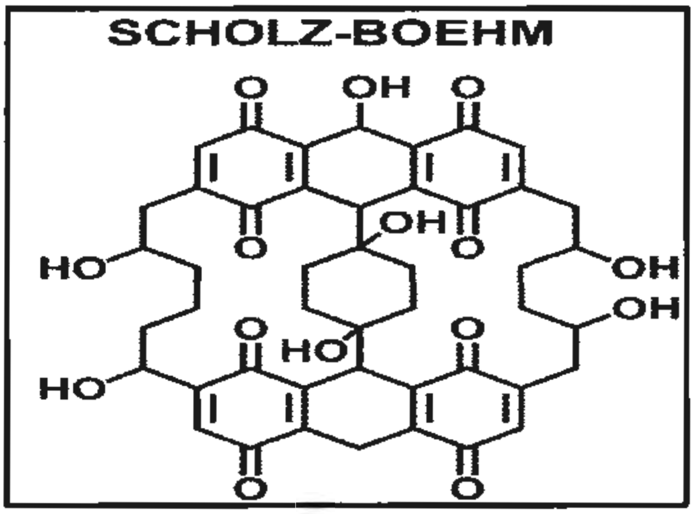

Figure 8: Structure proposed by Scholz and Boehm

Up till now the most accepted model for the structure of Graphene oxide is Lerf-Klinowski model.

1.5.2 Lerf-Klinowski model:

The most extensively accepted structural model for the graphene oxide is Lerf-Klinowski model. In this model, the cyclicity that occur in the structure was completely eliminated as they were replacing by a non-stochiometric amorphous alternative. This structure can be defined in two different ways. First one is the (graphene like) that is bit functionalized, mostly with the sp^2-hybridized carbon atoms and the second one is of majorly oxygenated mostly sp^3-hybridized carbon atoms. The basal plane in this model is proposed to be adorned by the epoxide and hydroxyl functional groups which are considered to be separated into islands that are surrounded by graphene-like sections that are highly oxidized, while on the boundaries of the graphene sheets carboxylic acids or carboxylates are present that mainly depend on the pH of the solution. Several papers were published on the hydration and structure by the Anton Lerf and Jacek Klinowski. Lerf and his coworkers have demonstrated the detailed analysis of the graphene oxide structure which based on not only SSNMR analysis, but also on the GO reactivity with a variety of compounds and infrared spectroscopic data. They observed that the double bonds were either conjugated or aromatic as that the separated double bonds would be unlikely to endure in the strong oxidizing conditions used (modified Hummers method). Infrared spectroscopic data indicating that the carboxylic acid groups were present in low quantities at the outer boundry of the graphitic platelets, in addition to the other keto groups.

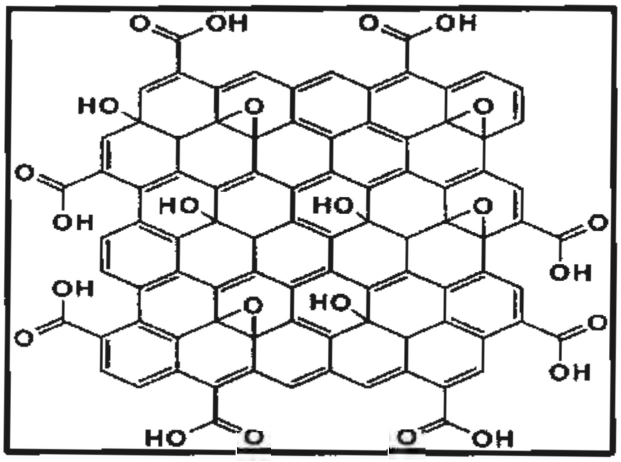

Figure 9: Structure proposed by Lerf-Klinowski

1.5.3 Properties of the Graphene Oxide:

- **Dispersibility:**

  Graphene oxide show easy dispersion in water, other organic solvents like (THF,NMP,DMF) as well as in several matrixes. But the most common reaction media for the GO is the water. The ways by which they get dispersed in water is through sonication and mechanical stirring. In most of the cases, machinal stirring is considered to be far better than the sonication. The reason behind it is that the sonication causes decrease in size(from few microns to several hundred nanometers) as well as creates a lot of defects[17-19]. It is observed that the measured dispersibility of the graphene oxide in water is round about 1-4mg/ml[20].It is supposed that the graphene oxide is considered to be more amphipathic just because of having more hydrophobic at its center and more hydrophilic at its edges considering as a surface-active agent[21, 22].

- **Interaction with water:**

  Graphene oxide having hydroxyl, epoxy and carboxyl gatherings make it hydrophilic and in entomb layer spaces adsorbed water particles are available even after industrious drying. Graphene oxide is solid hygroscopic and this property relies on upon ecological moistness level[23]. The mechanical, electronic and basic properties of graphene oxide can be affected when the stacked hydrogen bonds in water atoms introduce between its layers make bonds with the oxygen bunches display at first glance[24].Because of the lower dissolvability, bring down reactivity and more grounded holding of D_2O contrasted and H_2O diminish out there between the layers was watched. Carbonyl and opening arrangement amid graphene oxide diminishment process is because of the presence of water[23].

- **Thermodynamic stability of GO:**

  Xue Jiang and his co-workers reported that the graphene oxide systems exhibits more stable thermodynamically as the scope of hydroxyl and epoxy bunches builds, the development of vitality of graphene oxide turns out to be more negative, recommending that persistent functionalization of epoxy and hydroxyl gatherings are thermodynamically ideal[25].

- **Reactivity:**

  Graphene Oxide's colloidal scattering responds promptly with assortment of chemicals paying little heed to response media the vast majority of which have been utilized as reducing agents. Graphene oxide is thought to be a forerunner of Graphene[26].

- **Toxicity:**

  Akhvan and coworkers have been reported that the Graphene Oxide having sharp edges pulverized the cell layer of microbes characterizing the danger of graphene oxide to bacteria[27].Blood similarity and toxicity of Graphene Oxide was reliant on dosage, measure of shedding and size of sheet was additionally reported[28].

1.5.4 General Properties of Graphene Oxide:

Graphene Oxide claims tunable electronic properties. Because of the expansive segment of sp^3 hybridized carbon molecules combined with the oxygen-containing bunches graphene oxide carry on as insulator, bringing about a sheet resistance of $10^{12}\Omega^{-1}$ or might be higher. By controlling the relative proportion of the epoxy and hydroxyl bunches, coverage and arrangement, band gap of graphene oxide can be custom-made. However, GO additionally shows huge optical and mechanical properties for an extensive variety of uses. The optical retention of GO is managed by the π-π^*, that is reason for an absorption peak somewhere around 225 and 275 nm (4.5-5.5eV). It has been reported that the Young's modulus of graphene oxide is 6-42GPa and its intrinsic strength of graphene oxide nanosheets is found to be 76-293MPa. Structural/Chemical assortment of GO can be extended everywhere scale by the oxygenated assembles and further chemical modification or functionalization, which offer a real approach to adjust the physical and compound properties of GO to expected extents. Graphene oxide has a limited electronic bandgap created by the interruption of pi systems because of the development of oxygen containing bunches[29].

1.5.5 Applications of Graphene Oxide:

There have been enormous endeavors in creating GO-based materials for different sorts of Li batteries and supercapacitors, though there are likewise sure exercises on hydrogen era/stockpiling and also purging of water and air by utilizing GO-based materials. Graphene oxide is a promising material for vitality stockpiling and natural applications.

- **Cleansing of water:**

  Water contamination is an overall natural nervousness so to refine water, there are two primary steps:

  The first is adsorption of contaminations and second is to change over the toxins into helpful energies. For this reason, GO and GO based composites show solid activities. GO and GO based composites can without much of a stretch expel distinctive natural contaminants from water[30].

- **Energy storage devices:**

  Lithium particle rechargeable batteries experience the ill effects of its low theocratical limit restricts so in such manner graphene oxide and graphene oxide based segments demonstrate superb electrochemical exhibitions[30].

- **Immurement of harmful gases:**

  Graphene oxide sheets especially within the sight of water atoms sheets display astounding adsorption conduct so it can be utilized to catch the carbon dioxide in the nursery[30].

- **Transmutation of harmful gases:**

  Graphene oxide and its composites are presently a day generally to change the ruinous poisons into some valuable energies[30].

- **Photocatalytic water splitting:**

  Scheming a novel impetus that is equipped for satisfying all the modern prerequisites is a still huge challenge. GO and GO-based materials have been set up as exceptional impetuses for hydrogen era from water part determined by light. Considering the high surface range, higher electron portability, possible get together and tunable band gap[26].

1.6 Metal Oxides:

Metal Oxides are mixes framed by a metal and oxygen, in which the oxygen has an oxidation number –2. To catalyze the radical chain response in photocatalytic oxidation response semiconductors, for example, metal oxides can be utilized as activators. The reasons that semiconductors photocatalyst are supported in photocatalytic debasement of colors in waste water are such as:

They are less expensive.

They are for the most part nontoxic.

Their properties can be changed by doping, sensitizer or by diminishing size.

They can help in encouraging the multi electron exchange process.

Without a significant misfortune in photodegradation they are equipped for remarkable their utilization.

Reported a few metal oxides that are great photocatalyst are magnesium oxide, zinc oxide, tungstate, titanium oxide and numerous other a few metal oxides have been accounted for to be great photograph impetuses, they are turned out to be to be a dynamic photograph impetus for corruption of colors, being contemplated widely[31].

