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Minor Research Project
on
“Synthesis And Antimicrobial Evaluation Of Novel Heterocycles From 9-Acetyl Anthracene

By
Mr. Harshal Ashok Dabhane

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SYNTHESIS AND ANTIMICROBIAL EVALUATION OF NOVEL HETEROCYCLES FROM 9-ACETYL ANTHRACENE.

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Declaration

The research work embodied in this minor researchprojecthas been carried out by me in the Department of Chemistry, G.M.D. Arts B.W. Commerce and Science College, Sinnar, Nashik.The extent of information derived from the existing literature has been indicated in the body of the minor research report at appropriate places giving the references.The work is original.

Place : Sinnar — Mr.Harshal Dabhane

Date : — **Principal Investigator**

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General Introduction

The present work entitled “Synthesis and Antimicrobial Evaluation of Novel Heterocycles From 9-Acetyl Anthracene” describes the series of synthesis of 9-acetyl anthracene and their various activities.Literature survey of the above compounds revealed that Magnesium oxide nanoparticles are a class of nanomaterials characterized by their high surface area, thermal stability, and multifunctional properties. They have been extensively explored in various fields such as catalysis, environmental remediation, medicine, and energy storage.

MgO nanoparticles exhibit significant antimicrobial activity, making them effective against a variety of pathogens, including bacteria, fungi, and viruses. This property is attributed to their unique physicochemical characteristics, such as a high surface area, basic nature, and ability to generate reactive oxygen species (ROS).MgO nanoparticles represent a versatile and promising material with potential applications across various domains. Ongoing research aims to address current challenges and expand their usability, particularly in environmentally friendly and biomedical technologies.

The present work is broadly classified into Four Chapters:

Parts-I

Synthesis Of MgO NP :

This Part describe the synthesis of Magnesium Oxide Nanoparticle.

Chapter-II
Methods Of NP’s Synthesis

This Part the decsribe the various method for synthesis of Nanoparticles.

The synthesis of nanoparticles (NPs) is a crucial process in nanotechnoloas it allows for precise control over the size, shape, composition, and functionality of the particles. Methods for synthesizing nanoparticles can be broadly categorized into physical, chemical, and biological approaches.

Chapter-III

Charecterisation studies Of NP’s

This part describe the various technique to charectrised the Nanoprticles.

Characterization of nanoparticles involves analyzing their properties to determine their suitability for various applications, such as drug delivery, catalysis, imaging, and electronics. The characterization process focuses on understanding the size, shape, structure, surface properties, composition, and functionality of nanoparticles.

Chapter-IV

Application Of Nanoparticles.

This Part Describe the various application of Nanoparticle.

Nanoparticles are ultra-small particles ranging from 1 to 100 nanometer in size, possessing unique physical, chemical, and biological propertie Their applications span numerous fields due to their high surface area, quantum effects, and tunable properties.

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Chapter-I

Introduction Of Nanotechnology

1.1.1 Introduction:

Recently, concept of nanotechnology is a new emerging field of the technology which allows high exceptions of its talent to change the world. The nanotechnology is a branch of science and engineering deals with materials having dimensions in the range of 100 nm scale or less.The idea of nanotechnology was first recognized by Nobel Prize winner, An American scientist Richard Feynman who gave fanciful speech in 1959 but it was published in 1960. The history of nanotechnology dates very long back to 1959, when Richard Feynman a Physist at Cal Tech. In one of his famous lecture he said “There is plenty of room at bottom” and suggested that scaling down to nano level and starting from the bottom was key to future technology. The historic background of nanoparticles starting with name of Paul Ehrlich and then first efforts by Ursula Scheffel and his co-workers Professor Peter Speiser at the ETH Zürich in the late 1960s and early 1970s.

Now a day, nanotechnology is a facilitating technology that deals with nanometre sized particles in several fields of science such as Chemistry, Physics, Biotechnology and also in Material science. In recent years, many chemical and Literature Review on Metal Oxide Nanoparticles and its Applications in Organic Transformations Chapter-I Page 2 physical methods have been developed for the synthesis of metallic nanoparticles and it serves as an important method in the development of non-toxic, clean and eco-friendly procedure . Nanotechnology, which is one of the novel technologies, discusses to the development of structures, devices and systems having size ranges from 1nm to 100 nanometers (nm). Since the past few decades progress has been seen in every side of Nanotechnology such as nanoparticles, nanolayers, powders, optoelectronics, and mechanical nanodevices and in also nanostructured biological materials. Currently, nanotechnology is expected to be powerful technology in the next 20-30 years in all fields of science and technology.

Nanotechnology has capacity to control, appreciate and operate matter at the level of individual atoms and molecules. The first definition of the nanotechnology was introduced after the consolations with experts in over 20 countries in 1987-1998 and is defined as “The size range of the material structure under the consideration the length of intermediate range between a single atom and molecules about the 100 nm. It also defined as “It is study of design, production, characterization and application of structures, devices and systems or by controlling size and shape at the nanoscale level. In the second half of 1980s to the early 1990s a number of significant discoveries and innovations was made, which created a most essential effect on the further development of the nanotechnology. In 1991 first nanotechnological program was started in USA.

Before thousands of years, several nanomaterials were used for medicinal and esthetical purpose like colloidal gold metals was used to develop ruby glass and also for colored ceramics. At an ancient time, the British Colombian (BC) people were used various types of natural fabrics, cotton, silk, wool etc. They had developed network of pores with size up to 1-20 nm that means they were nonporous material in nature. Therefore, due to nonporous structure or material, natural fabrics possess very high useful properties and they also absorb sweet well, quickly swell and dry. On other side, silver metal nanocrystals used in 16th century. In ancient Egypt, peoples were commonly used hair dye in black for a many times

Saving nanosize. It was believed that the Egyptians people used natural vegetables and dyes. Burial sites directed by Ph. Walter showed that hair was dyed in black color with paste from lime, lead oxide and in small quantity of water.

1.1.2 Classifications of nanomaterials:

On the basis of particle size the nanomaterials are classified into following major types viz:

1.Nanoparticles

2.Nanocapsules

3.Fullerenes

4.Dendrimers

5.Quantum dots

6. Nanostructures

7.Nanopores

1.1.2.1.Nanoparticles:

Nanoparticles are particles which are 1 to 100 nanometers in size. In nanotechnology, a particle is defined as a small object that behaves as a whole unit with respect to its transport and properties. The particles are further classified according to diameter, ultrafine particles are the same as nanoparticles and between 1 and 100 nanometers in size. Coarse particles cover a range between 2,500 and 10,000 nanometers. Fine particles are sized between 100 and 2,500 nanometers. Nanoparticle research is currently an area of intense scientific interest due to a wide variety of potential applications in biomedical, optical and electronic fields.

1.1.2.2.Nanocapsules:

The nanocapsules are nanoscale shells made of a nontoxic polymer.They are vesicular systems that are made up of a polymeric membrane which encapsulates an inner liquid core nenanoscale level. It has a myriad of uses, which include medical promising applications for drug delivery, food enhancement, nutraceuticals, and for the self-healing of materials. These beneficial of encapsulation methods are protect in the adverse environment, for controlled release, and for precision targeting.

1.1.2.3.Fullerene nanomaterials:

Fullerene is a molecule with total carbon composition and it is available in the different forms like ellipsoid, tube and hollow sphere. There are several types of fullerene such as buckyball clusters, mega tubes, polymers nano-onions and linked ball and chain dimers. The cylindrical features are called as carbon nanotubes. The spherical fullerenes are called as bucky ball. The megatubes are larger in diameter than nanotubes and prepared with walls of different thickness. The polymers are chain, two dimensional and three dimensional polymers are formed under the high temperature and high pressure conditions. The nano-onions are spherical particles based on multiple carbon layers surrounding a bucky ball core proposed for lubricants. The linked ball and chain dimers are two bucky balls linked by carbon chain. The fullerenes are used in various fields such as optical devices, as photo resistant in certain photoistho graphic processes, in microelectronics devices and also used as anti-oxidants. They are also acts as HIV- protease and inhibit the catalytic activity of enzymes.

1.1.2.4.Dendrimers:

The dendrimer is another most important type of nanomaterial. Dendrimers are repetitively branched molecule. The dendrimers are polymers and it contains the several types of branches. The shape of the dendrimernanomaterials are chain like and also have numerous chain ends that can be made to have precise functions normally recycled for chemical reactions. Some types of dendrimer have three dimensional structures and have an interior cavity into which other molecules can fit. The drug supply is one use of this internal cavity found in dendrimers. They are also used in gene therapy as well as in medical diagnosis. Dendrimer is typically symmetric around the core, and often adopts a spherical three-dimensional morphology. A dendron usually contains a single chemically addressable group called the focal point. The difference between dendrons and dendrimers is illustrated in figure one, but the terms are typically encountered interchangeably.

1.1.2.5.Quantum dots:

A quantum dot is a semiconductor nanostructure that limits the motion of valence band holes, conduction band electrons, or excitations in all three spatial directions.The confinement can be due to electrostatic potentials (generated by external electrodes, doping, strain, impurities), the presence of an interface between different semiconductor materials (e.g. in core-shell nanocrystal systems), the presence of the semiconductor surface (e.g. semiconductor nanocrystal), or a combination of these.A quantum dot has a discrete quantized energy spectrum.The corresponding wave functions are spatially localized within the quantum dot, but extend over many periods of the crystal lattice.A quantum dot contains a small finite number (of the order of 1-100) of conduction band electrons, valence band holes, or excitons, i.e., a finite number of elementary electric

charges. Small quantum dots, such as colloidal semiconductor nanocrystals, can be as small as 2 t10 nanometers, corresponding to 10 to 50 atoms in diameter and a total of 100 to 100,000 atoms within the quantum dot volume 6 Nanostructures A Nanostructure is a structure of intermediate size between microscopic and molecular structures. Nanostructural detail is microstructure at nanoscale. In describing nanostructures it is necessary to differentiate between the numbers of dimensions on the nanoscale. Nanotextured surfaces have one dimension on thenanoscale, i.e., only the thickness of the surface of an object is between 0.1 and 100 nm. Nanotubes have two dimensions on the nanoscale, i.e., the diameter of the tube is between 0.1 and 100 nm; its length could be much greater. Finally, spherical nanoparticles have three dimensions on the nanoscale, i.e., the particle is between 0.1 and 100 nm in each spatial dimension.

1.1.2.7.Nanopores:

A nanopore is a tiny hole in a thin membrane, typically just big enough to allow a single molecule of DNA to pass through. They are powerful sensors of molecules and ions and have potential applications in many areas of technology.When a nanopore is present in an electrically insulating membrane, it can be used as a single-molecule detector. It can be a biological protein channel in a high electrical resistance lipid bilayer, a pore in a solid-state membrane or a hybrid of these - a protein channel set in a synthetic membrane. The detection principle is based on monitoring the ionic current passing through the nanopore as a voltage is applied across the membrane. When the nanopore is of molecular dimensions, passage of molecules (e.g., DNA) cause interruptions of the "open" current level, leading to a "translocation event" signal. The passage of RNA or single-stranded DNA molecules through the membrane-embedded alpha-hemolysin channel (1.5 nm diameter), for example, causes a ~90% blockage of the current (measured at 1 M KCl solution.)

1.1.3.Methods for Synthesizing Nanoparticles (NPs)

Nanoparticles (NPs) can be synthesized using various approaches broadly categorized into **top-down** and **bottom-up** methods. Each technique offers distinct
Advantages depending on the desired properties, application, and material type.

1.Top-Down Methods

In top-down approaches, larger bulk materials are broken down into nanoparticles through physical and mechanical means.

a.Mechanical Milling

Involves grinding bulk materials into nanosized particles using ball mills or attritors.

Advantages: Simple, scalable for industrial use.

Applications: Metallic and ceramic nanoparticles.

b.Laser Ablation

A high-energy laser beam is directed at a bulk material, vaporizing it to form nanoparticles.
Advantages: Produces highly pure nanoparticles.
Applications: Metal oxides, semiconductors.

c.Lithography

Techniques like electron beam lithography or photolithography .
createnanopatterns on a substrate.
Advantages: High precision and control over size and shape.
Applications: Electronics and photonics.

2.Bottom-Up Methods

In bottom-up approaches, nanoparticles are synthesized from atomic or molecular precursors through self-assembly or chemical reactions.

a.Chemical Reduction

Metal salts are reduced to nanoparticles using reducing agents (e.g., sodium borohydride or citrate).

Advantages: Simple, cost-effective.

Applications: Gold, silver, and other metal nanoparticles.

3.Sol-Gel Process

Metal alkoxides or metal salts are hydrolyzed and condensed to form nanoparticles.

Advantages: High control over size and composition

Applications: Oxides, ceramics.

c.Hydrothermal and Solvothermal Synthesis

Reactions are conducted in an aqueous or organic solvent under high pressure and temperature.

Advantages: Produces uniform and crystalline nanoparticles.

Applications: Metal oxides, sulfides, and hybrid materials.

d.Green Synthesis

Uses biological agents like plant extracts, bacteria, or fungi to
synthesize nanoparticles.
Advantages: Eco-friendly and non-toxic.

Applications: Biocompatible nanoparticles for medical use.

e.Vapour Deposition Technique

Chemical Vapor Deposition (CVD): Precursors in gaseous form react to deposit nanoparticles on substrates.

Physical Vapor Deposition (PVD): Material is vaporized and condensed into nanoparticles.
Advantages: High precision and scalability.
Applications: Thin films, coatings.

f. Co-Precipitation

Metal salts are precipitated in a solution to form nanoparticles, often stabilized with surfactants.
Advantages: Simple and widely applicable.
Applications: Magnetic and oxide nanoparticles.

1.1.4.Discussion:

Nanotechnology is an interdisciplinary field that focuses on understanding and manipulating matter at the nanoscale, where unique physical, chemical, and biological phenomena emerge. This concept has revolutionized multiple industries and scientific disciplines, offering both opportunities and challenges. Nanotechnology is a powerful and versatile field offering groundbreaking advancements. Its potential to revolutionize industries and improve quality of life is immense, but careful attention must be given to ethical, environmental, and regulatory concerns to ensure its responsible and equitable use. Nanotechnology represents a transformative frontier in science and engineering, enabling the manipulation of matter at the nanoscale to unlock properties and functionalities that are unattainable in bulk materials. Its interdisciplinary nature bridges physics, chemistry, biology, and engineering, offering innovative solutions to challenges across diverse fields such as medicine, electronics, energy, and environmental science.While the potential of nanotechnology is immense, it also introduces significant challenges, including potential health and environmental risks, ethical considerations, and the need for robust regulatory frameworks.

Addressing these concerns requires a collaborative approach involving researchers, policymakers, and industry stakeholders to ensure its responsible and equitable application.

As nanotechnology continues to evolve, it holds the promise of revolutionizing industries, improving quality of life, and contributing to a sustainable future. By harnessing its potential

responsibly, humanity can pave the way for groundbreaking advancements that benefit society as a whole.

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Chapter-II

Methodology for Nanoparticles Synthesis

2.2.1.Introduction:

Nowadays, size and shape selective syntheses of nanoparticles (NPs) and their variedcatalytic applications aregaining significant enthusiasm in the field for nanochemistry. Homogeneous catalysis is crucial due to its inherent benefits like high selectivityand mild reaction conditions. Nevertheless, it endures with serious disadvantages of catalysts and/or product separation/recycles as compared to their heterogeneous counterparts restricting their catalytic applications. The utilization of catalysts in the form of nano-size is an electivemethodology for combinations of merits of homogeneous and heterogeneous catalysis. Magnesium oxide (MgO)NPs are important as they find applications for catalysis, organic transformations, synthesis of fine chemicals and organic intermediates. The applications of MgO NPs in diverse organic transformations including oxidation, reduction, epoxidation, condensationand C-C, C-N, C-O, C-S bond formationin variety of notable heterocyclic reactions are also discussed. The use of MgO NPs in organic transformation is advantageous as it mitigates the use of ligands; procurable separation of catalyst for recyclability makes the protocol heterogeneous and monetary. MgO NPs gave efficacious catalytic performancetowards desired products due to high surface area. By considering these efficient merits, scientists have focused their attentions towards stupendous applications of MgO NPs in selective organic transformation.In current review article, we summarized the synthesis of MgONPsand numerous characterization techniques, whereas the application section illustrates their utility as a catalyst in several organic transformations. We believethis decisive appraisal will provide imperativedetails to further advance the applications of MgO NPs in the selectivecatalysis.

