

ONE POT SYNTHESIS OF ALPHA, BETA ACETAMIDO KETONE USING MAGNESIUM OXIDE/CARBON NANO COMPOSITES

Submitted in partial fulfilment of the requirements for the award of Bachelor
of Science in Chemistry

by

SUSHMA JAYA RAHINI.J (40030039)

ELAKKIYA.A (40030007)

MAHALAKSHMI.D (40030020)



DEPARTMENT OF CHEMISTRY

SCHOOL OF SCIENCE AND HUMANITIES

SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

Accredited with Grade "A" by NAAC | 12B Status by UGC | Approved by AICTE

JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI - 600 119



SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)

Accredited "A" Grade by NAAC | 12B Status by UGC | Approved by AICTE
www.sathyabama.ac.in

DEPARTMENT OF CHEMISTRY BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of Mrs. SUSHMA JAYA RAHINI.J (40030039), Mrs. ELAKKIYA.A (4003007), Mrs. MAHALAKSHMI.D (40030020) who carried out the project entitled "ONE POT SYNTHESIS OF ALPHA, BETA ACETAMIDO KETONE USING MAGNESIUM OXIDE/CARBON NANO COMPOSITE" under our supervision from November 2022 to May 2023.

Internal Guide

Dr. T. KRITHIGA, M.Sc., M.phil, Ph.D.,
Associate professor
Department of chemistry

Head of the Department

Dr. J. KARTHIKEYAN, M.Sc., Ph.D.
Associate Professor
Department of chemistry
Sathyabama Institute of Science and Technology
Jennolagar, Rajiv Gandhi Salai
Chennai - 600 119

Submitted for Viva voce Examination held on 08.05.2023

Internal Examiner

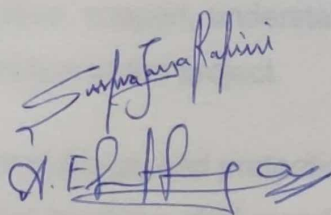
External Examiner

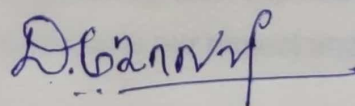
DECLARATION

We SUSHMA JAYA RAHINI.J(40030039), ELAKKIYA.A(40030007), MAHALAKSHMI.D(40030020), hereby declare that the Project Report entitled "ONE POT SYNTHESIS OF ALPHA,BETA ACETAIDO KETONE USING MAGNESIUM OXIDE/CARBON NANO COMPOSITE" done by us under the guidance of Dr. T. KRITHIGA, M.Sc., M.Phil., Ph.D., Associate professor, Department of Chemistry at Sathyabama Institute of Science and Technology, Jeppiaar Nagar, Rajiv Gandhi Salai, Chennai – 600119 is submitted in partial fulfillment of the requirements for the award of Bachelor of Science in Chemistry.

DATE: 08/05/2023.

PLACE: CHENNAI





J. SUSHMA JAYA RAHINI

A. ELAKKIYA

D. MAHALAKSHMI

SIGNATURE OF THE CANDIDATE

ACKNOWLEDGEMENT

We would like to express our deepest gratitude to the following individuals and organizations who have helped us throughout our project,

We pleased to acknowledge our sincere thanks to Board of Management of **SATHYABAMA** for their kind encouragement in doing this project and for completing it successfully.

We express our special thanks to **Dr. REKHA CHAKRAVARTHI**, Dean of the Department of chemistry for her support and also for giving long period of time to complete this project.

We convey our thanks to **Dr. J KARTHIKEYAN**, Head of the Department, Chemistry for providing me necessary support and details at the right time during the progressive reviews.

We would like to express our sincere and heartfelt thanks of gratitude to our Project Guide **Dr. T. KRITHIGA** for providing us with valuable advice, guidance and words of encouragement for the successful completion of project work.

Also, to our family who provided us with unending love, support, understanding, and more importantly financial assistance during the duration of the project.

Finally, we would like to thank the God for his enduring grace, and protection. And we wish to express our thanks to all Teaching and Non-teaching staff members of the Department of Chemistry who were taking the time to evaluate our project and provide helpful feedback to the completion of the project.

J. SUSHMA JAYA RAHINI

A. ELAKKIYA

D. MAHALAKSHMI

ABSTRACT

Magnesium oxide nanoparticles have been synthesized by sol-gel method. The synthesized nanocomposite has been characterized by FTIR, XRD and SEM. XRD studies show that MgO/C nanocomposite structure. The SEM analysis confirms the formation of spherical particles which are agglomerated. FTIR confirms the presence of Mg – O, C = O stretching bond. The catalytic activity of the synthesized catalyst MgO/C nanocomposite is tested by one pot synthesis of alpha, beta acetamido ketone. Optimization of reaction parameter such as effect of temperature, effect of catalyst weight was determined. The optimized condition for the highest yield of alpha, beta acetamido ketone is in pH of 5 and weight of the catalyst = 30mol%. The synthesized product was characterized by FTIR techniques.