1.7 Magnesium Oxide:

Metal Oxide nanomaterials are essential and amazing materials, in light of its uncommon properties like chemical stability, high photocatalytic action, high electrical permittivity, non-dangerous nature. So, it is utilized as a part of different applications like optical, electrical, electronic, disinfectant, antibacterial, ecological, semiconductors and reactant gadgets. The majority of the scientists are working with metal oxides nanoparticles because of their interesting properties, for example, hydrophobic, photocatalytic, strength etc. Hence, they are utilized as a part of numerous applications named as coatings, impetuses, antibacterial, therapeutic sciences, sensors, semiconductors, capacitors and batteries.

Magnesium oxide is second group component with nuclear no 12 and oxygen is 6th group component with nuclear no 8. Magnesium oxide (MgO), or magnesia, is a white hygroscopic strong mineral that happens actually as periclase. Magnesium Oxide is a vital material, which is utilized as a part of numerous applications like catalysis, harmful waste remediation, paint, superconducting items, antibacterial exercises against nourishment borne pathogens.

1.7.1 Structure of Magnesium Oxide:

Magnesium Oxide has an experimental recipe of MgO and comprises of a cross section of Mg^{+2} particles and O^{-2} particles that are held together by ionic bonding. The material shown in fig is magnesia(MgO) and the structure itself is alluded to as shake salt since basic table salt (sodium chloride, NaCl) has a similar structure. In the rock salt structure, every particle is encompassed by six quick neighbors of the inverse charge (e.g., the focal Mg^{2+} cation, which is encompassed by O^{2-} anions). This to a great degree proficient pressing takes into consideration neighborhood balance of charge and makes for stable holding. Oxides that take shape in this structure have a tendency to have moderately high liquefying focuses. MgO is an exceptionally important wide band gap insulator. Further it has homomorphous compound with rock salt structure(FCC), the magnesium ions occupying octahedral sites within the anion closed packed structure and its ionic constituents comprises a relatively small no of electrons.

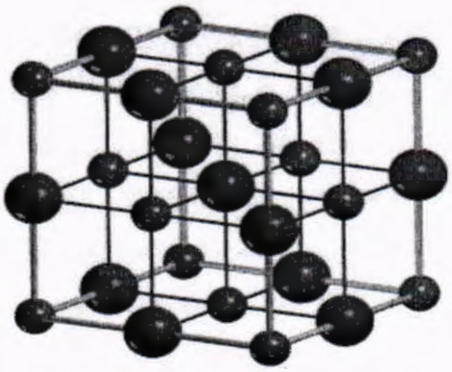

Figure 10: Crystal structure of magnesium oxide

1.7.2 Properties of Magnesium Oxide:

There are numerous properties of magnesium oxide such as optical, electrical, mechanical and chemical.

Table 1: Properties of magnesium Oxide

Properties	**Ranges**
Chemical Formula	MgO
Odor	Odorless
Appearance	White Powder
Melting Point	2852^0C (5166^0F, 3125^0K)
Boiling Point	3600^0C (6150^0F, 3870^0K)
Solubility in water	0.00062g/100mL at (0^0C)
Density (g/cm^3)	3.58g/cm^3
Resistivity	10-^8ohm.m
Dielectric constant	9.6 MHz
Thermal Expansion	10-^6/K
Thermal Conductivity	60 W/m. K
Specific Heat	1030 J/kg. K
Latent heat of Fusion	1880 kJ/kg
Band gap	7.8eV
Young's Modulus (GPa)	330 GPa
Shear Modulus (GPa)	122 GPa
Bulk Modulus (GPa)	277 GPa

1.7.3 Applications of Magnesium Oxide:

There are numerous applications of Magnesium oxide utilized for different purposes such as in refractories, cements, thermocouple tubes, plasma display screens etc.

- **Refractories:**

  Magnesia is generally utilized as a part of the steel business as a refractory block frequently impregnated with carbon (tar, pitch, graphite) to give ideal properties for erosion resistance in situations of fundamental slags, especially in BOF heaters or slag lines of treatment scoops. Magnesia blocks frequently in blend with spinel or chrome are likewise utilized as a part of ferroalloy, non-ferrous, glass and cement industries.

- **Cements:**

  Magnesia concrete is a refractory binder in view of a magnesium oxychloride detailing. It is quick solidifying and has various recalcitrant and general repair applications. Magnesia is likewise utilized as a room temperature curing operator for phosphate concretes.

- **Thermocouple Tubes:**

  Expelled magnesia defensive sheaths have been utilized to house thermocouples in forceful situations.

- **Plasma Display Screens:**

  Magnesia has been utilized as a defensive film for plasma show screens where its electro-optical properties are utilized to advantage.

- **Substrate for thin film formation:**

  High immaculateness evaluations of magnesia have been utilized to develop thin film semiconductors. Other comparable electronic applications have been explored.

- **Brake Linings:**

  Magnesia has been incorporated into brake linings because of its thermomechanical properties. Its middle of the road hardness gives adequately low wear on metal while directing warmth from the grating contact surfaces.

- **Heating Elements:**

  Magnesia powder is broadly utilized as a filling for electrical warming components for applications in contact with air or fluids, for example, electric cooker rings, stockpiling radiators, clothes washers, and diesel motor gleam plugs. Melded magnesia has the perfect mix of electrical resistance and warm conductivity.

1.8 Literature Review:

- Y. Mansowpanah et al[32] modify the GO Nano sheets with the polyethylene glycol (PEG/GO) in order to attain the better characteristics of polyamide (PA) thin layer membranes. UV irradiation was used to investigate the effects of irradiation on the membrane properties. In aqueous phase, different concentrations of PEG/GO (0.5,1.0 and 1.5wt%) were fabricated on these modified thin films. SEM and AFM characterization confirm the establishment of compressed and dense thin films. To compare the characteristics of the prepared thin layers, commercial membrane (NF2, Sepro) was used.
- Ju Ran Lee et al[33] synthesized the MgO/GO nanocomposites by the sol-gel process. SEM characterization revealed that the MgO nanoparticles were uniformly decorated on the GO nanosheets with an average diameter of 70nm.Through the reaction cycles and different concentrations of the MgO precursors, size of the resulting MgO particles and the loading density would be controllable. They concluded that the MgO/GO nanocomposites finds its future application in the field of chemical sensing and adsorption.
- Murugan Veerapandian et al[34] investigated the anti-bacterial properties of GO by irradiating its surface with ultraviolet(UV).Its Microscopic characterization confirm the formation of wrinkles, wavy features or thin grooves like structure. GO nanosheets irradiated by the UV have more noticeable /marked antibacterial behavior than GO nanosheets and standard antibiotic (kanamycin), through the results confirmed by minimum inhibitory concentration (MIC). The small antiseptic concentration of normal GO nanosheets was investigated to be twofold higher than its corresponding MIC value, suggesting its promising bactericidal activity.
- K. Ganapathi Rao et al[35] synthesized the MgO nanoparticles by chemical co-precipitation technique by using Magnesium Nitrate as core precursors. In this work, they emphasize on to the synthesis of magnesium oxide nanoparticles and its application in the field of environment. They also determine its structural properties through various characterization techniques.
- Zainab Raheem et al[36] at studied the photocatalytic activity of nanoparticles and microparticles of magnesium oxide (MgO).In this research, they used methylene blue dye of concentration 5ppm as simulation model. The nanoparticles and microparticles of MgO was exposed to UV radiation. Through the photodegradation of MB dye, they evaluated

the photocatalytic activity of MgO Powder. In degradation, MgO nanoparticle gives better results than MgO microparticles. They concluded that when exposed to UV light for 4hrs, the MB degradation was 94.8% in the presence of MgO nanoparticle while when exposed to the UV for 7hrs the MB degradation was 85.57% in the presence of MgO microparticle.

- S.Demirci et al[37] reported the synthesis of magnesium oxide nanoparticles by spray pyrolysis and sol-gel technique. By utilizing an aqueous methylene blue(MB) solutions they determined the photocatalytic activity of nano-scale particles. Through the absorbance measurements performed via UV spectrophotometer, they determined the photocatalytical degradation rates of MB solutions. They concluded that the MgO nanoparticles synthesized by both sol-gel and spray pyrolysis exhibited appreciable photocatalytic activity for the degradation of MB dye when exposed UV light irradiation with small differences. In this research, they also investigated the effects of particle size and surface area on the photocatalytic properties.
- M. Kandiban et al(2015)[38] synthesized MgO nanoparticles by wet chemical method via co-precipitation and hydrothermal method. XRD results predict that the prepared MgO nanoparticles exhibits crystalline form with cubic phase. UV results reveals that MgO nanoparticles prepared by co-precipitation method having absorption peak at wavelength of 295nm and MgO nanoparticles prepared by the hydrothermal method having absorption peak at the wavelength of 297nm.Their bandgap value were also determined and reported. Through the FTIR characterization, the vibrational frequency of Mg=O were observed at 548cm-1. The analysis of the photocatalytic property of the developed nanoparticles were done by the absorption of methylene blue dye. That confirms the degradation property of the MgO nanoparticles.
- N.K.Nga et al[39] have reported the synthesis of the MgO nanoparticles by using $MgCl_2$ $.H_2O$ and NaOH as starting materials. The formation of MgO is done by using hydrothermal process while using CTAB (cetyltrimethyl ammonium bromide) that act as a morphology controller in order to obtain a high surface area. The purpose of CTAB is that they increase the adsorptive performance of MgO nanoparticles.