2.2.2.Literature Survey:

Nanotechnology is one of the treasured and superb disciplines that serve the top-down and bottom-up approach, which contains physical, chemical, and biological (plant, microorganisms, and biomaterials) methods for synthesizing multifunctional nanomaterials .Nanoparticles (NPs) are a multifaceted class of materials that include particulate materials having dimensions 1–100 nm . Those mentioned above physical and chemical techniques are initial time eating, which utilizes excessive energy, the requirement of reducing agents. Also, they are not eco-friendly, which makes them want to discover and innovate methods that conquer the shortcomings of these chemical and physical methods. As evaluated with physical and chemical methods, the biological methods for synthesizing NPs seem pleasant due to their fascinating packages and fewer requirements. It is quick, nontoxic, economical, energy-

efficient, and follows the standards of principles of green chemistry . Plenty of research was done on synthesizing metal oxide NPs supported by other materials like g-C3N4, which enhances nanomaterial's photocatalytic and biological activity. There are numerous reports on the eco-benign production of MgO NPs, and the literature survey suggests that extract of diverse plants and their different parts have been used for the phytogenic fabrication of MgONPs . The MgO NPs possess excellent catalytic activities and are utilized for numerous organic reactions, as evident by literature . The exploitation of MgO NPs as a catalyst for various organic transformations is because of their non-toxic nature, high basicity, ready availability, low cost, and reproducibility . However, besides numerous applications of MgO NPs as a catalyst for organic transformations, the use of MgO NPs synthesized from the plant for organic reactions is not explored to its capacity. Nonetheless, few reports on using plant-mediatedMgO NPs for organic reactions .

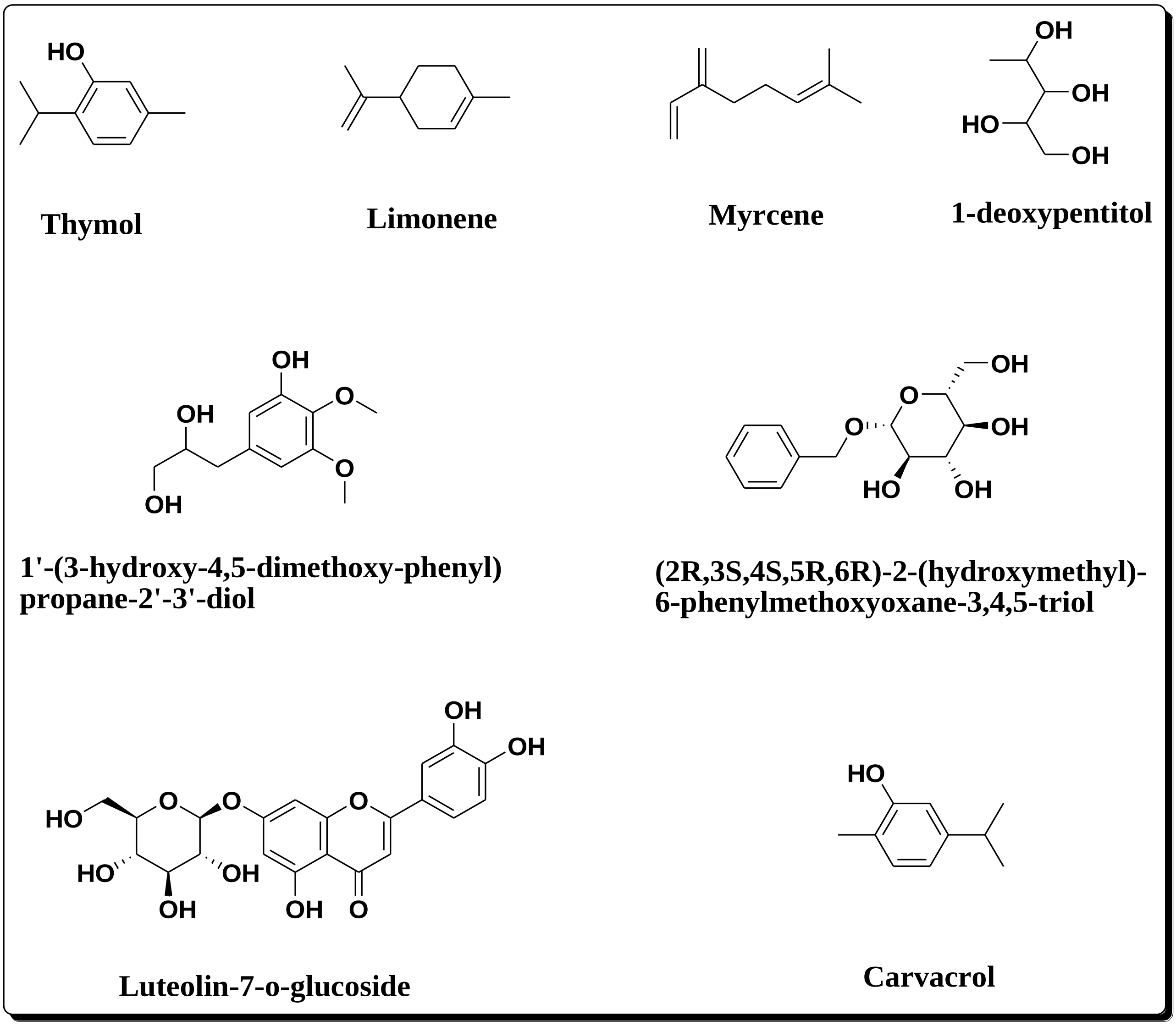


Fig.1 Active Phytochemical Constituent in Ajwain

Scheme 1. Model Claisen-Schmidt reaction of 9-acetylanthracene with benzaldehyde.

2.2.3.Present Work:

2.2.3.1.Chemical approach:

In chemical approaches, the chemical reduction method is commonly used for the fabrication of NPs.

Fig 3. Synthesis of MgO NPs by different chemical methods.

Along with this approach,chemical syntheses include a chemical vapor deposition (CVD) [24], sol-gel, thermal decomposition , co-precipitation , micro-emulsion , wet chemical method , chemical precipitation , sonochemical , microwave assisted , solvothermal , hydrothermal , spray pyrolysis, chemical bath , combustion , ionic liquid assisted , polyol-mediated , template assisted method ,andreversemicellar method (Fig**1**) etc.for syntheses of MgO NPs. In the case of chemical syntheses,magnesium metal salts are reduced by a reducing agent forming amagnesiumoxide nucleus and the growth of the nucleus of a particle is controlledby a capping agent who also resists

aggregation by electrostatic repulsion. The various sources of magnesium salt were used for the synthesis of MgO NPs like MgCl, $Mg(NO_3)_2$, $Mg(AcO)_2$, along with $NaBH_4$, phytochemicals present in plant, algae, bacteria, fungus and yeast which play different role as per condition as reducing, stabilizing and capping agents. The MgO NPs synthesized using various methods as mentioned figure 3 having different shape like spherical, rod, nanowires, nanoflowers, nanoflakes, ranging from 1 to 100 nm. The catalytic activity of synthesized MgO NPs depends upon their surface area, so the shape of MgO NPs effect the catalytic activity. At the time of fabrication of MgO NPs, the magnesium salt precursor mixed with suitable solvent and capping agents is used to control the morphology of MgO NPs, which minimizes agglomeration and an apt reducing agent.Reduces the starting magnesium salt precursor offavorableconditions with the subsequent formation of MgO NPs.

2.2.3.2..Physical approach:

Chemical routes for the synthesis of NPs usually includenoxious chemicals, which can be pernicious to the environment and human-being.Although
this approachcreates the shape and size selective MgO NPs, they needa capping agent, stabilizer as well as special additives to mitigate the agglomeration of MgO NPswhereas, physical approaches do not involve perilous chemicals and areusually rapid. Physical synthesis mostly includes the laser ablation [41], electric arc [42], thermal evaporation [43], flame combustion [44],pulsed laser deposition [45]and self-assemble process[70]for syntheses of MgO NPs(Fig. **4**). There is no solvent contamination in the synthesis of NPs using physical approach is the benefits of thisroutes in comparison with chemical approaches.

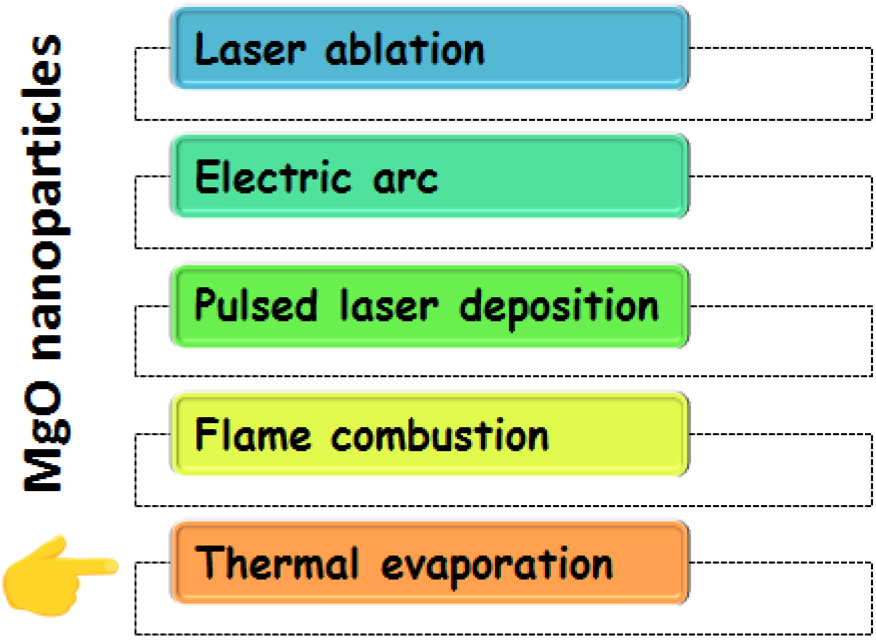


Fig 2. Synthesis of MgO NPs by different physical methods

2.2.3.3.Green approach:

Nowadays, research in biosynthesis is a completely greener domain forthe fabrication of NPs. The magnesium metal salt, reducing andcapping agents areefficient compounds in chemical method ofthe production of MgO NPs. Nevertheless, in biological approach,the reducing, capping and/or stabilizing agents can be used as naturalsources from the various plant extracts for the facile synthesis of MgO NPs of selective size and shape. Many researchers have focused on facileand greener approaches from the biogenic synthesis of NPs(Fig. **2**). Essien*etal.* applied greener approaches to the eco-benign synthesis of MgO NPsusingthe leaf extract from*Manihot esculenta*as reducing and/or capping agents withhexagonal shaped MgO NPs, having average size 36.7nm. Ogunyemi*etal.* reported the green synthesis of MgO NPsof diameter 18.2 nmusing *Matricaria chamomilla*flower extracts as reducing/capping agents at roomtemperature. Hong*et al.*demonstrated green synthesis of ball-like shaped MgO NPsof average size

50.95 nm fromthe leaf of *Pisonia grandis*.

Recently, researchers developed novel protocols for the procurable synthesis of MgO NPsusing diverse plant extracts as herbal reducing and/or capping agents to get various sizes and shapes of MgO NPs. For instance, the MgO NPssynthesis was achieved using Banyan latex , *Bauhinia purpurea*leaf extract ,*Calotropis gigantea*floral extract , *Carica papaya*leaf extract , *Chromolaena odorata* leaf extract[54], *Clitoria ternatea* extract, *Costus pictus* leavesextract etc. Wadhawan*et al.* demonstrated biogenic synthesis of MgO NPsby using *Syzygiumaromaticum* extract and theirapplication for the sensing of Fe^{3+} in real water samples .The use of microorganisms and fungi plays an extensive role in the synthesis of MgO NPs. The reducing and stabilizing constituents can be found in bacteria, fungi, yeasts, algae or plants which reduce the magnesium salt and control the size and shape of MgO NPs. In biosynthesis, the magnesium ions are precipitated and stabilized by various functional groups of the cell wall of fungi or bacteria. Subathra Devi*et al.* demonstrated MgO NPsfrom *Lactobascillus sp,* El-sayyad*etal.* used the *Penicillium chrysogenum*fungi for the synthesis of MgO NPs.Subathra Devi*et al.* reported biosynthesis of spherical MgO NPsusing microorganism and examined their anticancer activity against human leukemia celllines HL-60.In another study, Ebrahem*et al.* evinced the fungus-mediated biosynthesis ofspherical shaped MgO NPsusing *Aspergillus niger*with average size 43-91 nm .

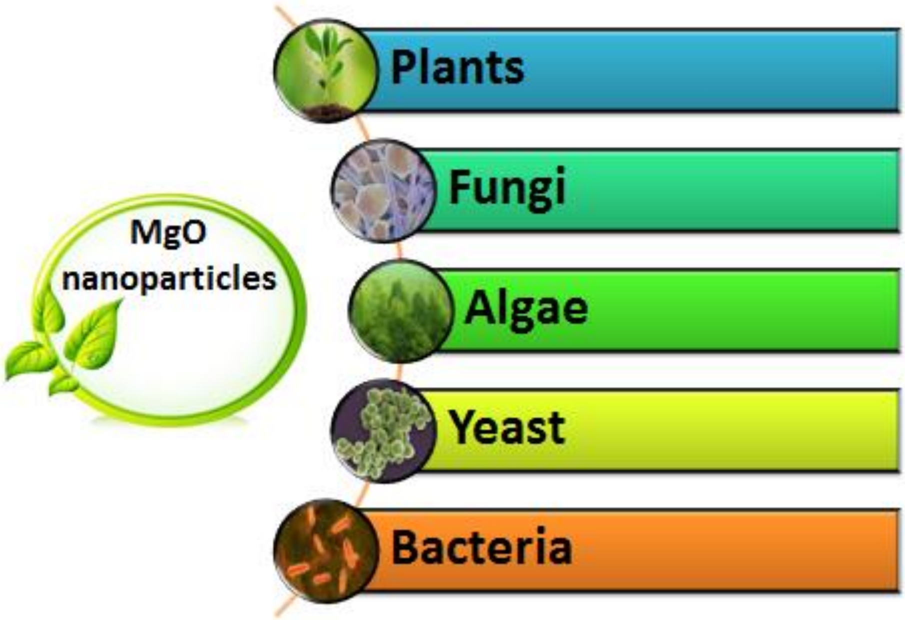


Fig 3. Synthesis of MgO NPs by different biological methods

2.2.3.4.Preparation of Ajwain leaves extract:

The fresh leaves of Ajwain (Trachyspermum ammi) were collected from Sinnar Tehsil and doubly washed with distilled water (DW). The 10 g of fresh leaves were crushed into tiny pieces and blended into 100 ml of DW, stirred, and then heated at 80–95 ◦C for 30 min. The resulting solution was filtered employing Whatman filter paper and further used to synthesize of MgO NPs.