- F.E Tanta way et al[40] have shown the formation of 1D nanowires of MgO by hydrothermal process having diameter of 6nm and length about 10μm at very low temperature.
- X.Qiao et al[41] synthesized MgO nanoparticles by co-precipitation method by using $MgCl_2 .6H_2O$ as salt precursors, anonionic surfactant such as polyethylene glycol(PEG 400) and ammonium hydroxide as precipitator.
- M Rezaei et al[42] prepare the nanocrystalline magnesium oxide having high surface area by reflux method. In this synthesis, Plutonic P123 block copolymer surfactant, ammonium solution and $Mg(NO_3)_2 .6H_2O$ as starting materials.
- Satish et al,(2013)[43] synthesized GO nanosheets by using modified Hummer's method. They also test its antibacterial activity using Klebseilla bacterial species. They found that GO exhibited stronger antibacterial activity against bacterial species.
- Bongiwe Silwana et al (2015)[44] demonstrated the synthesis and characterization of the reduced GO modified antimony nanoparticle composite, which are potentially useful in sensors and related fields.
- K Mageshwari et al,(2011)[45] used a cost effective route to prepare magnesium oxide(MgO) nanoparticles by wet chemical method. They also studied its photocatayltic performance. They concluded that the MgO nanostructures exhibited good photocatalytic activity.
- Yu et al[46] have reported that the surface of the GO nanosheets is modified with the carboxyl groups. In this synthesis, the GO is functionalized with carboxyl group through the covalent bonding. They concluded that modification of the surface of the GO nanosheets having the non-covalent bonding includes hydrophobic effects, van der waals forces, π stacking interactions and the electrostatic interactions.
- Min Song et al[47] prepared the GO-gold composites in aqueous solution by one step reaction. During their synthesis process, the gold nanoparticles were implanted on the graphene oxide. Its BET analysis indicate that the surface area of the composite film was increased as compared to that of the synthesized GO. Through the modification of the composite on electrode, the electrochemical evaluation revealed increase in voltammetry response. They suggest its potential applications for making electrochemical sensors.

- K. Ganapathi Rao et al (2015)[48] have reported the successful synthesis of the MgO nanoparticles by green synthesis method. This green synthesis is also the environmental friendly synthesis by providing the recycle, reduce and reuse concept. This synthesis gives good results as compared to that of the chemical methods in some cases. The preparation of the MgO nanoparticles is done by the solution combustion synthesis using citric acid is a fuel. This citric acid is available in many forms just like lemon, tamarind and orange peel. In this work, the researchers synthesized MgO nanoparticles from the extract of orange peel by using Green synthesis method. The average crystallite size of nanoparticles was obtained at 18nm and face centered cubic structure was observed through the XRD analysis. The average particle size was found to 29nm from particle size analyzer. The bond analysis was performed by using the FTIR spectrum. The above characterizations revealed that the prepared MgO nanoparticles were in nano-range.
- Muhan Cao et al[49] have reported the synthesis of graphene oxide modified with $AgCl/Ag_3PO_4$ by using a simple method. XRD analysis reveals that phase structure of the composites have different quantities of GO and Mostly the diffraction peaks of the composites corresponding the body centered cubic phase of Ag_3PO_4.SEM results shown that the particle size of 3µm for Ag_3PO_4 and have irregular crystal structure. It is observed that some particles of AgCl were spread on the surface of Ag_3PO_4.The crystals of $AgCl/Ag_3PO_4$ were enwrapped with wave like GO nanosheets. The particles of Ag_3PO_4 show uneven surface to some extent. UV Vis. spectroscopy reveals that the broad and strong absorption peaks in the visible regions. EDAX mapping confirms that the small particles of $AgCl/Ag_3PO_4$ were dispersed on GO surface. TEM images of the composites $AgCl/Ag_3PO_4$ on the GO surface have shown the crumpled structure on which the $AgCl/Ag_3PO_4$ particles are dispersed. The composites of $AgCl/Ag_3PO_4$ exhibits the best photocatalytic activity in degrading dye like MB as compared to that of the pure Ag_3PO_4.
- Yongsheng et al[50_ENREF_50] have reported synthesis of Nickle ferrite-graphene, a magnetically separable photocatalyst with varying graphene ratio. TEM results reveal that the graphene sheets were completely decorated with small nano crystals of Fe_2O_4 having an average diameter of 6.05nm. When photocatalytic activity of nickle ferrite decorated with the graphene sheets were observed, they noticed a sudden change of $NiFe_2O_4$ from inert to an effective catalyst under visible light in degrading MB (methylene blue) and they

also measured it. It was observed that the hydroxyl radical oxidation was dominant in the photo degradation.

CHAPTER 2

2 Characterization Techniques

2.1 Powder X-Ray Diffraction(XRD):

X-rays are the type of electromagnetic radiation having wavelength of about 1angstrom (10^{-10}), that is about the same size as that of an atom. In the electromagnetic spectrum, X-rays lie in that portion between the gamma rays and the ultraviolet rays. X-rays was discovered in 1895 that enabled the scientists to investigate/examine crystalline structure at the atomic level. The diffraction of the X-rays is mainly utilized in two main areas, for the determination of their structure and for the finger print characterization of crystalline materials.

Bragg law is given by the equation

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

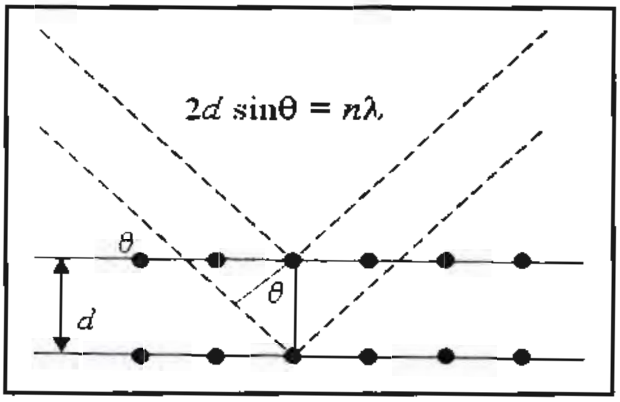

Figure 11: Schematic diagram of Bragg's Law

where

d is the spacing between the crystal planes

λ is the wavelength of the incident X-ray beam

n is an integer

ϑ is the angle between the incident ray and surface of the crystalline material that is derived by the English physicist Sir W.H Bragg and his son Sir W.L.Bragg in 1913 in order to explain the reason behind why the cleavage faces of crystals seems to reflect X-ray beam at certain angles of incidence(theta). The basis of the powder X-ray diffraction is that some of the crystallites of the powder sample will always be oriented to as to satisfy the Bragg equation and diffraction will occur. The requirement of the X-ray diffraction experiment is an X-ray source, the sample under investigation and a detector to pick up the diffracted X-rays.

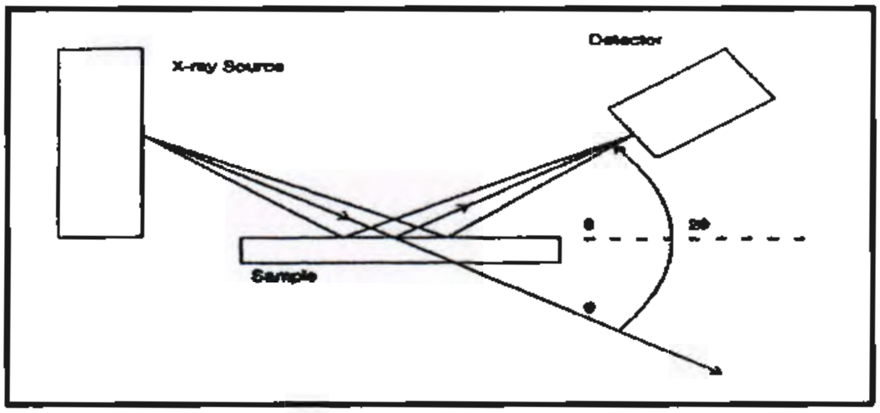

Figure 12: Schematic diagram of XRD

Schcrer in 1918 developed a method to calculate the crystallite size from XRD line broadening by using the Scherer equation:

$$d = K\lambda / (\beta \cos\vartheta)$$

where, d is crystallite size, K is Scherer constant, λ is wavelength of radiation, β is difference between the measured FWHM (full width at half maximum) and the broadened FWHM of single crystal, ϑ is the Braggs angle

2.2 Scanning Electron Microscope(SEM):

Scanning electron microscope is a type of microscope that utilizes electrons rather than that of light in order to form an image. Instead of using a light microscope, there are numerous advantages to using the SEM. The image resolution of a microscope has inverse relation to that of the wavelength of the electromagnetic radiation that illuminates the sample. Optical microscopes that utilizes the visible light having wavelength of about (400-800nm) have a maximum magnification of 1000 to 3000x.Whereas an electron microscope utilizes a monochromatic beam of electrons, that gives a much higher magnification in response. As SEM has quite large field depth, which permits the huge amount of the sample to be in focus at one time. High resolution of the SEM predicts that the closely spaced features can be probed at a higher magnification. Samples preparation for SEM is quite easy. Since mostly the requirement of the SEM for the sample is only to be conductive. Today in research areas , due to the combination of the higher magnification, greater resolution, larger depth of focus and ease of sample observation makes the SEM one of the most heavily used instruments.

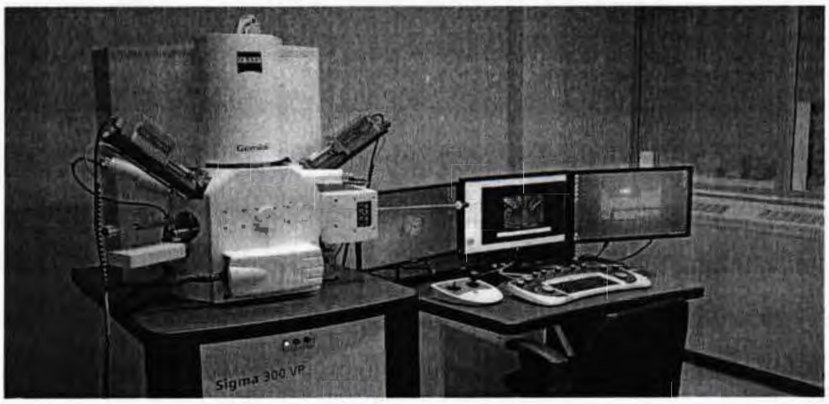

Figure 13: Scanning Electron Microscope

2.3 Fourier Transform Infra-Red Spectroscopy(FTIR):

FTIR is a noninvasive, powerful and widely applicable spectroscopy technique that is implemented in order to identify chemical functional groups. The development of FTIR is possible due to the invention of Michelson interferometer in 1880[51]. FTIR is considered to be an analytical instrument that is utilize to study the materials in the gas, liquid, or solid phase. For the qualitative and quantitative analysis of organic materials and particular polymers, FTIR spectroscopy has yet become one of the most important tool. The basis of the FTIR spectroscopy is on the interaction of infrared light with molecules. FTIR spectrum is created by the chemical bonds that absorbs energy. That energy content of the light is directly proportional to its wave number.

$$E=hcw$$

Where E is energy, w is the wavenumber ,both terms h and c are constant: h is the plank's constant ($h=6.63\times10^{-34}$) and c is the speed of light(3×10^{-8}).

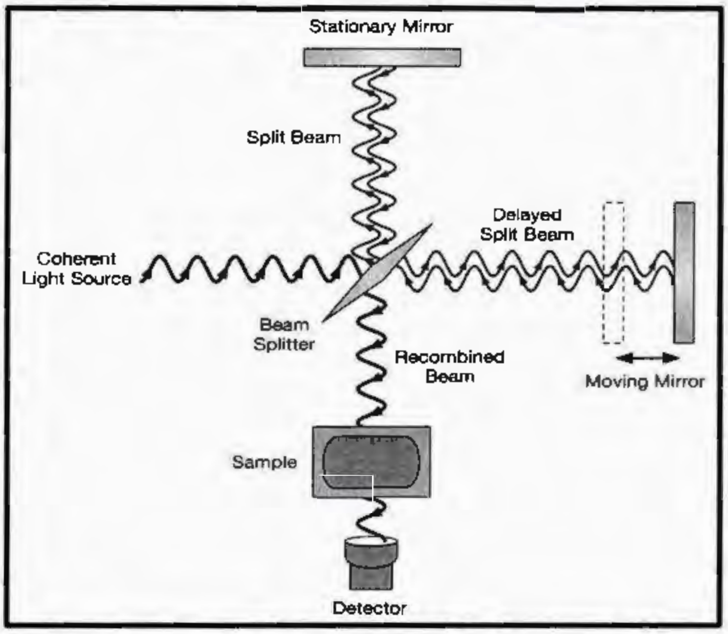

Figure 14: Schematic diagram of Michelson's Interferometer

Infrared light ranges of the wavenumbers between (4000-400 cm^{-1}). Actually different atomic groups absorbs at different IR wavenumbers that utilizes to investigate the structure of molecules. Consequently, the infrared spectrum can be considered as a finger print for the molecules

2.4 Energy Dispersive Analysis(EDS):

Energy Dispersive Analysis(EDS) gives its contribution by offering the elemental information. Basically, this elementary level information is about the composition of the sample that is of the surface of the sample. EDS is executed in conjugation with the SEM. All elements having atomic number lower than the carbon can be viewed with EDS.

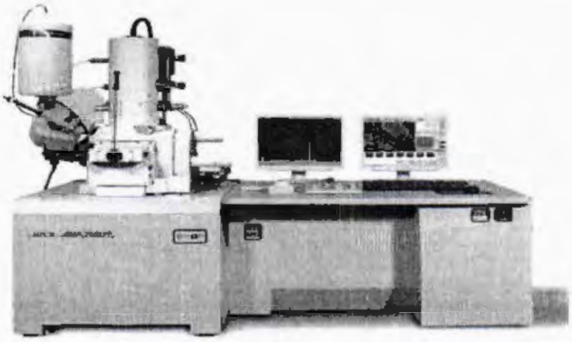

Figure 15: Energy Dispersive Analysis

2.5 Ultraviolet -Visible Absorption Spectroscopy:

(UV-Vis) retention spectroscopy is the estimation of light when it went through a sample. The rule of UV-Vis spectroscopy depends on the capacity of atom to assimilate ultraviolet and visible light. The retention of light relates to the excitation of external electrons in the molecule. At the point when an atom retains energy and the external electrons in the particle energized from the most astounding involved sub-atomic orbital to the least vacant sub-atomic orbital.

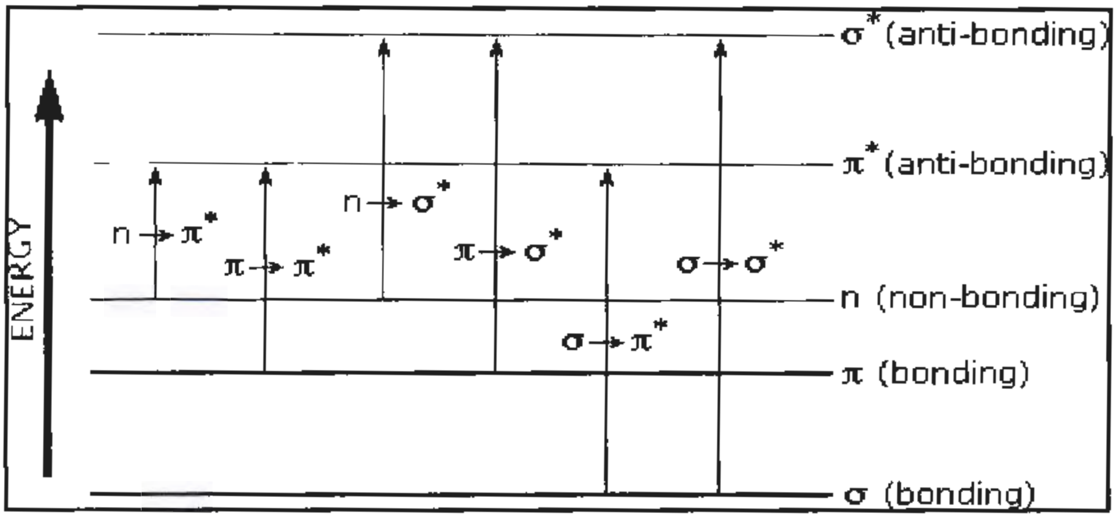

Figure 16: Schematic diagram of transitions in UV-Vis Spectroscopy

The assimilation can be measured at a solitary wavelength or on spectral expanded range. Ultraviolet and Visible spectroscopy are sufficient energetic to energize external electrons to high energy level and it is exceptionally helpful for amount estimation. The Beer Lambert law is utilized to decide the concentration of analyte by measuring the absorbance at different wavelengths. Actually, the relationship between the concentration and the absorbance is the Beer-Lambert Law. This relationship is given by the equation:

$$A=\epsilon cL$$

Where,

A is the absorbance

ϵ is the molar absorptivity

c is the concentration of the sample

L is the length of the cell

UV-Vis spectrophotometer can be utilized for the analysis of the absorption spectra. The light created from a Xenon flash lamp and is gone through the monochromator which splits the beam into various wavelengths out of the constant range. The intensity measured by the part of beam splitter. The transmitted power of the light beam is measured at photodetector and the absorbance is computed by the accompanying equation.

The assimilation is plotted as a component of the wavelength in a retention range. The molar absorptivity is computed by utilizing the above eq of Beer-Lambert Law.

$$A = \log\frac{I_0}{I} \quad \text{or} \quad A = -\log T$$

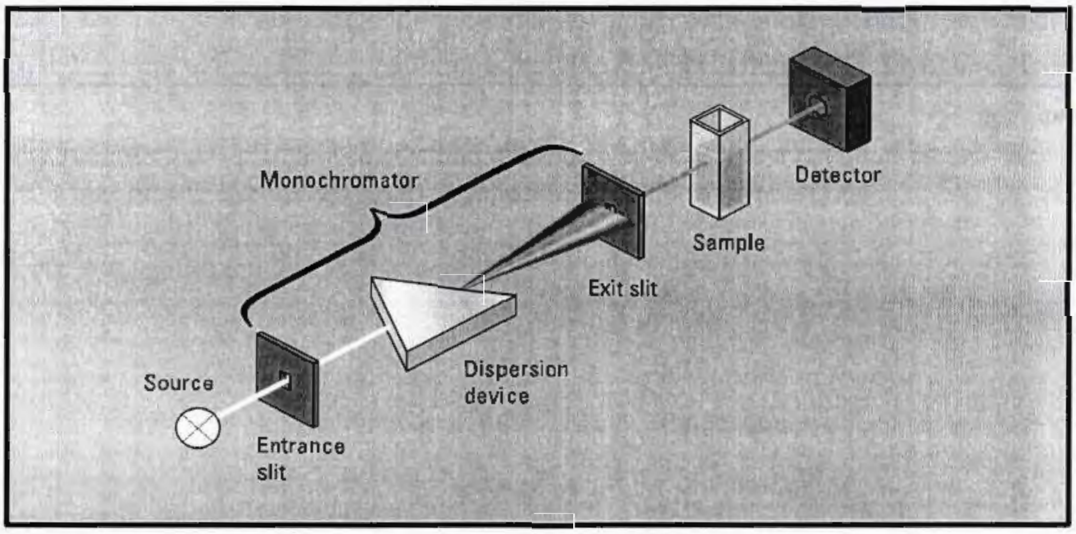

Figure 17: Schematic diagram of UV-Vis Spectrophotometer

Chapter No. 3

3 Experimental Procedure

In the experimental procedure, I synthesized magnesium oxide nanoparticles and graphene oxide nanosheets. After synthesis, I modify the surface of the graphene oxide nanosheets with the magnesium oxide nanoparticles.