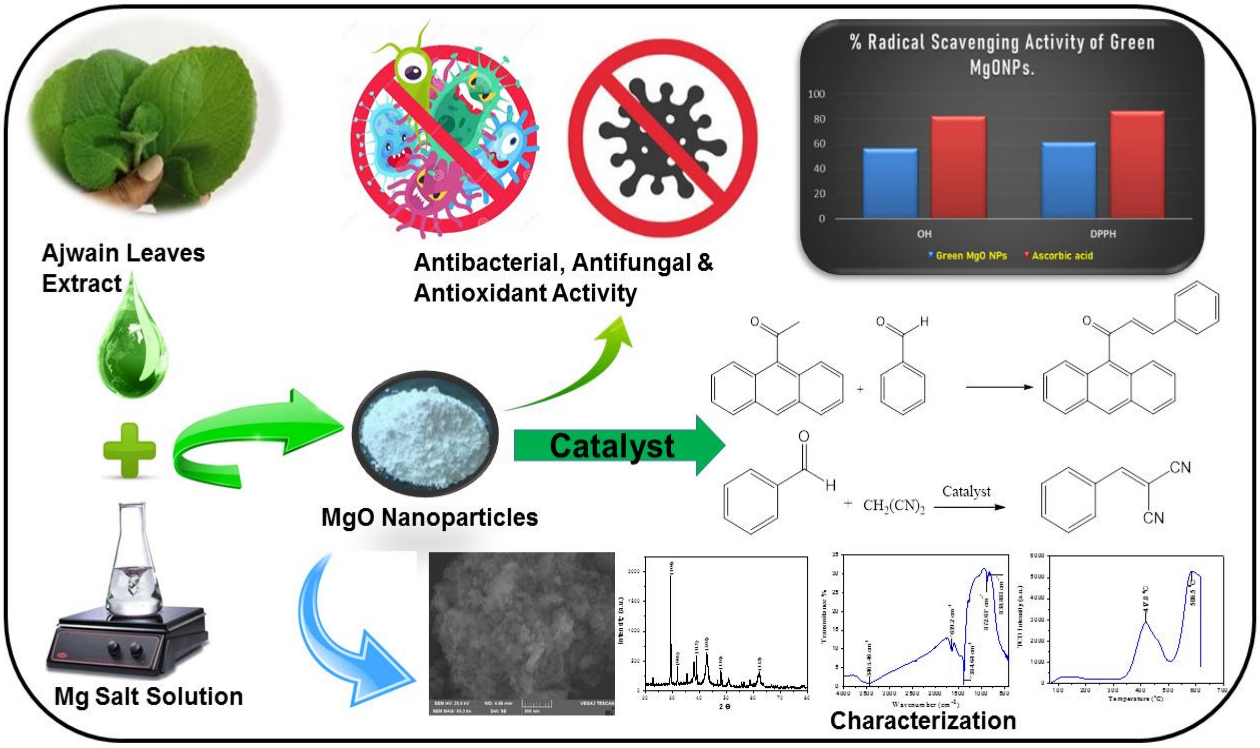
2.2.3.5.Green synthesis of MgO NPs:

In a typical process, the freshly prepared extract of Ajwain (Trachyspermum ammi) leaves was heated at 60 ◦C, and the pH was kept basic by the addition of 0.1 M NaOH. Next, the hot solution was added magnesium nitrate hexahydrate (1.48 g) slowly, and the resultant mixture was further heated at 60 ◦C for 15–20 min. Later, the reaction mixture was continuously stirred at 25 ◦C for 2 h. The solid particles were obtained, washed several times with DW followed by alcohol, and finally collected by centrifugation. The obtained solid was then dried in an oven at 70 ◦C and finally calcinated in a muffle furnace at 300 ◦C and used for further characterization and applications.

2.2.3.6.General procedure for the synthesis of chalcone by Claisen-Schmidt reaction

The 9-Acetylanthracene:

0.22 g (0.001 mol) and benzaldehyde 0.106 g (0.001 mol) were taken in a 50 ml round bottom (RB) flask, 10 ml of 50% ethanol was used as a solvent with 15 mg MgO NPs as a catalyst. The reaction mixture was stirred in an oil bath at 80 ◦C for 1 hr. The reaction progress was monitor by TLC method. After completing the reaction, reaction mixture was poured into ice-cold water to separate the product and catalyst. (Scheme 1)



Discussion:

The Ajwain plant facilitated production of MgO NPs was affirmed by UV–visible spectroscopy. The peak at 284 nm in confirms the formation of MgO NPs. Further, the band gap of eco-friendly synthesized MgO NPs was obtained by intercept line graphically, as shown in . It was found to be 3.9 eV. described the FT-IR spectrum of MgO NPs, the broadband at 3465.46 cm− 1 indicate the bending vibration of surface hydroxyl group, resulting in either alcoholic or phenolic, moisture adsorption stretching. In contrast, the peaks at 1639.2 cm^{-1} attributed to a C = C stretching in aromatic compounds and those at 1384.64 cm^{-1} corresponds to carbonyl stretch in biomolecules present in the plant extract. The stretching vibration appears at 872.63 cm^{-1}, and 838.88 cm^{-1}, indicating the Mg-O bonds. The FTIR results confirm the presence of the bio-molecules in leaf extract of Ajwain (Trachyspermum ammi), i.e. flavonoids (Luteolin-7-o-glucoside) and other biomolecules accountable for the capping and/or stabilization of MgO NPs [49]. The plant-mediated MgO NPs were tested against fluorescence studies and exhibited visible photoluminescence.

Green synthesis of magnesium oxide nanoparticles (MgO NPs) is an eco-friendly, sustainable, and cost-effective method that has garnered significant interest due to its potential to reduce environmental and health hazards associated with conventional chemical and physical synthesis methods.

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Chapter-III
Charecterisation Studies Of Nanoparticles

3.3.1.Introduction:

Nanotechnology is one of the treasured and superb disciplines that serve the top-down and bottom-up approach, which contains physical, chemical, and biological (plant, microorganisms, and biomaterials) methods for synthesizing multifunctional nanomaterials. Nanoparticles (NPs) are a multifaceted class of materials that include particulate materials having dimensions 1–100 nm. Those mentioned above physical and chemical techniques are initial time eating, which utilizes excessive energy, the requirement of reducing agents. Also, they are not eco-friendly, which makes them want to discover and innovate methods that conquer the shortcomings of these chemical and physical methods.As evaluated with physical and chemical methods, the biological methods for synthesizing NPs seem pleasant due to their fascinating packages and fewer requirements. It is quick, nontoxic, economical, energy-efficient, and follows the standards of principles of green chemistry .Plenty of research was done on synthesizing metal oxide NPs supported by other materials like g-C3N4, which enhances nanomaterial's photocatalytic and biological activity. There are numerous reports on the eco-benign production of MgO NPs, and the literature survey suggests that extract of diverse plants and their different parts have been used for the phytogenic fabrication of MgO NPs. The MgO NPs possess excellent catalytic activities and are utilized for numerous organic reactions, as evident by literature.The exploitation of MgO NPs as a catalyst for various organic transformations is because of their non-toxic nature, high basicity, ready availability, low cost, and reproducibility. However, besides numerous applications of MgO NPs as a catalyst for organic transformations, the use of MgO NPs synthesized from the plant for organic reactions is not explored to its capacity. Nonetheless, few reports on using plant-mediated MgO NPs for organic reactions. Ajwain (Trachyspermum ammi) of the family Apiaceae, is an essential therapeutic, spice and aromatic plant. As displayed, Trachyspermum ammi extract contains diverse active bio-compounds, namely thymol, limonene, myrcene, 1′ -(3-hydroxy-4,5-dimethoxy-phenyl)propane-2′ -3′ -diol, Luteolin-7-o-glucoside, and carvacrol. Diverse therapeutic uses were noted in the literature for this plant. Further, Trachyspermum ammi is also used for pharmacological and biological activities such as antidiarrhoeal, antifungal, insecticidal, antibacterial. Active phytochemical constituents in Ajwain. Scheme 1. Model Claisen-Schmidt reaction of 9-acetylanthracene with benzaldehyde. Scheme 2. Knoevenagel reaction of an aldehyde with malononitrile. antihypertensive, anthelmintic, antispasmodic, nematicidal, antiinflammatory, anti-lithiasis, anti-nociceptive, antiplatelet-aggregatory, antifilarial, abortifacient, antitussive, enzyme modulation, antihyperlipidemic, antioxidant,

antiepileptic, and analgesic activity. Considering the catalytic potential of plant-mediated MgO NPs, here we first report the green fabrication of MgO NPs employing leaves broth of Ajwain (Trachyspermum Ammi) and their use as a catalyst for ClaisenSchmidt and Knoevenagel reactions. The MgO NPs synthesized using the developed protocol were found to show excellent catalytic activities and could be reused up to five times without considerable loss in its catalytic effectiveness for both reactions. In addition, the antimicrobial and antioxidant potential of MgO NPs were also tested, which shows moderate to considerable efficacies compared to standards.

3.3.2.Present Work:

Characterization is critical to ensure the synthesized nanoparticles meet the desired application requirements. The selection of techniques depends on the type of nanoparticles and their intended use. For example, biomedical applications may emphasize biocompatibility and surface functionalization, while environmental applications may focus on stability and catalytic activity. Characterization of synthesized nanoparticles (NPs) is essential to understand their physical, chemical, and functional properties. This process involves various techniques to determine size, shape, crystallinity, surface properties, and more.

The synthesized MgO NPs were explored using diverse spectroscopic and microscopic techniques such as UV spectroscopy by JASCO V-770 spectrophotometer, XRD data was recorded using Model-D8 AdvanceBruker, IR spectra were recorded using FT/IR-4600 type A, and PL spectra using FP-8200

instrument. The topology of synthesized MgO was analyzed by SEM using JSM-6380, and basicity was studied by CO2-TPD by BELCAT II Version 0.5.1.10. Below is an overview of key characterization techniques used for nanoparticles.

3.3.3. Charecterisation Of Nanoparticles:

3.3.3.1. UV-DRS (Ultraviolet Diffuse Reflectance Spectroscopy):

The Ajwain plant facilitated production of MgO NPs was affirmed by UV–visible spectroscopy. The peak at 284 nm in Fig. 2a confirms the formation of MgO NPs. Further, the band gap of eco-friendly synthesized MgO NPs was obtained by intercept line graphically, as shown in Fig. 2b. It was found to be 3.9 eV.

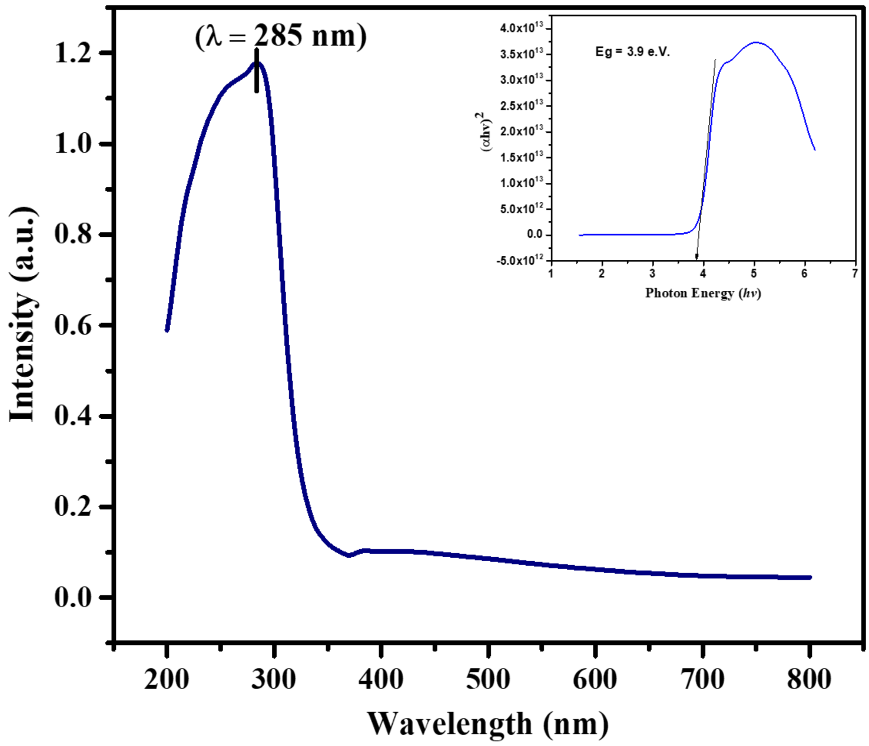


Fig. 1. (a) UVDRSspectra Green-MgO NPs(b) Band gap of Green-MgO NPs.

3.3.3.2. IR-Spectra Infrared Spectroscopy:

Infrared (IR) spectroscopy is a widely used analytical technique to identify and characterize molecular structures by analyzing their interaction with infrared light. It provides valuable information about the functional groups, molecular bonding, and chemical composition of a sample.

FT-IR spectrum of MgO NPs, the broadband at 3465.46 cm− 1 indicate the bending vibration of surface hydroxyl group, resulting in either alcoholic or phenolic, moisture adsorption stretching. In contrast, the peaks at 1639.2 cm− 1 attributed to a C = C stretching in aromatic compounds and those at 1384.64 cm− 1

corresponds to carbonyl stretch in biomolecules present in the plant extract. The stretching vibration appears at 872.63 cm− 1 , and 838.88 cm− 1 , indicating the Mg-O bonds. The FTIR results confirm the presence of the bio-molecules in leaf extract of Ajwain (Trachyspermum ammi), i.e. flavonoids (Luteolin-7-o-glucoside) and other biomolecules accountable for the capping and/or stabilization of MgO NP's.

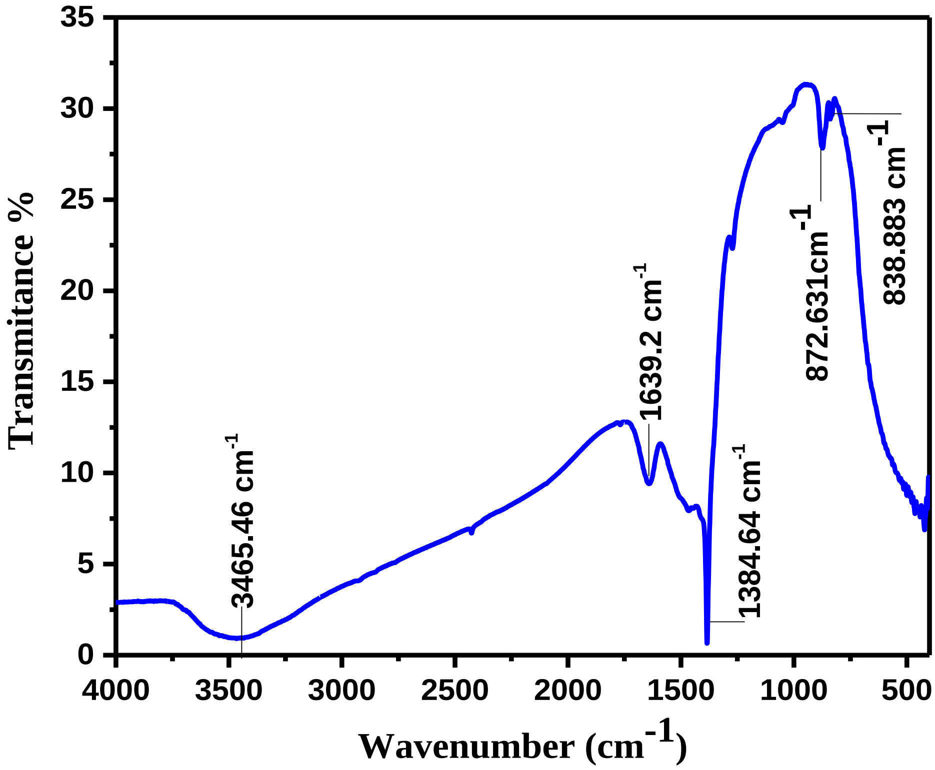


Fig. 3. IRspectra of Green-MgO NPs.

3.3.3.4.Photoluminescence Spectra:

Photoluminescence (PL) spectroscopy is a powerful optical technique used to study the electronic and optical properties of materials, including nanoparticles (NPs). It provides information about energy band structures, defect states, and recombination processes of charge carriers.

The plant-mediated MgO NPs were tested against fluorescence studies and exhibited visible photoluminescence. The fluorescence spectrum is shown in Fig. 4. The enhanced MgO NPs were found to emit two emissions at 283.1 nm and 565.5 nm for excitation at 250 nm.

The band gap of biosynthesized MgO NPs was calculated using equation,

$$E = hc/\lambda,$$

where λ is the highest wavelength taken in fluorescence spectra.

The luminescence observed may be due to the availability of phytochemicals or antioxidants in the plant extract.