3.1 Instruments:

Following instruments were used during the synthesis process.

- Beakers
- Magnetic stirrer
- Hot plate
- Centrifuge machine
- Furnace
- Sonication bath
- Photocatalytic reactor assembly
- Drying Oven

3.2 Chemicals used in the Synthesis:

Table 2: Chemicals used in the Synthesis

CHEMICALS	FORMULAS
Magnesium Nitrate	$Mg(NO_3)_2$
Sodium Hydroxide	$NaOH$
Pluronic P-123	$CO(CH_2CH_2O)_{20}(CH_2CH(CH_3O)_{70}(CH_2CH_2O)_{20}H$
Ethanol	$CH_3\text{-}CH_2\text{-}OH$
Sulphuric Acid	H_2SO_4

Nitric Acid	HNO_3
Natural Graphite	C
Potassium Permanganate	$KMnO_4$
Hydrogen Peroxide	H_2O_2
Hydrochloric Acid	HCl
Deionized Water	H_2O

3.3 Different ways to obtain the MgO nanoparticles:

Different synthesis routes can be followed to prepare magnesium oxide nanoparticles such as:

- sol-gel
- hydrothermal
- microwave assisted sol-gel
- green synthesis
- co-precipitation
- solvothermal

I followed the co-precipitation technique in order to prepare the magnesium oxide nanoparticles.

3.4 Why Co-precipitation?

I used co-precipitation technique just because it's considered to be the far best method for the synthesis of the nanoparticles without producing agglomeration in the yield. The second reason behind it is that size can be easily controllable in this technique. It is supposed to be noncomplex method and also produces a noticeable effect on surface properties of the final product as well as on the morphology.

3.5 Synthesis of the MgO nanoparticles by Co-precipitation method:

Following steps were followed for the synthesis of the Magnesium oxide nanoparticles via co-precipitation method.

- First of all, add 0.5gm of Pluronic (P-123) in 100ml of ethanol and stir it for 15 minutes and then add 15gm of Magnesium Nitrate $Mg(NO_3)_2$ in it. Then this solution was kept for stirring by using the magnetic stirrer until the complete dissolution of the contents. Name this solution as solution 1.
- Then add 6gm of Sodium Hydroxide (NaOH) in 50ml of ethanol and then stir it until the complete dissolution of the content. Name this solution as solution 2.
- After doing this, add solution 2 in solution 1 dropwise with the burette under the constant stirring for 3 hours. Keep the time interval 4-5 minutes between 2 drops.
- Keep this solution in a room temperature for 24 hours.
- After the completion of 24 hours, the solution is kept for drying on a hot plate until the formation of powder.
- Then the powder was washed 4 times with distilled water and 4 times with the ethanol.
- Sonicate the above solution for 1 hour.
- Again, heat the above solution on a hot plate until the formation of powder.
- Calcine the above powder of magnesium oxide at 500^0C for 5 hours.
- After calcination, the Magnesium Oxide nanoparticles were finally synthesized.

Note: The magnesium nitrate $Mg(NO_3)_2$ and sodium hydroxide (NaOH) were used as a precursor and Pluronic (P-123) were used as a stabilizing agent.

3.6 Different ways to obtain graphene oxide sheets:

Different synthesis routes can be followed to prepare the graphene oxide sheets such as:

- Brodie method
- Staud's method
- Fast and facile method
- Hummer's method
- Improved Hummer's method
- Modified Hummer's method etc

3.6.1 Why Modified Hummer's method:

I followed the modified Hummer's method in order to synthesized the graphene oxide nanosheets just because it is considered as safe route for the synthesis of graphene oxide nanosheets. We can achieve better exfoliation state and faster oxidation rate through this method. High productivity of final oxidized product with the decrease demand of acid is also achieve through this method.

3.7 Synthesis of the Graphene Oxide Nanosheets:

3.7.1 Formation of the Expanded Graphite:

Following steps were followed in order to form expanded graphite.

- In the mixture of the concentrated solution of the nitric acid (HNO_3) and the sulphuric acid (H_2SO_4) of the same ratio (40ml:40ml), adding 18g of the graphite powder in it and then kept it for three days at the room temperature in order to expand graphite.
- Because of this process, sulphate ions and nitrate ions were introduces that's become the reason of causing the expansion between the graphite planes.
- The fluffy texture of the final expanded graphite was observed from the expansion of the graphite and consider it be having very low density.

3.7.2 Synthesis of the Graphene Oxide through Modified Hummer's method:

Following steps were followed to prepare the graphene oxide nanosheets via modified Hummer's method by using expanded graphite as precursors.

- Firstly, take a beaker containing 80ml of sulphuric acid(H_2SO_4) and stir it for 20 minutes at the room temperature.
- Keep it on stirring at the room temperature while adding 18g expanded graphite slowly with small interval.
- The beaker was then placed on an ice bath and setting temperature at 0^0C while adding 12g potassium permanganate ($KMnO_4$) in extremely small interval.
- After adding $KMnO_4$, remove the ice bath and continue stir solution for some time.
- Then add 400ml of deionized water in the solution for dilution and keep stirring at temperature 90^0C.Dark brown colour of the solution appears.

- Place the beaker on a water bath and for the uniform heating keep it at 90^0 C for 3 hours.
- After removing from the water bath, keep the beaker aside in order to settle down for 12 hours at a room temperature.
- After 12 hours, start stirring the solution for 30 minutes and then 8ml of hydrogen peroxide (H_2O_2) was added slowly in a small interval in the solution in order to reduce the residual $KMnO_4$ and MnO_2 to the soluble $MnSO_4$. This addition of H_2O_2 causes yellowish bubbles, which is the indication as the graphite is oxidized in to the graphite oxide.
- Centrifuge the above solution by adding 300ml deionized water while keeping stirring for some time.
- In order to remove the impurities of the metal ions from the solution, add 12ml of HCl in the solution and stirred it continuously for 3-4 hours.
- After washing the solution, I separate the particles of graphene oxide by subjecting the solution on the centrifugation.
- After centrifuging the solution, add double distilled in it and stirred it for 30 minutes and again apply the centrifugation phenomenon.
- Two colors were seen in the tube after the final centrifugation phenomenon. upper layers were yellowish brown indicating the single layer of graphene oxide and lower layer was the greyish black color graphene oxide. The upper layer was separated from the lower.
- The final product was dried on a hot plate in order to obtain the powdered form.
- Graphene Oxide nanosheets were ready to use for different purpose.

3.8 Surface Modification of GO with Magnesium Oxide Nanoparticles:

Following steps were followed for the surface modification of the graphene oxide nanosheets with the magnesium oxide nanoparticles.

3.8.1 Modification of MgO/GO nanocomposites with 5% dispersion phenomenon:

- Firstly, 1g of graphene oxide was added into 40 ml of ethanol and then stirred it for 3-4 hours in order to obtain the well dispersed solution. Name this solution as solution 1.

- Secondly, 0.05g of magnesium oxide was added into 30ml of ethanol and then stirred it for 3-4 hours in order to obtain the well dispersed solution. Name this solution as solution 2.
- Thirdly, adding solution 2 in solution 1 dropwise with a burette. The time interval between two drops is kept for 3-4 minutes.
- Centrifuge the above solution.
- After centrifugation, dried the above solution on a hot plate in order to make powder.

In this way, we finally dispersed the magnesium oxide nanoparticles on the graphene oxide nanosheets. Finally, we can say that modification is done through the dispersion phenomenon.

3.8.2 Modification of MgO/GO nanocomposites with the 10% dispersion phenomenon:

Same above procedure is done for modification of MgO/GO with 10 % dispersion. Only the quantity of magnesium oxide varies. In 10% dispersion, 0.1g magnesium oxide is used and other things remains same.

3.9 Photocatalytic Investigation:

Photocatalytic activity of the different materials like Graphene Oxide(GO), Magnesium Oxide(MgO) and GO/MgO nanocomposites with 5% and 10% dispersion is investigated under the exposure of visible light in degrading the organic dye like methylene blue(MB). Aqueous arrangement of MB is set up by including 0.025g of MB color in 1 liter of water. Outer cooling coat with reused water is utilized to control the temperature of photocatalytic reactor.

3.10 Photocatalytic investigation of GO/MgO nanocomposite:

In MB solution:

0.025g of created GO/MgO with 5% dispersion is scattered in 25ml arrangement of MB dye. Photocatalytic examination was begun by presenting the solution for Visible light. To accomplish the adsorption-desorption equilibrium suspensions is mixed consistently for 15 minutes. Some measure of the suspension is gathered after at regular intervals to screen the degradation of dye methylene blue(MB).