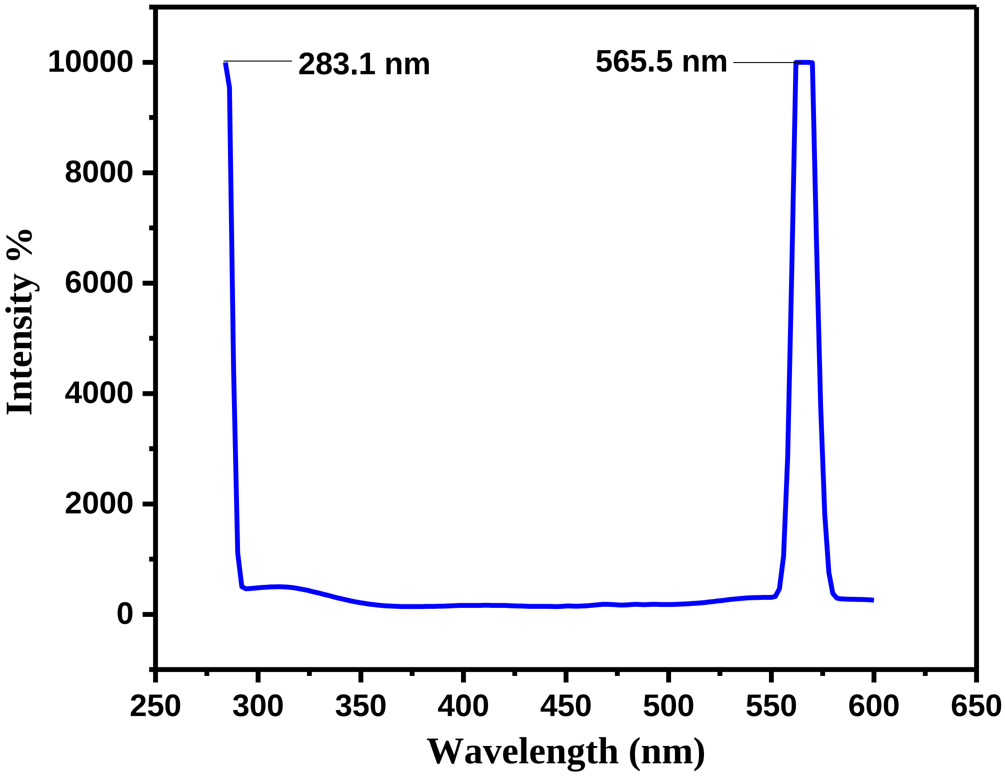
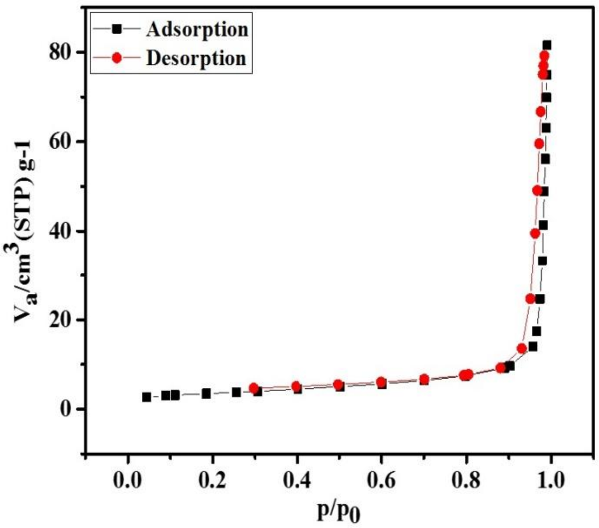


Fig. 4. Photoluminescence spectra of Green-MgO NPs.

3.3.3.5. BET Plot Of NP's:



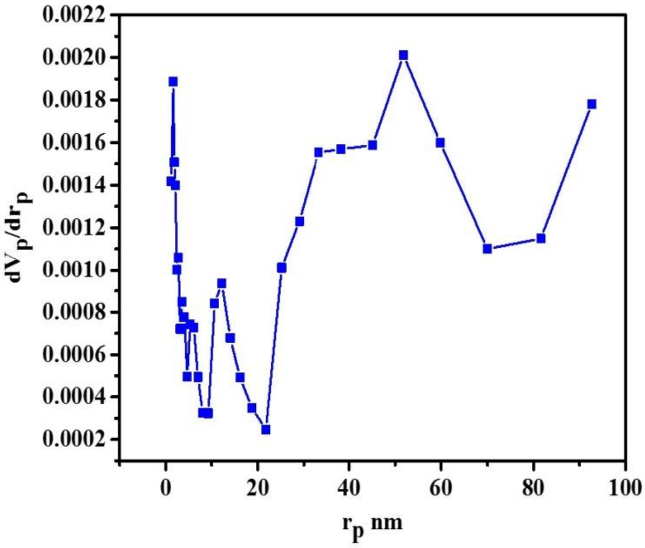


Fig 5. (a)BET plot of Green-MgO NPs, (b)BJH plot of Green-MgO NPs

Fig. 5a represents the BET plot of MgO NPs, the surface area of MgO NPs measured applying the multipoint BET equation and was found at 12.411 m2 /g. In addition, Fig. 5b presents the typical BJH desorption pore sizes distribution curve of MgO NPs. The pour size obtained from peak position was about 51.77 nm, and pore volume was found 0.1201 cm3 /g, specifying the moderately small pore size. The BET and BJH Fig. 7. FE-SEM of Green-MgO NPs. Fig. 8. EDX of green synthesized NPs.

3.3.3.6. X-ray Diffraction (XRD):

X-ray diffraction (XRD) is a powerful analytical technique used to determine the crystalline structure of materials. It involves directing X-rays at a sample and measuring the angles and intensities of the diffracted beams. This data provides insights into the crystal lattice, phase identification, and other structural properties.

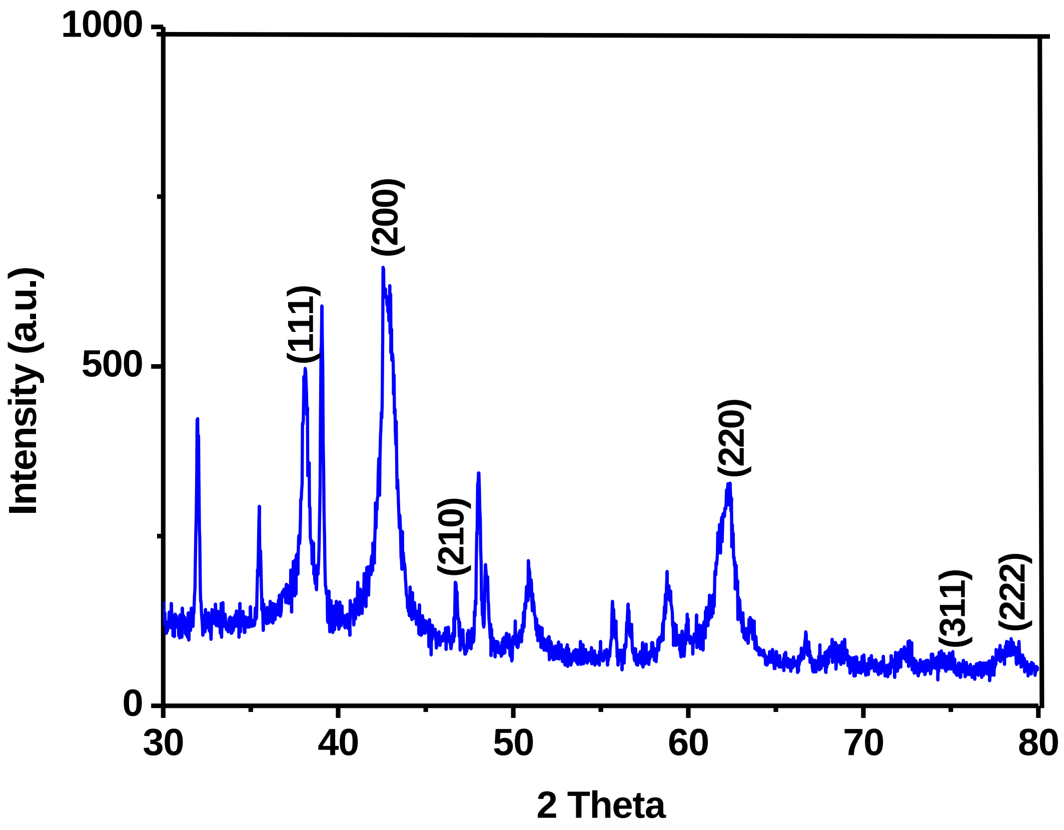


Fig. 6.

XRD profile of Green-MgO NPs.

Fig. 6 displays the XRD pattern of resulting MgO NPs. The XRD profile of MgO NPs synthesized in this study shows six distinct diffraction peaks corresponding to (111), (200), (210), (220), (311), and (222), which confirms hexagonal structure (JCPDS file: 00–004-0823). The XRD pattern reveals that synthesized MgO NPs are polycrystalline, and data were matched previous works [25–26]. In addition, the mean size of MgO NPs was determined from XRD data, which was found to be 83.24 nm.The crystalline size of MgO NPs was ascertained according to Scherrer's equation(1)

$$D = K\lambda / \beta\cos\theta \ldots\ldots\ldots\ldots\ldots\ (1)$$

Where,

D = Crystalline size
K = 0.9 (Scherrer's constant)
λ = 0.15406 nm (wavelength of X-ray source)
β = FWHM (radians)
θ = Peak position (radians)

3.3.3.6.FE-SEM Field Emission Scanning Electron Microscopy:

Field Emission Scanning Electron Microscopy (FE-SEM) is a powerful imaging technique that provides high-resolution images of the surface structure of materials. It is an advanced form of Scanning Electron Microscopy (SEM), using a field emission electron gun (FEG) instead of a conventional thermionic electron source.

Fig. 7 shows representative SEM images that confirm the agglomeration of MgO NPs. The average particle size of MgO NPs revealed by SEM analysis was 78.48 nm. Further, the EDX profile is additional information to affirm the production of MgO NP's.

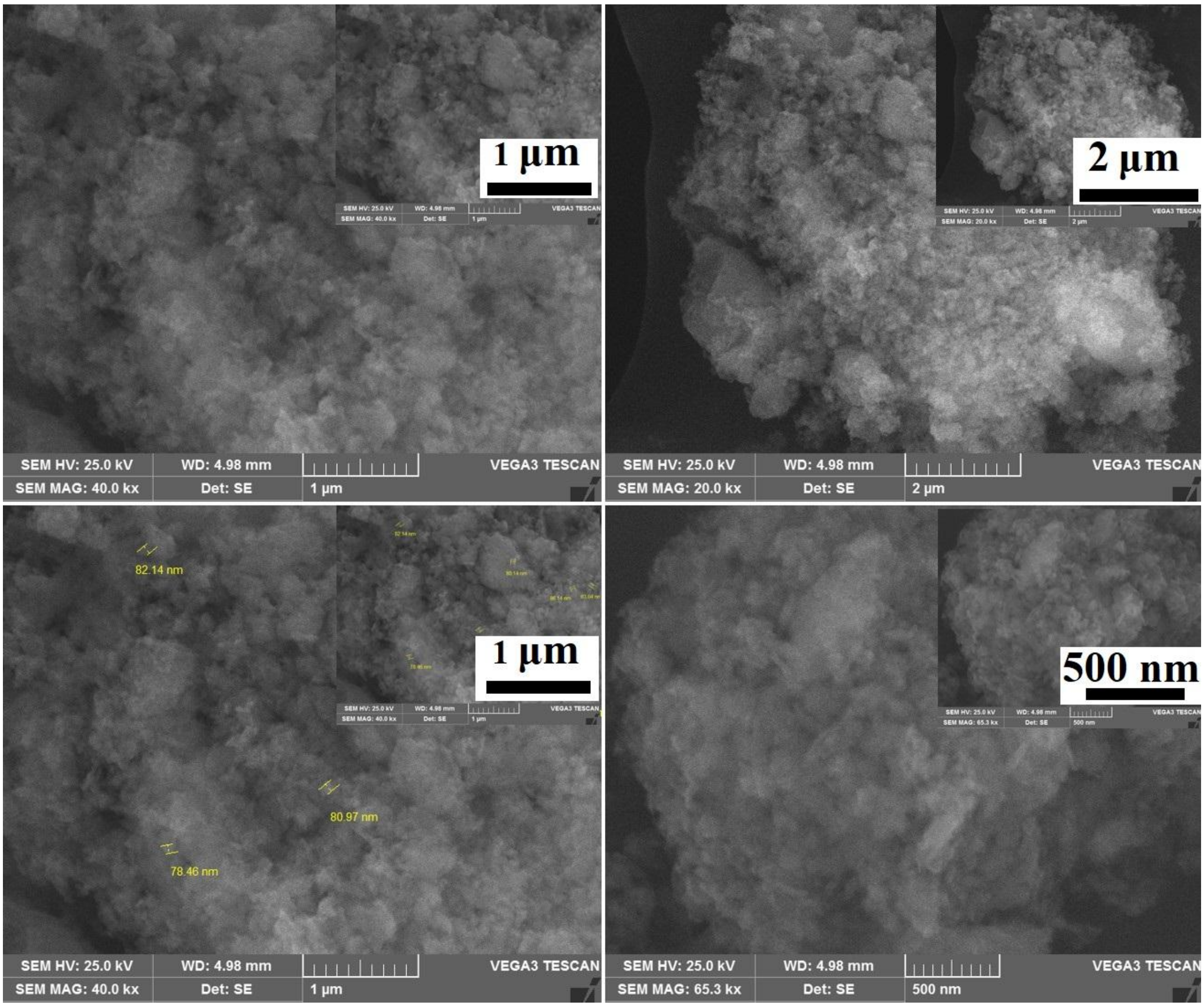


Fig. 7.FE-SEM of Green-MgO NPs.

3.3.3.8.EDX (Energy Dispersive X-ray Spectroscopy):

Energy Dispersive X-ray Spectroscopy (EDX or EDS) is an analytical technique used to identify the elemental composition of a sample. It is commonly coupled with Scanning Electron Microscopy (SEM) or Transmission Electron Microscopy (TEM), allowing for both imaging and elemental analysis in a single experiment. EDX detects characteristic X-rays emitted from a sample when it is bombarded with an electron beam, providing information about the elements present and their relative quantities.

EDX is a versatile and essential tool for elemental analysis, providing valuable information about the composition and distribution of elements in materials. It is widely used in research, industrial applications, and quality control processes across a range of fields, from materials science to geology to biology.

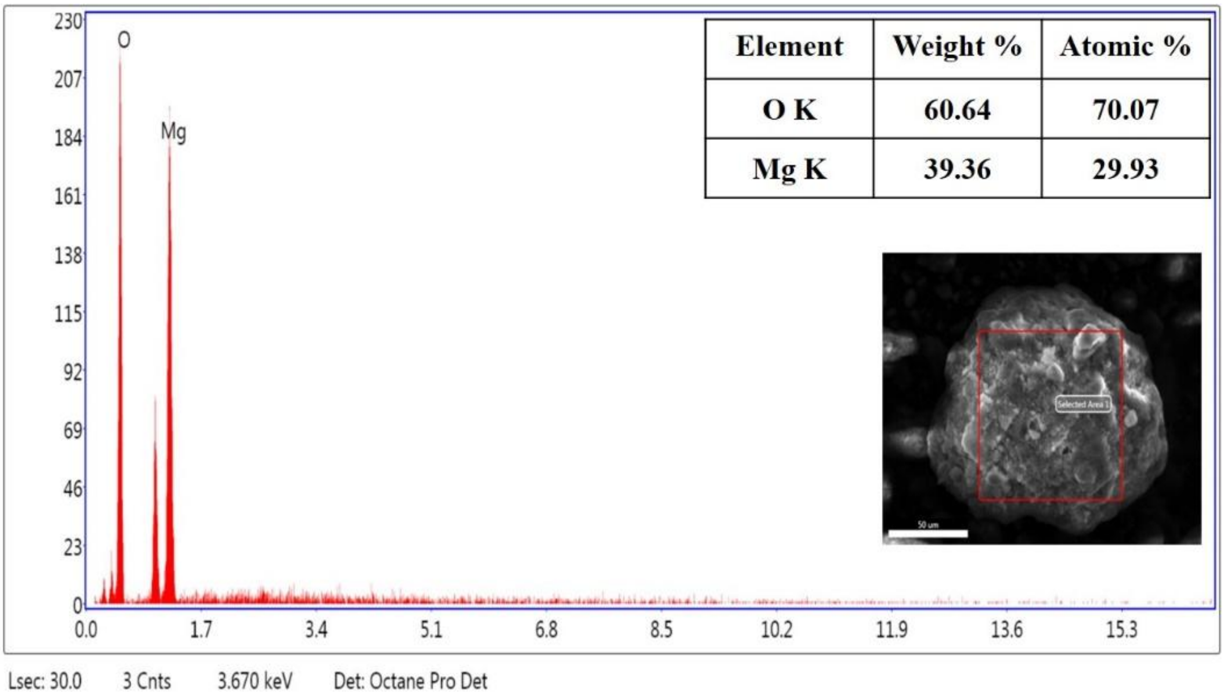


Fig. 8. EDS of green synthesized MgONPs.

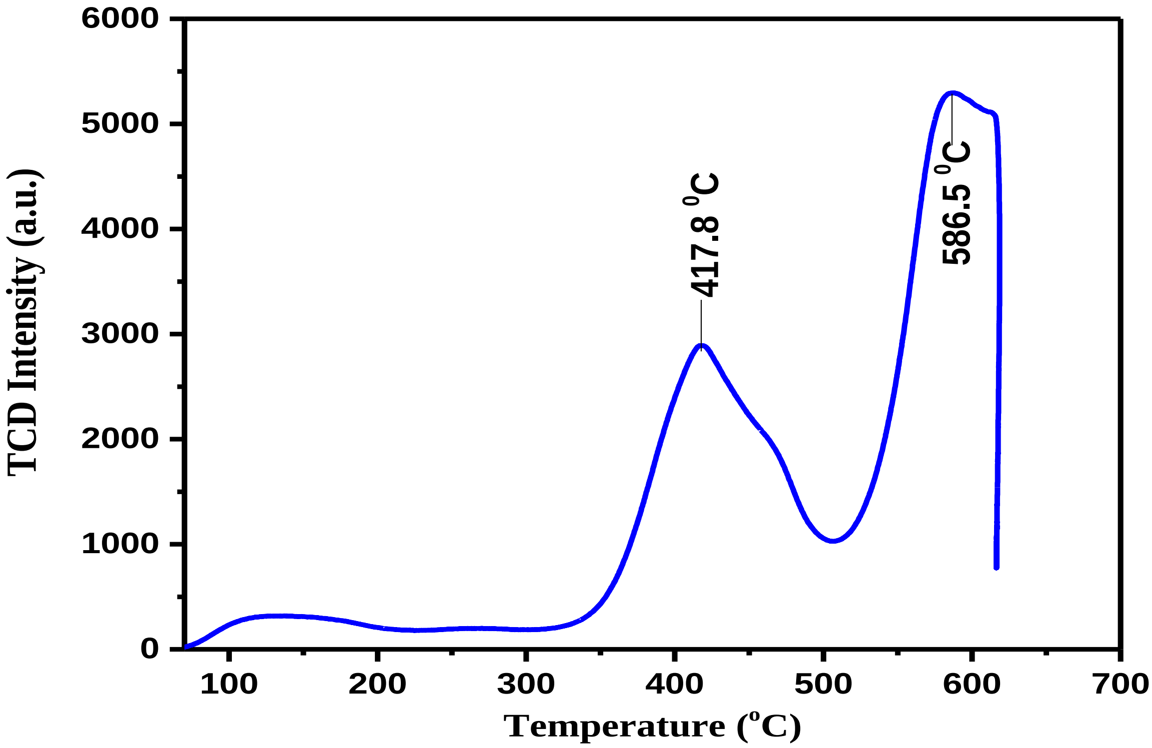


Fig. 9. CO_2-TPD plot of Green-MgO NPs.

3.3.3.8.CO2-TPD (CO_2 Temperature-Programmed Desorption):

CO_2 Temperature-Programmed Desorption (CO_2-TPD) is an analytical technique used to characterize the surface properties of materials, particularly their acidity or basicity. It involves exposing a sample to CO_2 gas at varying temperatures and then analyzing the desorption behavior of CO_2. The data obtained provides valuable insights into the surface chemistry of the material, such as its acid-base properties, the strength of surface sites, and their distribution.

3.3.4.Discussion:

The Ajwain plant facilitated production of MgO NPs was affirmed by UV–visible spectroscopy. The peak at 284 nm in Fig. 2a confirms the formation of MgO NPs. Further, the band gap of eco-friendly synthesized MgO NPs was obtained by intercept line graphically, as shown in Fig. 2b. It was found to be 3.9 eV. Fig. 3 described the FT-IR spectrum of MgO NPs, the broadband at 3465.46 cm− 1 indicate the bending vibration of surface hydroxyl group, resulting in either alcoholic or phenolic, moisture adsorption stretching. In contrast, the peaks at 1639.2 cm− 1 attributed to a C = C stretching in aromatic compounds and those at 1384.64 cm− 1 corresponds to carbonyl stretch in biomolecules present in the plant extract. The stretching vibration appears at 872.63 cm− 1 , and 838.88 cm− 1 , indicating the Mg-O bonds. The FTIR results confirm the presence of the bio-molecules in leaf extract of Ajwain (Trachyspermum ammi), i.e. flavonoids (Luteolin-7-o-glucoside) and other biomolecules accountable for the capping and/or stabilization of MgO NPs . The plant-mediated MgO NPs were tested against fluorescence studies and exhibited visible photoluminescence. The fluorescence spectrum is shown in Fig. 4. The enhanced MgO NPs were found to emit two emissions

at 283.1 nm and 565.5 nm for excitation at 250 nm. The band gap of biosynthesized MgO NPs was calculated using equation $E = hc/\lambda$, where λ is the highest wavelength taken in fluorescence spectra, and it was found to be 2.19 eV. The luminescence observed may be due to the availability of phytochemicals or antioxidants in the plant extract. The catalytic properties of catalysts depend on the surface area and its porosity, large surface area, enhances the catalytic properties of MgO NPs. Fig. 5a represents the BET plot of MgO NPs, the surface area of MgO NPs measured applying the multipoint BET equation and was found at 12.411 m2 /g. In addition, Fig. 5b presents the typical BJH desorption pore sizes distribution curve of MgO NPs. The pour size obtained from peak position was about 51.77 nm, and pore volume was found 0.1201 cm3 /g, specifying the moderately small pore size. The BET and BJH curves conclude that most microspores have a size smaller than 51.77 nm. Fig. 6 displays the XRD pattern of resulting MgO NPs. The XRD profile of MgO NPs synthesized in this study shows six distinct diffraction peaks corresponding to (111), (200), (210), (220), (311), and (222), which confirms hexagonal structure (JCPDS file: 00–004-0823). The XRD pattern reveals that synthesized MgO NPs are polycrystalline, and data were matched previous works. hows representative SEM images that confirm the agglomeration of MgO NPs. The average particle size of MgO NPs revealed by SEM analysis was 78.48 nm. Further, the EDX profile is additional information to affirm the production of MgO NPs. In Fig. 8, the peaks of O and Mg elements in between 0.5 and 1.5 KeV suggest the presence of MgO NPs. The elemental analysis reveals that the percentage of Mg and O element in the material is 39.36 and 60.64 %, respectively. The basicity of green synthesized MgO NPs using leaf extract of Ajwain (Trachyspermum ammi) was studied by CO2-TPD technique. In the beginning, the material was pre-treated with helium gas from 24 ◦C to 400 ◦C for one hour to remove the absorbed moisture and impurities. Then, this material was used for TPD analysis after cooling to room temperature and saturation CO2 at 50 ◦C. Finally, the TPD analysis was carried out from 24 ◦C to 600 ◦C at a temperature range of 10 ◦C/min employing helium as inert gas at a 20 cm3 /min flow rate. As a result, the amount of CO2 desorbed from MgO NPs was 420 µmol/g and 586 µmol/ g, which shows that MgO NPs are more basic in nature .

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Chapter-IV

Applications Of Nanoparticles

4.1.Introduction:

In recent years, metal oxide NPs are notable for their magnificent and eclectic applications in the disciplines of bio-technology, catalysis, medicine, bio-engineering, agriculture, textile engineering and water treatment . The NPs addition-nally have huge significant applications in other areas such as photocatalysis, biosensors, cancer therapeutics, labeling for cells, cosmetics, magnetics, solar cells, optoelect-ronics and space industry . The synthesis of metal oxide NPs and their multifarious applications in these areas are of great and imperative interest for further study. The preparation of size and shape selective nanosize NPs has generated an innumerable enthusiasm due to their distinctive structures, size, and their peculiar chemical, physical, as well as biological features/properties compared with their bulk compounds. Magnesium and its compounds are important materials through-hout the history, finding multifarious remarkable applications (Figure 1). MgO NPs are used for the production of industrially valuable compounds in pharmaceuticals, agro-chemicals and their profitable intermediates . Several researchers have developed novel protocols for the rapid synthesis of MgO NPs along with their applications in various fields such as photocatalyst, sensors, electronic devices, drug delivery, agriculture and catalysis. Moreover, MgO NPs also showed the biological properties such as antibacterial, antifungal, cytotoxic, antioxidant and anticancer activity.

Present review article covers the procurable synthesis of MgO NPs by chemical, physical and biological approaches along with their recent diverse characterization techniques followed by a discussion on MgO nano catalysis. MgO NPs play an overriding role as a sustainable catalyst due to their distinctive features/properties for several organic transformations which are extensively discussed here.

4.2.Present Work:

Size and shape selective syntheses of nanoparticles (NPs) and their varied catalytic applications are gaining significant enthusiasm in the field for nanochemistry. Homogeneous catalysis is crucial due to its inherent benefits like high selectivity and mild reaction conditions. Nevertheless, it endures with serious disadvantages of catalysts and/or product separation/recycles as compared to their heterogeneous counterparts restricting their catalytic applications. The utilization of catalysts in the form of nano-size is an elective methodology for combinations of merits of homogeneous and heterogeneous catalysis. Magnesium oxide (MgO) NPs are important as they find applications for catalysis, organic transformations, synthesis of fine chemicals and organic intermediates. The applications of MgO NPs in diverse organic transformations including oxidation, reduction,

epoxidation, condensation and C-C, C-N, C-O, C-S bond formation in variety of notable heterocyclic reactions are also discussed. The use of MgO NPs in organic transformation is advantageous as it mitigates the use of ligands; procurable separation of catalyst for recyclability makes the protocol heterogeneous and monetary. MgO NPs gave efficacious catalytic performance towards desired products due to high surface area. By considering these efficient merits, scientists have focused their attentions towards stupendous applications of MgO NPs in selective organic transformation. In current review article, we summarized the synthesis of MgO NPs and numerous characterization techniques, whereas the application section illustrates their utility as a catalyst in several organic transformations. We believe this decisive appraisal will provide imperative details to further advance the applications of MgO NPs in the selective catalysis.

This article discuss the applications of MgO NPs as catalysts for diverse reactions, including oxidation, reduction, epoxidation, condensation and notable heterocyclic reactions (Figure 2). The synthetic strategies and relevant examples of MgO NPs are discussed with brief descriptions of relevant characterization techniques such as UV–visible spectroscopy, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Energy Dispersive Spectroscopy (EDS), Fourier Transform Infrared (FTIR) Spectroscopy, Atomic Absorption Spectroscopy (AAS), Dynamic Light Scattering (DLS), Electrophoretic Light Scattering (ELS), Atomic Force Microscopy (AFM), Scanning Tunneling Microscopy (STM) and X-ray Photoelectron Spectroscopy (XPS). We believe that a detailed overview of the current protocols for preparing MgO NPs and their multifarious applications would be encouraging to a broad community of scientists working in the disciplines of chemical engineering, inorganic chemistry, material chemistry, nanotechnology and organic chemistry. Moreover, most of the chemistries detailed in this review, namely, various reactions to benign media, such as water, ethanol and recyclability of nano catalyst will address current outstanding developments in the field of nanochemistry.

4.3.Application Of Nanoparticles:

The investigation into NPs catalyzed reactions is projected in modern research because NPs play an important role in several organic transformations *via* heterogeneous catalysis. Nanomaterials showed good catalytic performance and selectivity due to their topography, size, and high surface area to volume ratio [1]. The NPs have diverse merits in catalysis such as NPs are insoluble in the reaction medium, mild reaction conditions, easy workup, excellent yield, easy separation and recyclability of catalyst. Strikingly, the organic transformations using NPs do not need any ligand source, they accelerate the reaction using low catalyst loading with low temperature, which makes the protocol simple, one pot, affordable and cost effective.

MgO NPs has been used as catalysts for a wide variety of organic transformations. They are especially appealing to this goal because they often enable organic reactions to be conducted under

durable or green reaction conditions that would mitigate the performance of conventional catalysts. This section describes the MgO NPs as catalyst for various organic transformations like oxidation, reduction, epoxidation, condensation and C-C, C-N, C-O, C-S bond formation in variety of notable heterocyclic reactions.

4.3.1. Claisen-Schimidt Condensation

4.3.2. Knoevenagel reaction

4.3.3. Synthesis of Heterocyclic Compounds

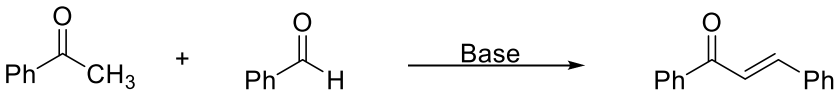
a. Synthesis of Chomenes.
b. Synthesis of Coumarin
c. Synthesis of Pyridine
d. Synthesis of Pyridines
e. Synthesis of Imidazole
f. Synthesis of Thiazoles
g. Synthesis of Pyrazoles
h. Synthesis of Isoxazol
i. Synthesis of Amide

4.3.4.Other Reactions

Fig 1. Applications of MgO NPs in organic reactions

4.3.1. Claisen-Schimidt Condensation:

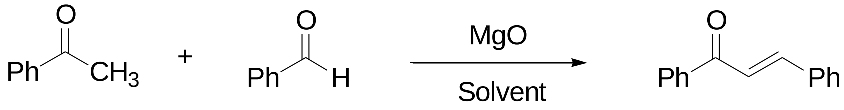
Claisen-Schmidt's reaction is one of the old yet very important reaction utilizing carbonyl group containing starting materials and yields chalcone as a product. Specifically, the reaction between an aldehyde and ketone having α-hydrogen with an aromatic carbonyl compound without α-hydrogrn results in formation of chalcone shown in (Scheme 1). The chalcones are having several applications such as antioxidative, antibacterial, antihelmintic, amoebicidal, antiulcer, antiviral, insecticidal, antiprotozoal, anticancer, cytotoxic and immunosuppressive. The activities exhibited by chalcones are dependent upon the substituents present over it.



Scheme 1. Claisen-Schmidt Reaction.

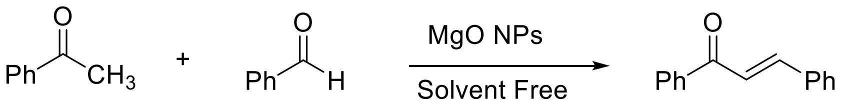
Chalcone is the precursor of compounds for flavonoids biosynthesis in plants and drug discovery . These natural products and synthetic compounds have shown numerous interesting biological activities with clinical potentials against several diseases.

synthesized MgO NPs by reverse micellar method which gave MgO NPs with particle size 8-10 nm [67]. The characterization of MgO NPs was done with the help of HR-TEM, SEM, XRD, and FTIR to conclude the formation of MgO NPs. The synthesized MgO NPs were used in Claisen- Schmidt reaction (Scheme-2).



Scheme 2. Claisen- Schmidt reaction using MgO NPs.