Same procedure is followed for the photocatalytic investigation of GO/MgO nanocomposite with the 10% dispersion.

Chapter No. 4

4 Results and Discussion

Structural and optical properties of the prepared nanoparticles and nanosheets by the co-precipitation and modified Hummer's method respectively as depicted in chapter 3 can be dissected by characterization. Following diverse characterization techniques has been utilized for investigating the properties.

- X-ray Powder Diffraction
- Scanning Electron Microscope
- Fourier Transform Infrared Spectroscopy

4.1 Structural Examination:

X-Ray diffraction is the most essential system for assurance structure of crystalline solids. It has been generally utilized for the assurance of crystallinity, crystal structure, nanoparticle size and lattice constants of nanowires, thin films and nanoparticles. As nanoparticles were created in fluid media, so they were changed over into thin movies through hot plate vanishing. Drops of water containing nanoparticles were included slide one by one. XRD estimations of the considerable number of tests were completed utilizing X-Ray diffractometer utilizing Cu Kα X-ray beam (λ=0.154nm). The outcomes got for various examples are examined in the accompanying segment.

4.2 X-ray Diffraction Examination:

XRD information and its investigation for every one of the examples in subtle elements are as per the following:

4.2.1 Magnesium Oxide:

The XRD pattern of MgO nanoparticles got from co-precipitation method were appeared in **Figure 18**. The outcomes demonstrated that the structure was in cubic structure and these outcomes were coordinated with the JCPDS card number (00-045-0946).Peaks were ingested at 2θ=36.9°,43°,62.2°,74.5°,78.6° alongside the miller indices (111),(200),(220),(311) and (222) respectively. As the width of the peak expands size of the particle estimate decreases, which takes after that present material in nano-range.

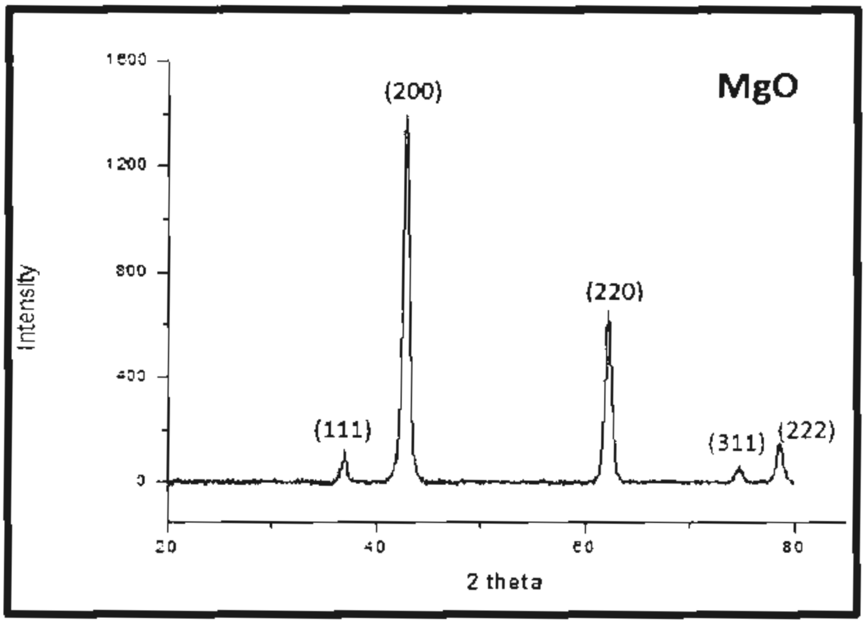

Figure 18: XRD pattern of MgO

4.3 Particle Size Examination:

Phase distinguishing proof utilizing XRD relies on upon position of peaks and in addition relative intensities of the peaks. Little crystallite estimate causes peak expanding. Notwithstanding portrayal of different crystalline structures, XRD can likewise be utilized to discover normal crystallite estimate. Crystallite size was gotten by Debye Scherer recipe by condition.

$$D = k\lambda / \beta\cos\theta$$

Where,

K is a constant (K=0.9)

λ =0.154 nm for Cu kα radiation

θ is diffraction angle

β is the full width at half maximum (β= FWHM*(π/180) is Bragg's angle of the most intensive peak divided by 2).

The average crystallite size of magnesium oxide nanoparticles prepared by the co-precipitation method calculated from the Debye Scherrer's formula was measured as 23.5nm.

Table 3: Structural parameters of MgO obtained from XRD results

Sr No.	[2 Theta]	Heights	FWHM[2 Theta]	d-spacing	hkl	Rel.Int[%]	Size D(nm)	Average crystallite size D(nm)
1-	36.9542	119.40	**0.3444**	2.43255	111	9.41	24.11	23.534
2-	43.0167	1269.08	0.1476	2.10273	200	100.00	56.42	
3-	62.2219	616.82	0.5904	1.49205	220	48.60	14.05	
4-	74.5214	54.61	0.7872	1.27334	311	4.30	10.53	
5-	78.6559	148.50	0.6600	1.21545	222	11.70	12.56	

4.4 Comparative XRD Analysis of Different Materials:

In **Figure 19**, curve a GO (Graphene oxide) shows its prominent peak at 2θ =25.4° degree indicating the (002) plane. Curve b MgO (Magnesium Oxide) shows its prominent peaks were ingested at 2θ= 36.9°,43°,62.2°,74.5°,78.6°degrees alongside the miller indices (111), (200), (220), (311) and (222) respectively. Other details of this material were given in the previous section. Whereas curve c and d with 5% and 10% dispersion, the prominent peak of graphene oxide is still present at 2θ =25.4° degree depicting the (002) plane. While the prominent peak of MgO is suppressed at 2θ =43.0° degree indicating the (200) plane just because its dispersion is less in this direction and give more dispersion along (111) plane at 2θ =34.8° degree with a slight shifting from 2θ =36.9° degree of MgO.

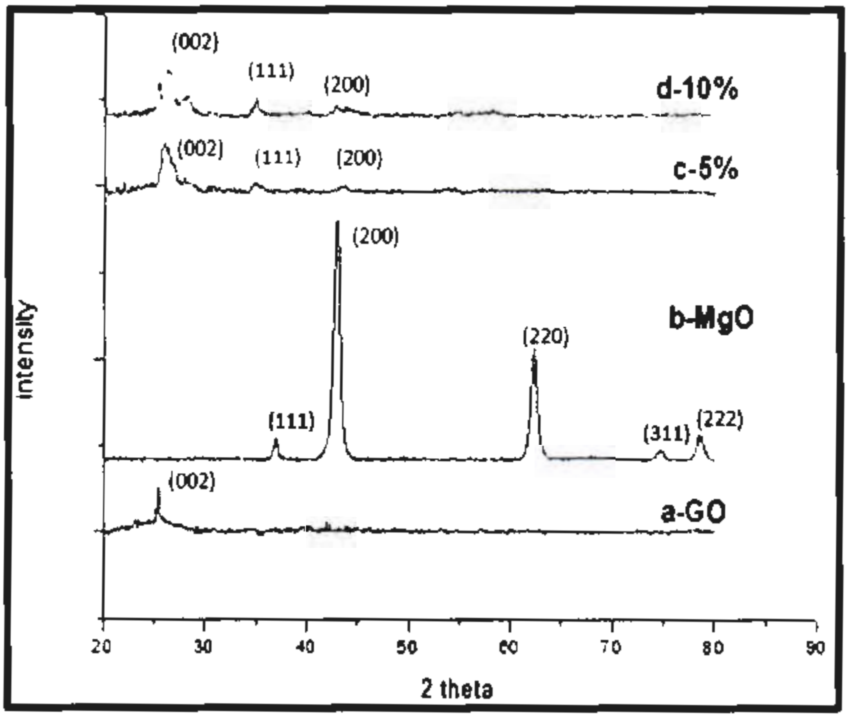

Figure 19:Comparative XRD Analysis of all materials

4.5 Scanning Electron Microscope Examination:

To acquire the size of nanoparticles, and in addition to watch the structural morphology of nanoparticles, SEM estimations and examination was performed. As nanoparticles were blended in fluid media, so for SEM examination drops of water containing nanoparticles was dried on slide. At that point these slides were placed in particle sputtering gadget demonstrate for lead plating. SEM of the considerable number of tests was done utilizing expository Scanning Electron Microscope.

4.5.1 SEM Examination of Magnesium Oxide Nanoparticles:

Surface morphology and size of magnesium oxide has been studied by using scanning electron microscope. **Figure 20** shows SEM micrographs of the synthesized MgO nanoparticles. The SEM results clearly reveals the spherical granules structures and the size of the nanoparticles ranging from 56.77 nm to 67.59 nm.