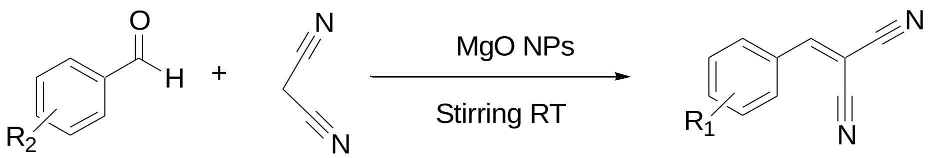
MgO nanocrystals was synthesized by novel and green methods by Patil et al. MgO NPs were prepared from a mixture of magnesium acetate and 1,4-butanediol keeping in sunlight. The synthesized MgO NPs was confirmed by spectroscopic and microscopic techniques. The MgO NPs was use for the syntheses of chalcone in which acetophenone and benzaldehyde derivatives reacted in presence of 10 % (weight) MgO NPs at 140 °C for 4 hrs under solvent free condition to yield the desired products in good to excellent amount (Scheme-3)



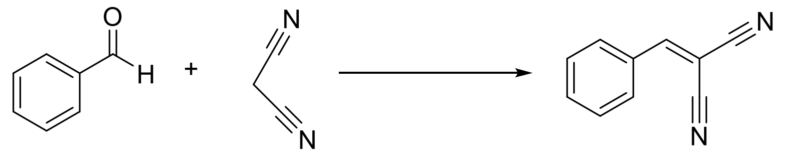
Scheme 3. Solvent-free Claisen- Schmidt reaction using MgO NPs.

4.3.2. Knoevenagel Reaction:

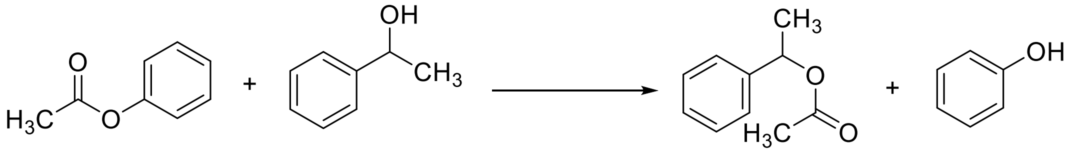
The reaction of the compound having active methylene group with substituted aromatic aldehyde generally referred to as Knoevenagel reaction, the resultant product can be used for the synthesis of novel heterocyclic molecules. This reaction is reported to be catalyzed by using several bases ranging from strongly basic to the bases having weak basicity. The MgO NPs having strong basic properties as a result it was also explored for Knoevenagel reaction as a catalyst. In this context, Roy *et al.* explored this reaction by condensing malononitrile with an aromatic aldehyde in 50% ethanol and MgO NPs as a heterogeneous recyclable catalyst at RT (scheme-8).



The reaction was carried out at room temperature, the time reported for different aldehyde ranges from 25 to 90 mins providing more than 85 % yield of desired products. The solvent effect was also studied and it was found that in ethanol it gives 99 % yield. The study of effect of amount of catalyst was also explored and the protocol was used to synthesize library of products**.** Vidruk *et al.* synthesized MgO-aerogel by sol-gel method using $Mg(OEt)_2$, MeOH and toluene. Characterization was done with electronic microscopic techniques such as SEM-EDS, TEM, XRD, XPS and basicity was obtained by titration method and CO_2-TPD technique as well. The synthesize MgO-aerogel was subjected to following reactions as depicted in Scheme 9 and 10.



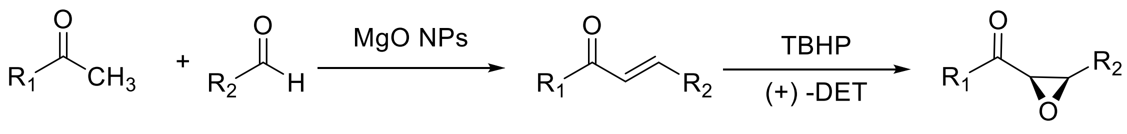
Scheme 9. Knoevenagel condensation reaction carried out in the toluene and N_2 atmosphere.



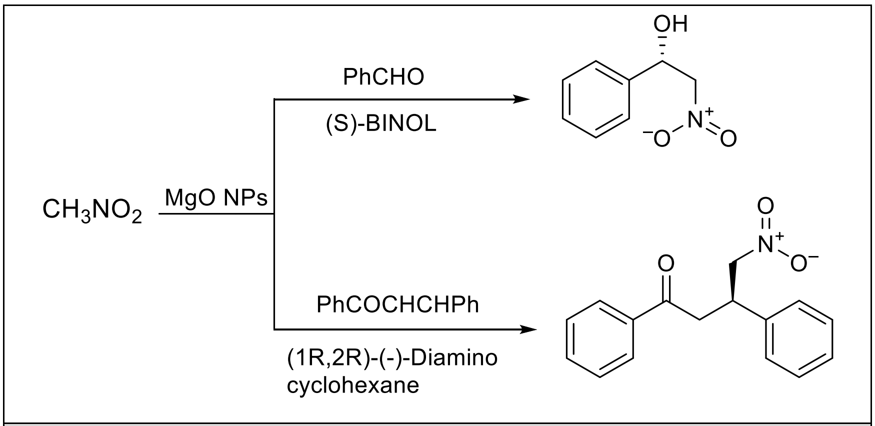
Scheme 10. Trans-esterification of phenyl acetate with 1-phenyl ethanol carried out in toluene and N_2 atmosphere.

The effect of densification of magnesia aerogel precursor on the surface basicity of nanostructured MgO material was tested by the authors. The densified nanostructured MgO material showed higher basic sites as compare to undensified magnesium aerogels. The synthesized material was used for the organic transformations as mentioned above. The rate of reactions was found to be multiple fold greater when densified nanostructured MgO material was used as base catalyst.

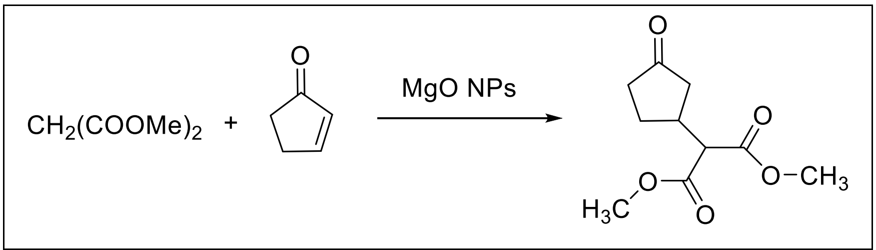
Nanocrystalline MgO also reported in the asymmetric synthesis by Roya *et al.* They discussed the applications of MgO NPs in C-C bond formation reactions such as Claisen-Schmidt reaction followed by asymmetric epoxidation (Scheme-11a), Henry (Scheme-11b) and Michael Reactions (Scheme-11c) [74]. The catalyst was found to provide good to excellent yields of desired products in all the reported transformations.



Scheme 11a. Claisen-Schmidt reaction catalysed by MgO NPs.

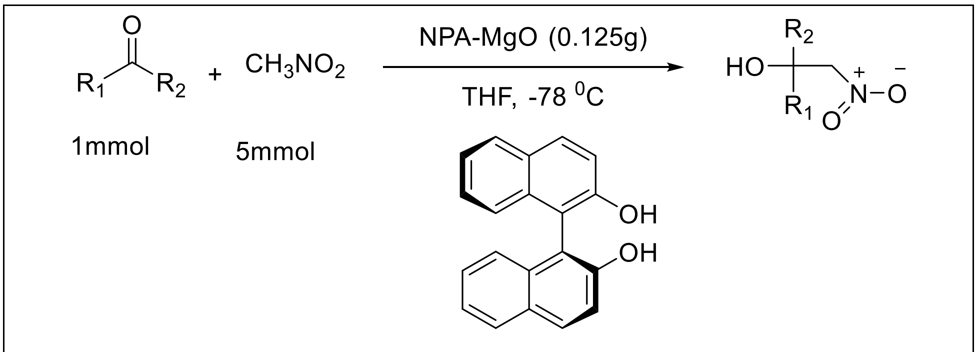


Scheme 11b. Henry reaction catalysed by MgO NPs.

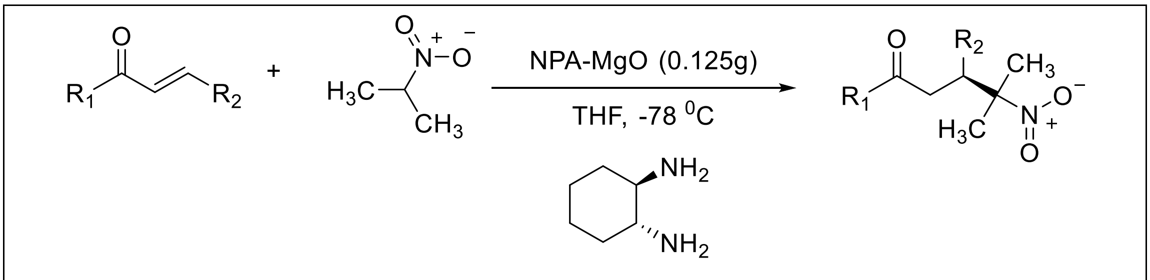


Scheme 11c. Michael reaction catalysed by MgO NPs.

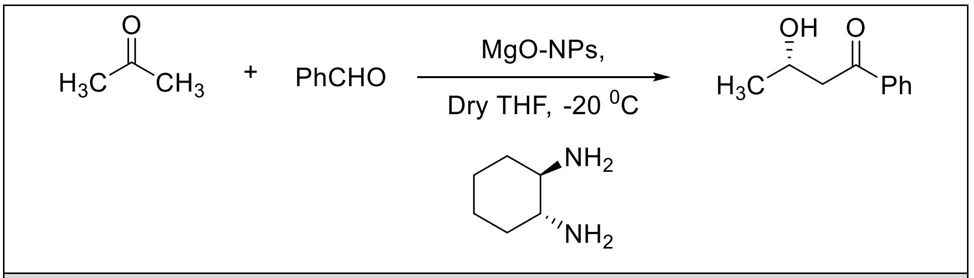
Similarly, Kobayashi *et al.* reported the Henry (Scheme-12), asymmetric Aldol (Scheme-14), asymmetric Michael reaction (Scheme-13 and Scheme-15) by using



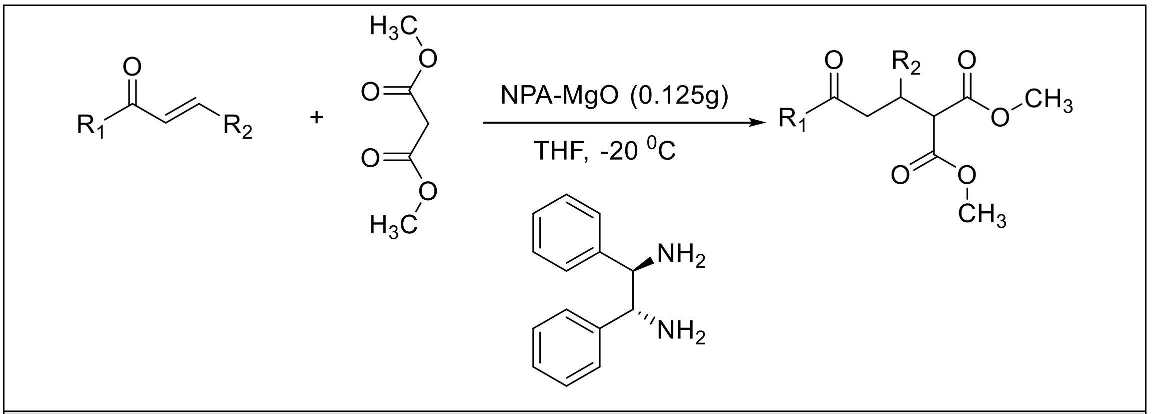
Scheme 12. NAP–MgO catalyzed asymmetric Henry reactions.



Scheme 13. NAP–MgO-catalyzed asymmetric Michael reactions with nitro alkanes.



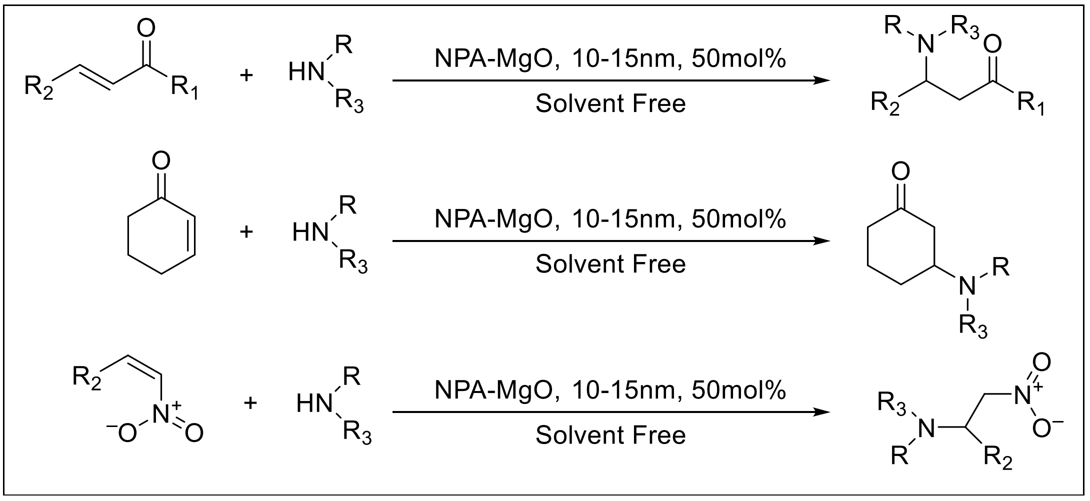
Scheme 14. NAP–MgO-catalyzed asymmetric aldol reactions.



Scheme 15. NAP–MgO-catalyzed asymmetric Michael reactions with malonates.

as heterogeneous reusable catalyst.

They studied the effect of various catalysts on the outcome of the reaction, where the highest yield of product was obtained with MgO NPs. The protocol was used to prepare variety of products with the aid of different unsaturated compounds and amines at room temperature (Scheme-19).



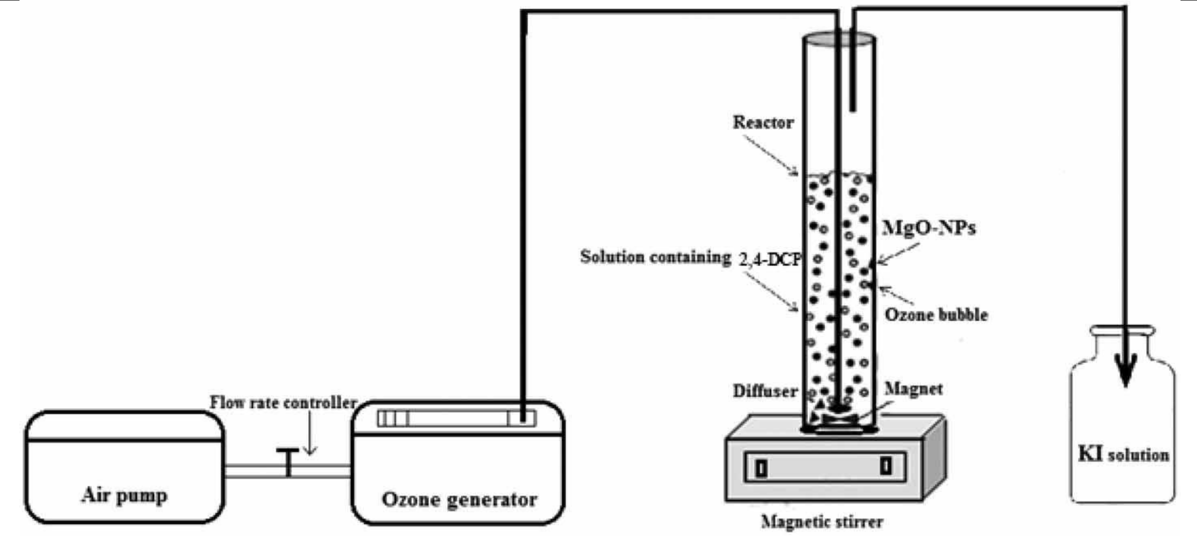


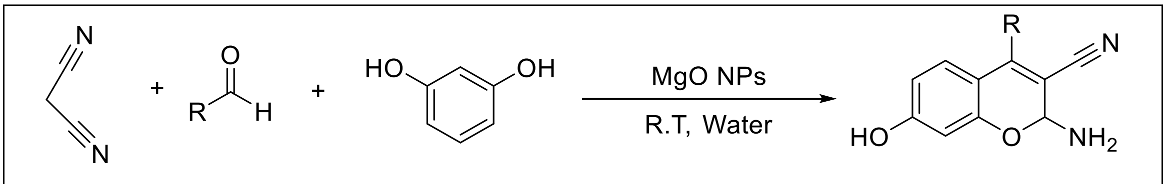
Figure 2. Schematic of the used catalytic ozonation reactor (Reproduced from ref. 80)

4.3.3. Synthesis of Heterocyclic Compounds:

Heterocyclic compounds play a key role in many fields of chemistry like organic synthesis, pharmaceuticals, agriculture, dyes, pigments, etc. everywhere heterocyclic compounds are present and the synthesis of such biologically active heterocyclic compounds containing different hetero atom is a challenge towards researchers. The synthesis of various active heterocyclic compounds such as a pyrazole, imidazole, thiazole, pyridine, chromene, coumarin and many more were reported by using MgO NPs as a catalyst, in this section light is thrown on such organic transformations.

.Synthesis of Chromenes :

Benzopyran is a polycyclic organic compound that results from the fusion of a benzene ring to a heterocyclic pyran ring referred to as chromene and they are recognized as antimicrobial, antiviral, mutagenic, antiproliferative, antitumor agents, and also useful in cosmetic formulations and pigments. Safari *et al.* reported a simple, convenient and eco-friendly multicomponent reaction (MCR) for synthesis of chromenes using MgO NPs as a catalyst (Scheme-16). The reaction was exploited in aqueous medium and found to give excellent yields of product at room temperature [81].



Scheme 16. Nanocrystalline MgO catalyzed MCRs leading to 2-amino-4H-chromenes.

Benzopyran is a polycyclic organic compound that results from the fusion of a benzene ring to a heterocyclic pyran ring referred to as chromene and they are recognized as antimicrobial, antiviral,

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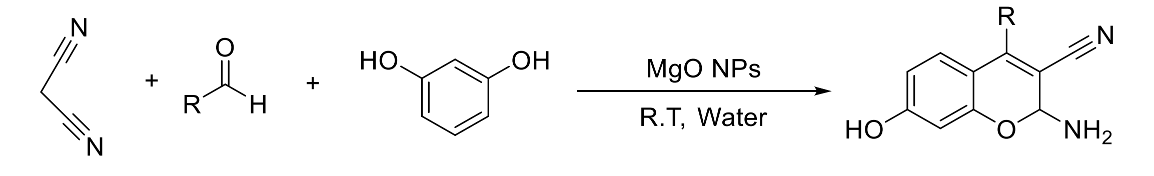


Scheme 17. Nanocrystalline MgO catalyzed MCRs leading to 2-amino-4H-chromenes.

b.Synthesis of Coumarins:

Coumarin and its derivatives are widespread and are very important organic compounds. These compounds have attracted much attention due to their wide range of biological and pharmacological activities. Some biological properties such as molluscacidal, anthelmintic, and hypnotic activities and anticoagulant agents were reported for them (Fig2). Coumarin derivatives also find applications in fragrance, agrochemical industries, food, cosmetics, optical brighteners, dispersed fluorescent, and laser dyes. So, the synthesis of coumarins and their derivatives is of increasing interest. Several routes have been reported for the synthesis of coumarins such as Pechmann, Perkin, Knoevenagel, Reformatsky, and Wittig reactions.

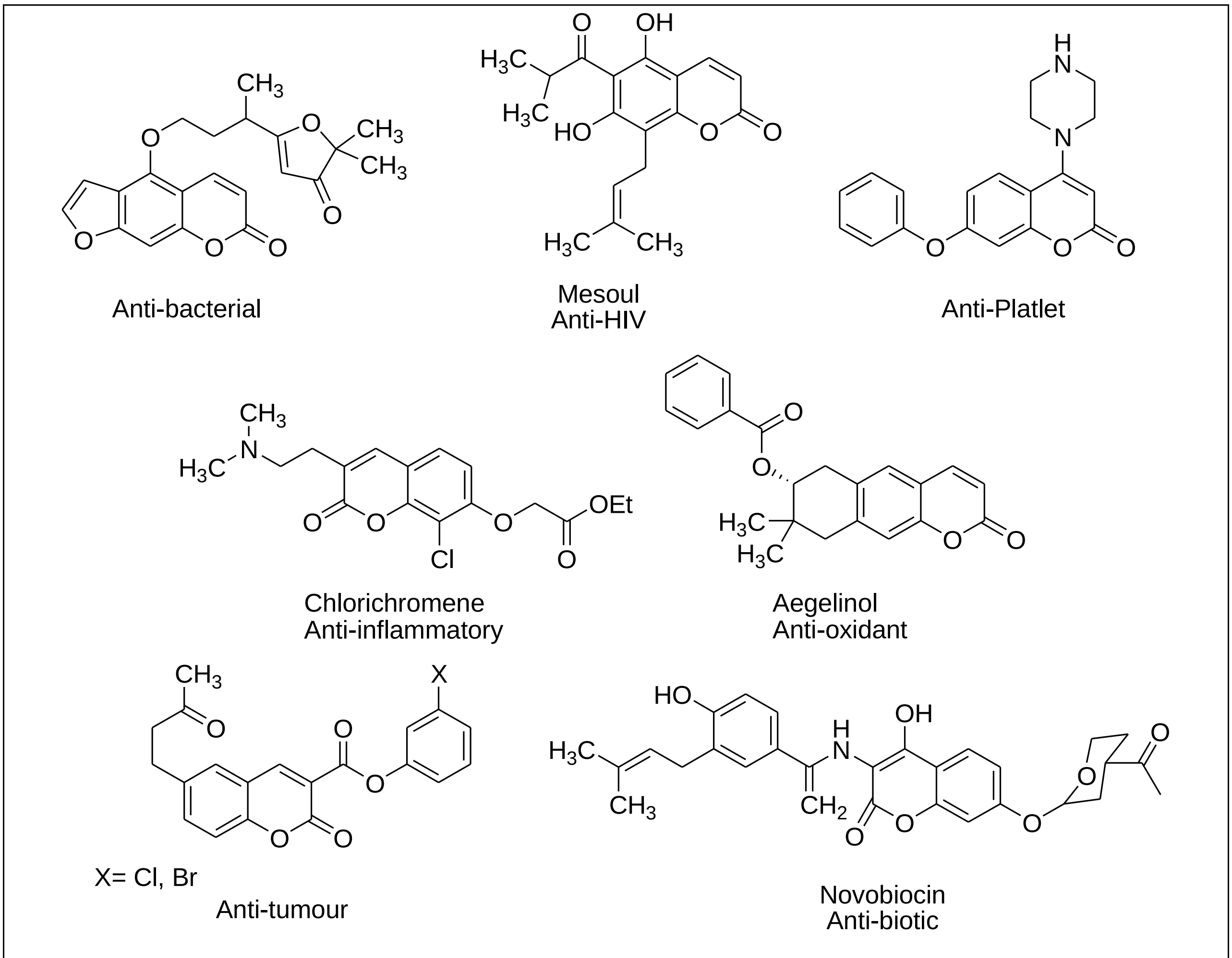
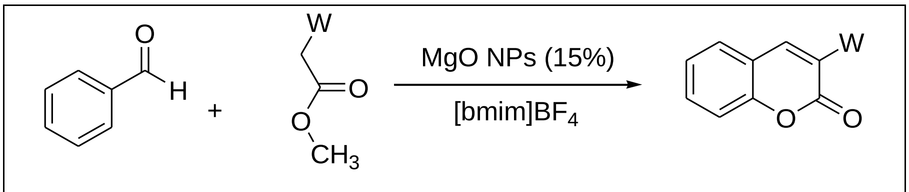


Fig 18. Biologically active some coumarin containing drug molecules (Reproduced from ref. 91)

Recently, coumarins are synthesized using NPs as the catalyst, the best known basic NPs is MgO. Many authors reported the synthesis of coumarin derivatives using MgO NPs which were in turn are synthesized by different methods.

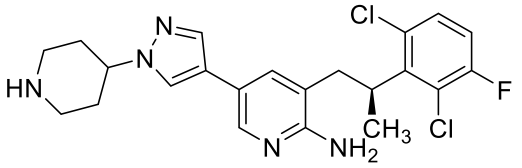
Valizadeh *et al.* reported MgO nanoparticles as an efficient and eco-friendly catalyst for the rapid synthesis of coumarin derivatives in $[bmim]BF_4$ without use of solvent. The salicylaldehyde was reacted with active methylene compound in presence of MgO NPs as a catalyst in $[bmim]BF_4$ at solvent-free conditions to yield coumarin derivatives as a product. The MgO NPs were recovered, wash, dried and further used for the second cycle, it was found that the yield of product comparatively decreased from the first cycle.



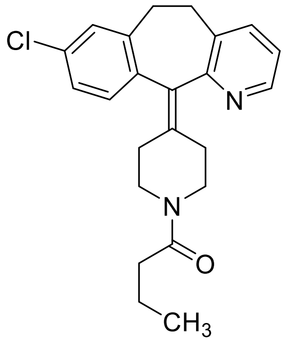
Scheme 19. MgO nanoparticles catalyzed solvent-free synthesis of coumarins. (W=CO_2Et, CO_2Me, CN).

c.Synthesis of Pyridines:

Pyridines represent an important class of bioactive molecules and are particularly useful synthetic intermediates in the preparation of complex nitrogenous natural products and pharmaceutical targets (Fig 2). Pyridines have a widespread natural occurrence and are used to treat a broad spectrum of medical conditions, such as asthma, epilepsy, cancer and kidney diseases, etc.



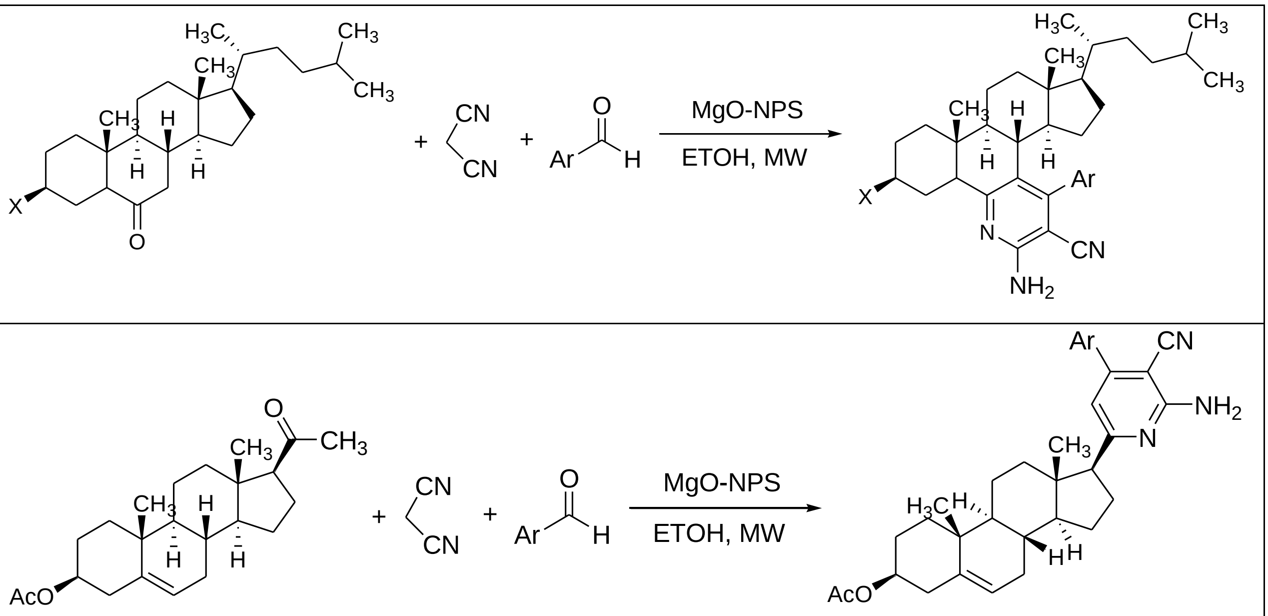
Crizotinib: treatment of lung cancer.



Claritin: treatment of allergies.

Fig 3. Biologically active molecules containing pyridine ring (Reproduced from ref. 95)

Ansari *et al.* synthesized the MgO nanoparticles using precipitation method and characterized with the help of analytical tools like XRD, SEM, FTIR, TGA/DTA etc. The well characterized MgO nanoparticles later explored for the synthesis of series of polysubstituted steroidal pyridines. During the synthesis, steroidal carbonyl compounds were reacted with a substituted aromatic aldehyde, malononitrile, ammonium acetate (Scheme-20) and catalytic amount of MgO NPs. The author tested effect of various parameters on reaction outcome viz., effect of base, solvent, time, temp, etc., and trhe optimized conditions were used for synthesis of library of polysubstituted steroidal pyridines.



Scheme 20. Synthesis of steroidal pyridine derivatives using MgO NPs.

d.Synthesis of Imidazoles:

The imidazolidine- and tetrahydropyrimidine-2-thione derivatives shows excellent medicinal properties such as antiviral, antitumor, anti-inflammatory, and analgesic activities, some of the active molecules are shown in fig.3. Application of NPs for the synthesis of such compound is also noted in literature.

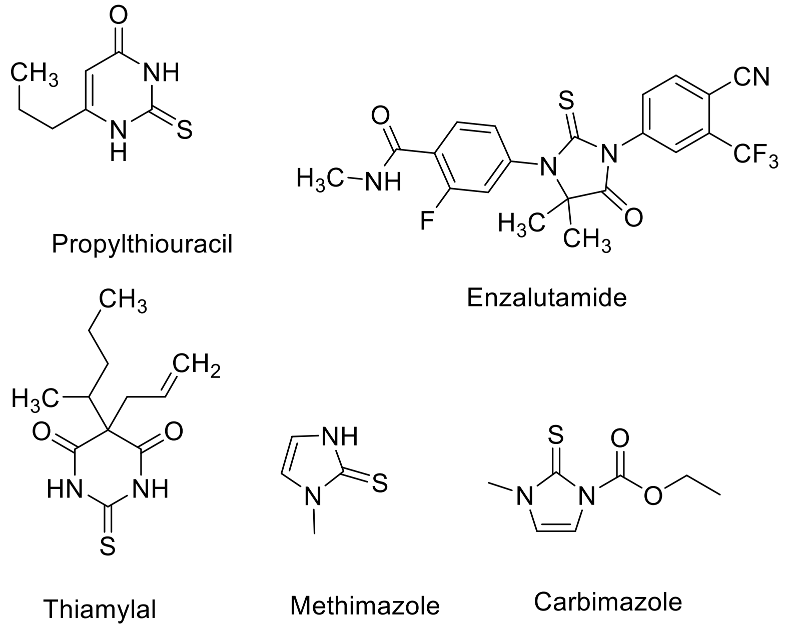
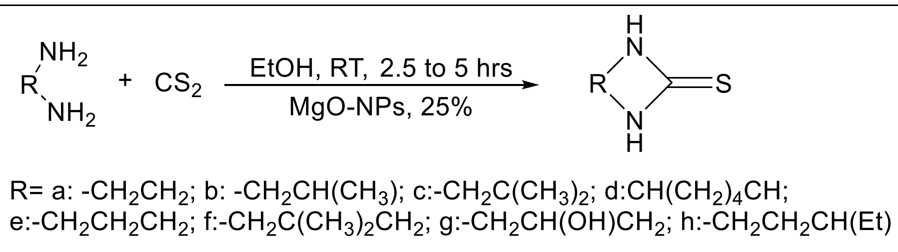


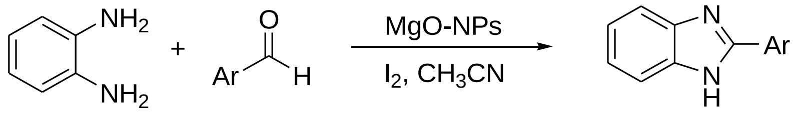
Fig 4. Some biologically active drugs with imidazolidine and tetrahydropyrimidine-2-thione skeletons

Beyzaei *et al.* reported the synthesis of cyclic thiourea using MgO NPs (Scheme-42), which can be used in the synthesis of imidazolidine and tetrahydropyrimidine-2-thione derivatives. The reaction was optimized at different amounts of catalyst and it was found that the higher % of yield obtained at the maximum concentration of MgO NPs. Antibacterial activity of newly synthesized compounds was studied against 14 pathogenic bacteria including gram-positive and gram-negative strains.