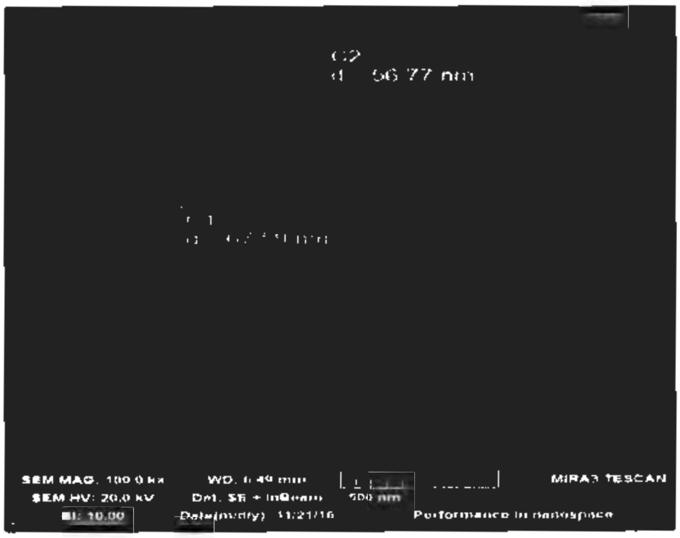

Figure 20: SEM image of MgO nanoparticles

4.5.2 SEM Examination of Graphene oxide Nanosheets:

From the SEM picture given in **Figure 21**, It is observed that GO has layered structure, which bears thin GO nanosheets. They are transparent with few layers, which affirmed the existence of 2-D nanosheets of GO. Wrinkles are conspicuous in a GO nanosheets are like wavy silk cover. These sheets are collapsed with few layers and it is conceivable to recognize the edges of individual sheets, including the crimped or wrinkled zones.

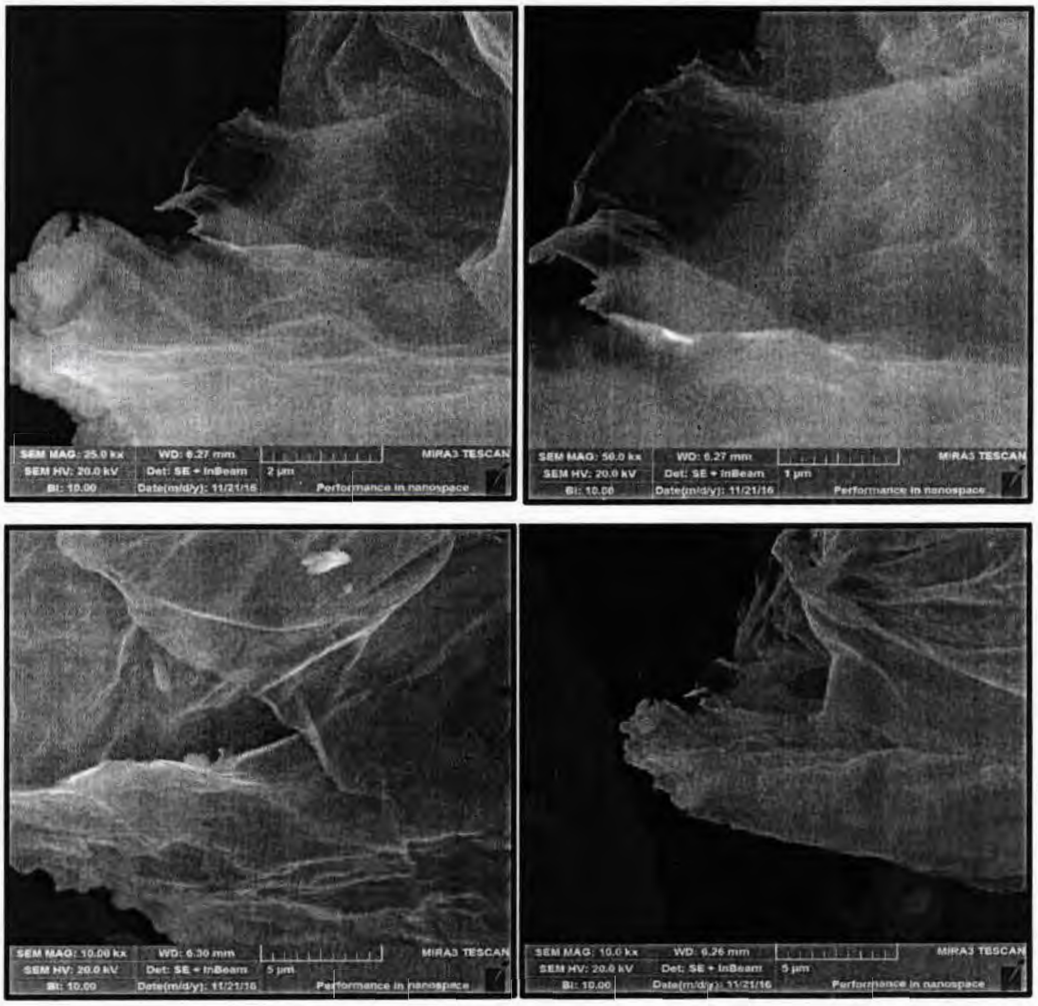

Figure 21: SEM image of Graphene Oxide nanosheets

4.5.3 SEM Examination of MgO/GO nanocomposites with 5% Dispersion:

Figure 22 shows the SEM image of surface modification of MgO/GO nanocomposites with the 5% dispersion. It is observed that the average 62.18 nm sized MgO nanoparticles are dispersed separately and also uniformly on the surface of the Graphene Oxide nanosheets.

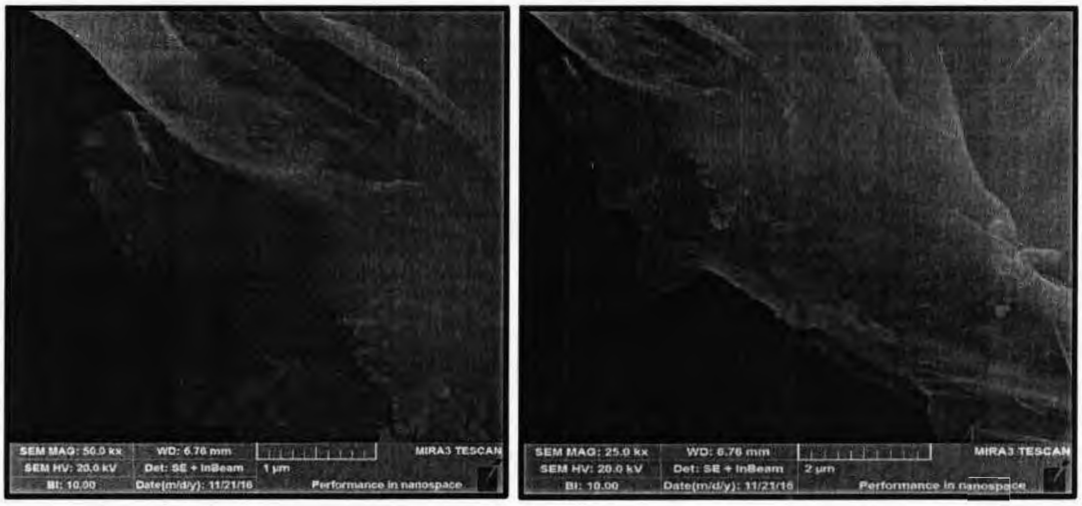

Figure 22:SEM image of MgO/GO nanocomposites with 5% dispersion

4.5.4 SEM Examination of MgO/GO nanocomposites with 10% Dispersion:

Figure 23 shows that the SEM image of surface modification of MgO/GO nanocomposites with the 10% dispersion, it is observed that most of the MgO nanoparticles were dispersed separately and also uniformly on the surface of GO nanosheets but a few points agglomerated MgO nanoparticles were also seen. These dispersions cause the suppression of the GO nanosheets.

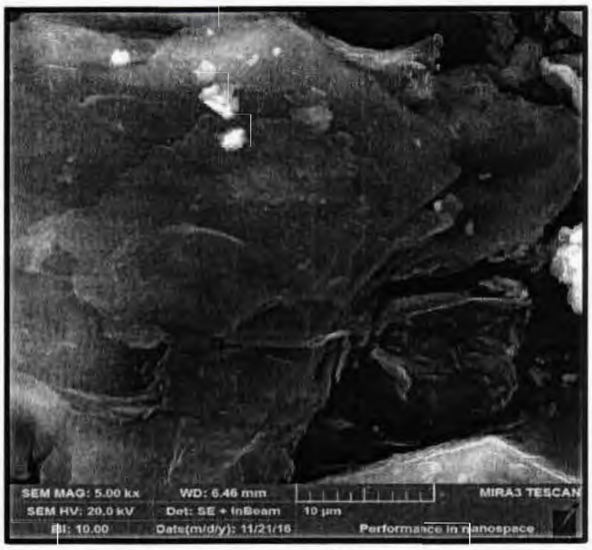

Figure 23: SEM image of MgO/GO nanocomposites with 10% dispersion

4.6 FTIR Examination of different Materials:

4.6.1 FTIR Spectrum of Graphene Oxide:

Figure 24 shows that the FTIR spectrum of the Graphene Oxide. Sharp absorption peak at 3464cm-^{1} that lie in the region (3600-3200) cm-^{1} depicting the stretching vibrations of the surface hydroxyl(-OH) groups on the surface of Graphene Oxide. The peak at 1656cm-^{1} that lie in the region (1680-1640) cm-^{1} indicating the C=C stretching vibrations of the aromatic rings. The peak at 1407cm-^{1} is found to be the stretching vibrations of the C-O of the carboxylic acid. Two peaks at 820cm-^{1} and 700cm-^{1} that lie in the region (870-610) cm-^{1} representing the C-H stretching vibrations of the phenyl rings and alkynes.

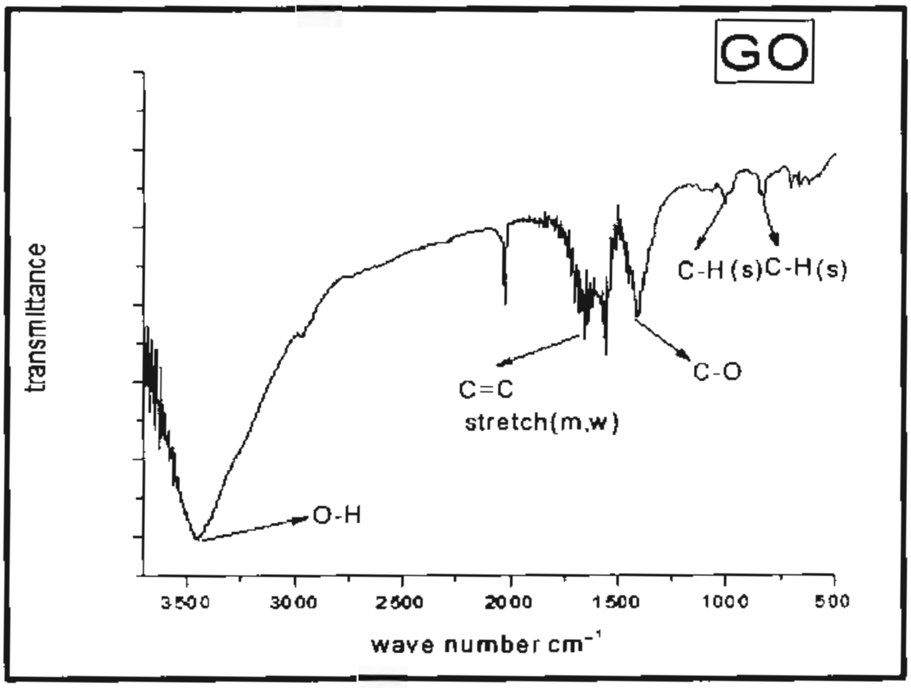

Figure 24: FTIR spectrum of Graphene Oxide

4.6.2 FTIR Spectrum of Magnesium Oxide:

Figure 25 shows that the FTIR spectrum of Magnesium Oxide. The wide band at 3463.9cm-^{1} that lie in the region (3600-3160) cm^{-1} is ascribing the extending recurrence of the hydroxyl (-OH) groups on the surface of graphene oxide. Broad band close to 1382.7cm^{-1} is because of C=O extending recurrence demonstrates the aromatic ring. For the most part wide band at around 433-769cm^{-1} is assigned to the metal oxide twisting vibrations. The band at 548cm-^{1} demonstrating the Mg-O.

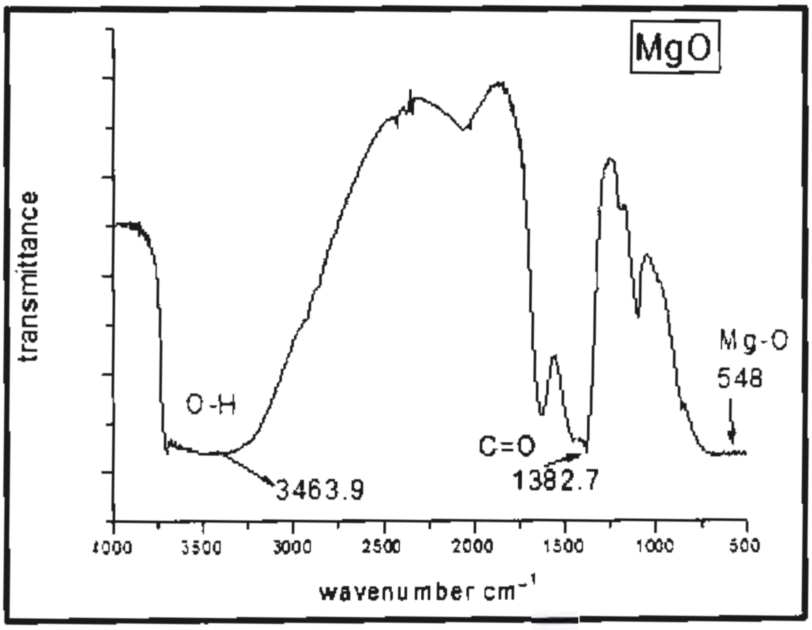

Figure 25: FTIR spectrum of Magnesium Oxide

4.6.3 FTIR Spectrum of nanocomposites (5% Dispersion):

Figure 26 shows that the FTIR spectrum of 5% dispersion. Absorption peak at 3462.8cm-^{1} lying in the region (3600-3200) cm-^{1} portrays the extending vibrations of surface hydroxyl (- OH) groups on GO surface. Peak at 1658cm-^{1} depicting the C=C extending vibrations of the aromatic rings. Peak at 821cm-^{1} showing the C-H extending vibrations of the phenyl rings. Small peak at 548cm-^{1} delineates the Mg-O of magnesium oxide.

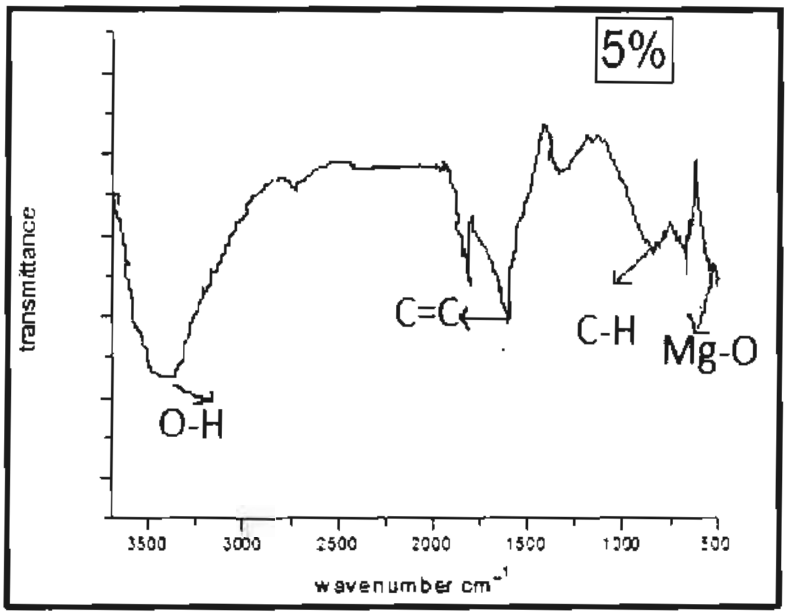

Figure 26: FTIR spectrum of nanocomposites (5% dispersion)

4.7 Photocatalytic applications of MgO/GO nanocomposite:

Figure 27 shows that the comparative analysis of the % Degradation of photocatalysts under Visible light irradiation for an irradiation time of 4 hours by utilizing the Methylene Blue Dye. No clear degradation happens when GO utilized as photocatalyst showing no photocatalytic activity. Whereas dispersion of MgO on GO displays upgraded photocatalytic execution contrasted with that of GO. The reason behind it is that connecting MgO with different materials, for example, respectable semiconductors and carbon based materials, for example, graphene oxide has demonstrated the change of photocatalytic movement by decreasing the electron-gap match recombination rate.

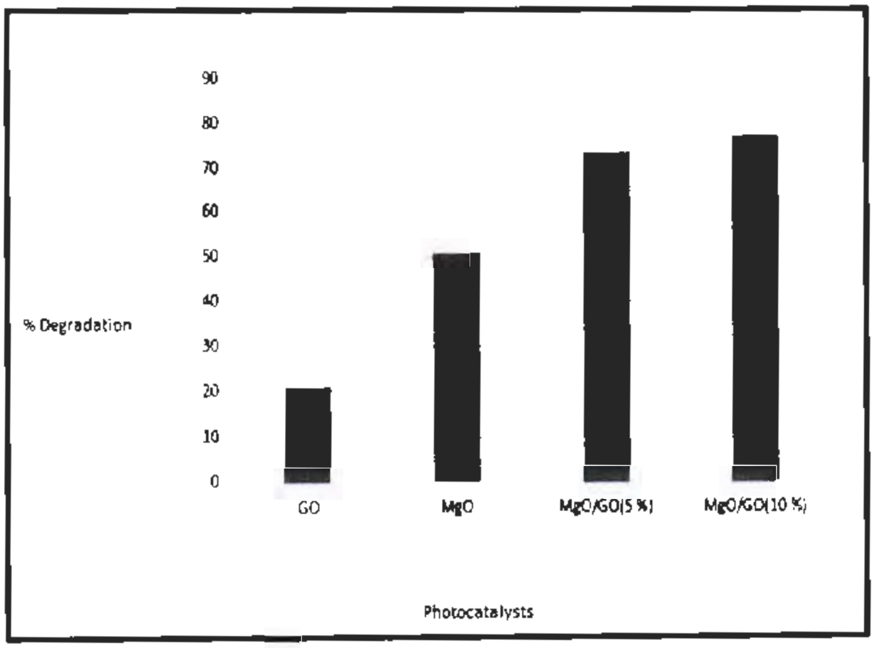

Figure 27: % Degradation of photocatalysts

Conclusion

Magnesium Oxide and Graphene Oxide has been successfully synthesized by utilizing the co-precipitation method and modified hummer's method respectively. Surface modification of Graphene Oxide with Magnesium Oxide is done through the dispersion method that were further proved to be photocatalytic under the visible light irradiation for an irradiation time of 4 hours. Prepared samples were characterized through SEM, XRD and FTIR techniques. These characterizations revealed that average 62.18 nm sized magnesium oxide nanoparticles were dispersed uniformly and separately on the GO nanosheets. FTIR results confirmed the bonding of Mg-O. The dispersion of MgO on GO causes an increase in photocatalytic activity in contrasted with that of the GO.

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