Scheme 21. Total synthesis of thiourea derivatives using MgO NPs.

An efficient single-step synthesis of 2-substituted benzimidazole derivatives by nanocrystalline MgO as a solid base catalyst and supported by iodine under acetonitrile solvent at room temperature has been reported by Naeimi *et al.* (Scheme-21).



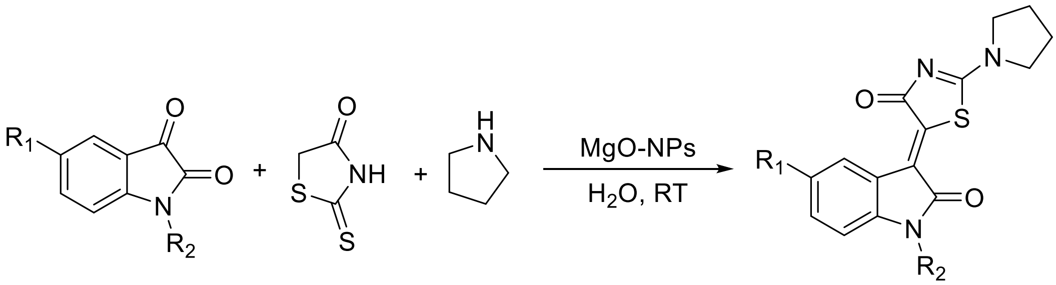
Scheme 22. Synthesis of 2-substituted benzimidazoles from o-phenylenediamine and aryl aldehydes in the presence of nanocrystalline MgO/I_2 using MgO NPs.

The MgO nanocrystals were synthesized using polyvinyl alcohol (PVA), the characterization done by XRD, SEM, TEM, and used as a catalyst in the above said reaction. The reaction was tried in different solvents and different available MgO materials. The reaction found to give best result in acetonitrile (95% yield), also the catalytic activity of nanocrystalline MgO was found to be superior compared with commercial MgO. The proposed mechanism for the reaction.

e.Synthesis of Thiazoles:

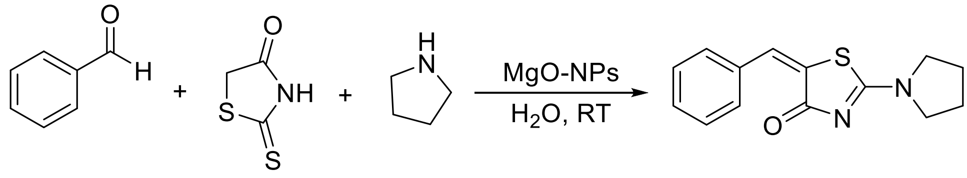
Thiazole moiety plays an important role in the construction of many drugs molecules such as antimicrobial acinitrazole, sulfathiazole, antibiotic penicillin, antidepressant pramipexole, antineoplastic agents bleomycin, tiazofurin, anti-HIV drug ritonavir, antiasthmatic drug cinalukast, antiulcer agent nizatidine contain thiazole ring. The synthesis of thiazole was reported by many researchers using different methods, nanoparticles are also studied for the synthesis of this type of materials, and here some such reports are discussed.

Beyzaeil *et al.* synthesize MgO NPs by wet chemical method in which starch and magnesium nitrate were used along with NaOH. The synthesized MgO nanoparticles were explored for the synthesis of few novel 4-thiazolylpyrazoles by modified Hantzsch method, under solvent-free conditions.



Scheme 23. Preparation of isatin-based 2-amino thiazol-4-one conjugates using MgO NPs.

In the current investigation, the reaction was studied using various inorganic bases along with MgO NPs in different proportions and various amines are tried in aqueous medium at room temperature. The best results were obtained by using 0.3 (eq.) MgO NPs in the water at room temperature. The characterization of synthesized compounds were done with the help of analytical techniques such as IR and NMR, also physical properties such as bond length of synthesized compounds were calculated using ORTEP software. A similar reaction was reported and studied by the same author with aromatic aldehyde as one of the reactant, as shown in reaction Scheme- 45.

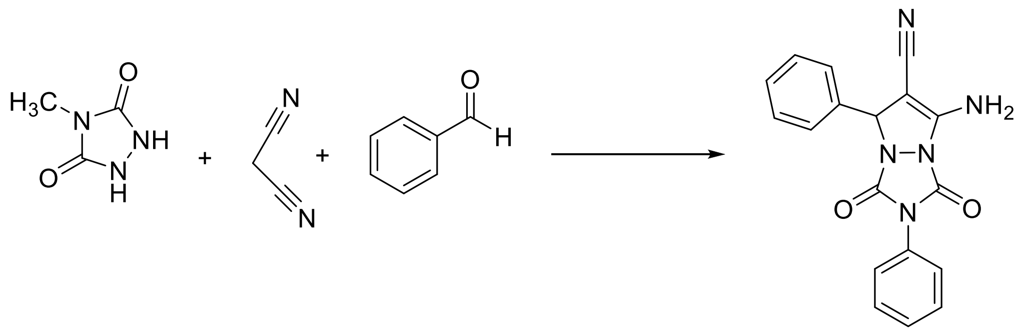


Scheme 24. Preparation of 2-amino-5-arylidenethiazol- 4-ones using MgO NPs.

f.Synthesis of Pyrazoles:

Pyrazoles and its derivatives represent one of the most active class of compounds, which show a variety of biological activities like anti-bacterial, anti-convulsant, analgesic, anti-microbial, anti-inflammatory, anti-diabetic, sedative anti-rheumatic, anticancer, and anti-tubercular activities. Synthesis of pyrazoles was reported by various methods in which the use of metal NPs are among the best methods.

Naeimi *et al.* reported the one-pot synthesis of pyrazolotriazoles in the green medium using MgO NPs as a catalyst. MgO NPs were prepared using a reported procedure and characterized by XRD and TEM techniques. In general the aromatic aldehydes, 4-phenyl-1,2,4-triazolidine-3,5-dione, and malononitrile or alkyl cyanoacetates were reacted in one pot in presence of catalytic amount of nanocrystaline MgO material to produce the desired product. The reported protocol has some advantages like, excellent yields of desired products, easy workup process, use of greener medium and reusability of the catalyst (Scheme-25).

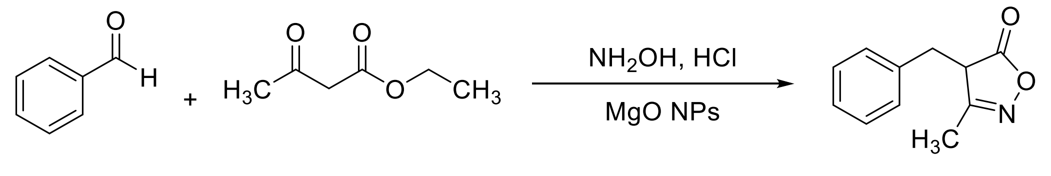


Scheme 25. Synthesis of pyrazolotriazoles.

g.Synthesis of Isoxazole:

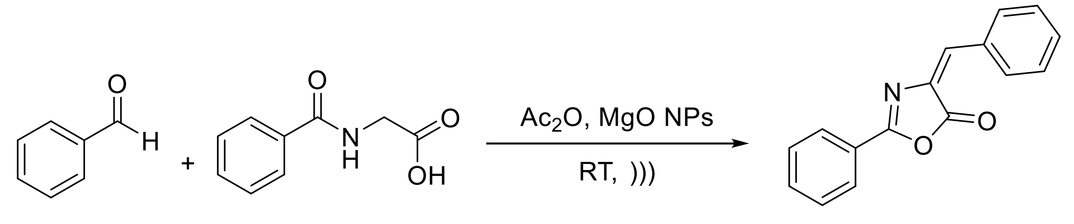
Isoxazole is a member of heterocyclic compounds contains several biological activities such as anti-diabetic, anti-fungal and was used for different purposes, by understanding the utility of isoxazole many researchers interested in the green synthesis of it. Ghorbani *et al.* reported the green synthesis of 3,4-disubstituted isoxazole-5(4H)-ones catalyzed by nano-MgO. The MgO NPs were synthesized using precipitation and hydrothermal methods and characterization were done by XRD

and SEM techniques. The synthesis was attempted with various inorganic and organic bases and best results were obtained with MgO NPs as catalyst (Scheme-26).



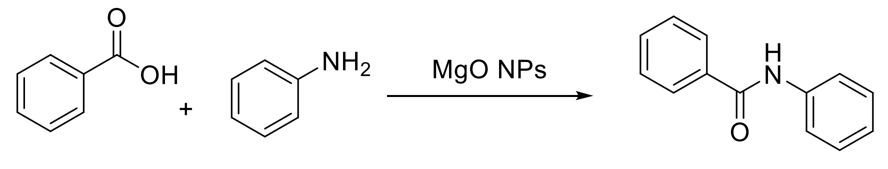
Scheme 26. The reaction of the green synthesis of 3,4-disubstituted Isoxazole-5(4H)-ones using MgO NPs.

Similarly, Sadeq *et al.* reported one-pot synthesis of 4-arylmethylidene -2-phenyl-4H-oxazole-5-ones using nano-MgO as an efficient catalyst exploring ultrasonic waves (Scheme-26). Nano-MgO was prepared by the solution combustion technique, magnesium nitrate and amino acid were taken in petri dish and heated to get semi-solid material which on calcination at 400 ºC gave MgO NPs. The characterization was done with the help of XRD, SEM, and TEM. The well characterized material was then used for aforesaid reaction, which provided good to excellent yields of desired product at room temperature under ultrasonic conditions.



i.Synthesis of Amides:

Synthesis of amide was reported by Das *et al.* using MgO NPs under solvent-free condition [106]. MgO NPs were prepared and analyzed by electronic microscopic techniques such as SEM and TEM, also FTIR, EDX, and XRD were used for further analysis. The catalytic activity of MgO NPs was tested for amide synthesis as shown in Scheme-27.

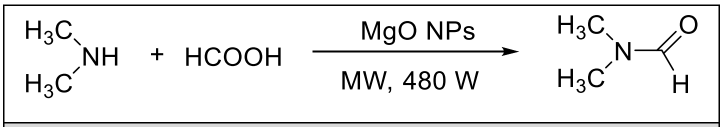


Scheme – 28. Synthesis of amide using MgO NPs.

The reaction of acid with amine was carried out with different solvents and under solvent-free condition as well. The reaction found to provide excellent yield under solvent free conditions in just 10 minutes using MgO NPs as catalyst.

The formylation of amine was reported with the help of MgO-NPs by Bhanage *et al*. MgO NPs were prepared by microwave irradiation method and the characterization of MgO-NPs was done by FT-IR, XRD, SEM, TEM, and CO_2-TPD. In the present paper, the comparative catalytic study was

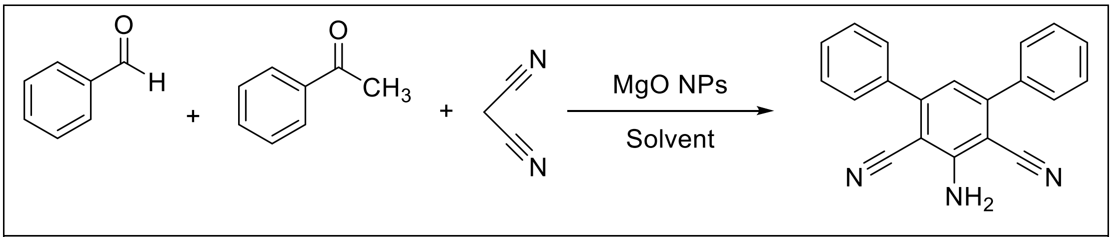
done by taking commercial and synthesized MgO-NPs (Scheme-30). The developed conditions were used for the synthesis of library of N-formylated products. The products were well characterized by analytical tools like, FTIR, mass and NMR.



Scheme – 30. Formylation of amine using MgO NPs.

4.3.4.Other reactions:

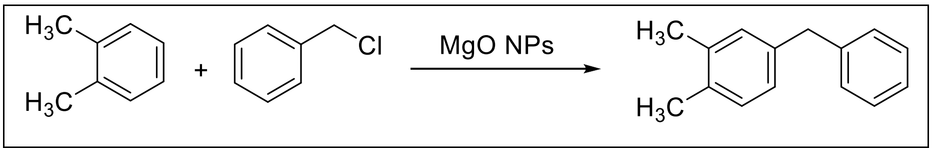
Safaei-Ghomi *et al.* reported the one-pot syntheses of synthesis of 2,6-dicyanoanilines and 1,3-diarylpropyl malononitrile under different conditions using MgO nanoparticles as an efficient, green and reusable catalyst [108].



Scheme 31. The three-component reaction of aldehydes, acetophenone, and malononitrile catalyzed by MgO NPs.

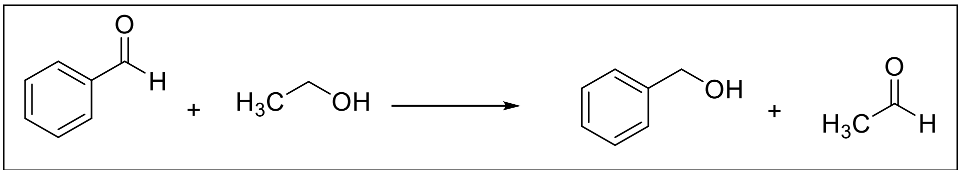
The MgO NPs material was obtained by using magnesium nitrate, the synthesized MgO NPs was characterized using electronic microscopy such SEM and nature of MgO NPs was found with help of XRD techniques. The catalytic activity was tested against organic transformation and found to be an active catalyst shown in Scheme-31.

Benzylation of Aromatic Compounds was reported by Choudary *et al*. (Scheme-32) using polycrystalline CM-MgO, microcrystalline CP-MgO and nanocrystalline aerogel prepared AP-MgO in o-xylene and toluene, due to the higher surface area of AP-MgO having grater active sites which leads higher yield [109].

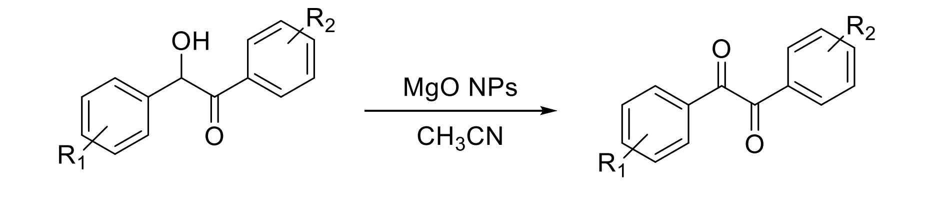


Scheme 32: Benzylation of aromatic compounds using MgO NPs.

Another important application of MgO NPs was reported by Wang *et al*., he studied the applications of MgO nanosheets, nanodisks and nanofibers against Meerwein–Ponndorf–Verley reaction. The synthesized MgO nanomaterials were analyzed by spectroscopic, electron spectroscopy and CO_2-TPD technique (Scheme-53). Safari et al. reported the green synthesis of benzil using MgO NPs shown in (Scheme-34).



Scheme 33. Meerwein–Ponndorf–Verley reaction.



Scheme 34. Oxidation of benzoin derivatives to benzil derivatives using nanocrystalline MgO.

R_1 and R_2 – H, 4-OCH_3, 4-NCH_3, 4-CH_3, NC_5H_5, 2, 4-diOCH_3.

Discussion:

Over the past decades, researchers have concentrated on the synthesis and development of shape and size selective nanocrystals. Several scientists have made attempts towards the fabrication of MgO NPs, but few of them accomplished size and shape controlled NPs. In this review, we firstly summarized the current advances in protocols for the synthesis of MgO NPs, including chemical, physical and biological synthesis routes and their diverse properties/features. Then we discussed the characterization of MgO NPs by different known techniques. Finally, we have discussed several MgO NPs catalyzed organic transformations such as epoxidation, condensation and C-C, C-N, C-O, C-S bond formation in variety of notable heterocyclic reactions.

Notably, the catalysis using MgO NPs does not necessitate any ligand precursor but low catalyst loading with low temperature, mild reaction conditions and easy separation of catalyst with recyclability makes it attractive.

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