CATALYST

TYPES OF CATALYST

HOMOGENEOUS CATALYST

HETEROGENEOUS CATALYST

MODIFIED HOMOGENEOUS CATALYST

NO-CATALYST

BIOMIMETIC

TYPES OF NANOPARTICLE

ORGANIC NANOPARTICLE

ORGANIC INORGANIC NANOPARTICLE

INORGANIC NANOPARTICLE

TABLE OF CONTENT

CHAPTER NO	TITLE	PAGES NO
	ABSTRACT	i
	LIST OF FIGURES	v
	LIST OF ABBREVIATIONS	vi
1	INTRODUCTION	1
1.1	CATALYST	1
1.1.1	TYPES OF CATALYST	1
1.1.2	HOMOGENEOUS CATALYST	1
1.1.3	HETEROGENEOUS CATALYST	2
1.1.4	HETEROGENIZED HOMOGENEOUS CATALYST	2
1.1.5	BIO CATALYST	2
1.2	NANOPARTICLES	2
1.2.1	TYPES OF NANOPARTICLES	3
1.2.2	CARBON BASED NANOPARTICLE	3
1.2.2.53	CERAMIC BASED NANOPARTICLE	3
1.2.4	SEMICONDUCTOR NANOPARTICLE	4

1.2.5	POLYMERIC NANOPARTICLE	4
1.2.6	LIPID BASED NANOPARTICLE	4
1.2.7	METAL OXIDE NANOPARTICLE	4
1.2.8	MAGNESIUM OXIDE NANOPARTICLE	4
1.3	CARBON AS CATALYST	5
1.4	NANOCOMPOSITES	6
1.4.1	NON – POLYMER NANOCOMPOSITE	6
1.4.2	POLYMER NANOCOMPOSITE	7
1.4.3	METAL BASED NANOCOMPOSITE	7
1.4.4	MAGNESIUM OXIDE/CARBON NANOCOMPOSITE	7
2	LITERATURE SURVEY	8
3	AIM AND SCOPE	10
3.1	AIM	10
3.2	SCOPE	10
4	MATERIALS AND METHODS	11
4.1	MATERIALS	11
4.2	CHARACTERIZATION TECHNIQUES	11
4.2.1	POWDER X – RAY DIFFRACTION (XRD)	11

4.2.2	FOURIER TRANSFORM INFRARED (FTIR)	12
4.2.3	SCANNING ELECTRON MICROSCOPY (SEM)	13
4.3	METHADODOLOGY	15
4.3.1	SYNTHESIS OF MgO/C NANOCOMPOSITE	15
4.3.2	SYNTHESIS OF ALPHA, BETA ACETAMIDO KETONE	15
5	RESULTS AND DISCUSSION	16
5.1	CHARACTERIZATION OF MAGNESIUM OXIDE/CARBON NANOCOMPOSITE	16
5.1.1	FTIR SPECTRUM OF MAGNESIUM OXIDE NANOCOMPOSITE	16
5.1.2	SEM IMAGE OF MAGNESIUM OXIDE/CARBON NANOCOMPOSITE	17
5.1.3	XRD PATTERN OF MAGNESIUM OXIDE/CARBON NANOCOMPOSITE	17
5.1.4	CATALYTIC STUDIES	18
6	CONCLUSIONS	20
	REFERENCES	21

LIST OF FIGURES

Figure No	Title	Page No
1.2	NANOPARTICLES	3
1.2.8	SYNTHESIS OF MANESIUM OXIDE NANOPARTICLE	5
1.3	CARBON AS CATALYST SOURCE	6
1.4	TYPES OF NANOCOMPOSITES	6
4.2.2	BRAGG'S LAW OF REFLECTION	12
4.2.3	INSTRUMENTATIONAL OF FTIR	13
4.2.4	SCHEME OF SCANNING ELECTRON MICROSCOPY	14
4.3.2	SYNTHESIS OF ALPHA, BETA ACETAMIDO KETONE	15
5.1.1	FTIR SPECTRUM OF MgO/CNANOCOMPOSITE	16
5.1.2	SEM IMAGE OF MgO/C NANOCOMPOSITE	17
5.1.3	XRD PATTERN FOR MgO/C NANOCOMPOSITE	18
5.1.4	EFFECT OF CATALYST LOADING ON ALPHA, BETA ACETAMIDO KETONE CONVERSION OVER MGO/C COMPOSITE	18
5.1.5	EFFECT OF TIME ON ALPHA, BETA ACETAMIDO KETONE CONVERSION OVER MGO/C	19

LIST OF ABBREVIATION

FTIR	- Fourier Transform Infrared
XRD	- X – Ray Diffraction
SEM	- Scanning Electron Microscopy
EDX	- Energy Dispersive X – Ray Analysis
IR	- Infrared radiation
nm	- Nanometer
cm	- Centimeter
ml	- Milliliter
g	- Gram
MgO/C	- Magnesium Oxide/Carbon
MgNO ₃	- Magnesium Nitrate

CHAPTER 1

INTRODUCTION

1.1 CATALYST

Catalysis is the process of increasing the rate of a chemical reaction by adding a substance known as a catalyst. Catalysts are not consumed in the reaction and remain unchanged after it. If the reaction is rapid and the catalyst recycles quickly, very small amount of catalyst often suffice, mixing, surface area, and temperature are important factors in reaction rate. Catalysts generally react with one or more reactants to form intermediates the subsequently give the final reaction product, in the process of regenerating the catalyst. Catalysis is a process of adding a catalyst to ease the reaction using the catalyst leads to fastening the chemical reaction.

1.1.1 TYPES OF CATALYSTS

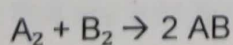
Mainly catalysts are four types,

- Homogeneous catalyst
- Heterogeneous catalyst
- Heterogenized homogeneous catalyst
- Biocatalyst

1.1.2 HOMOGENEOUS CATALYST

Homogeneous catalyst is a type of catalyst which have same phase as the phase of reaction throughout the reaction. Some of the homogeneous catalysts are transition metal, organometallic complexes, organocatalysts. Homogeneous catalysis is carbonylation, oxidation, hydrocyanation, metathesis, and hydrogenation.

Example: The production of Sulphur trioxide by the reaction of Sulphur dioxide and Oxygen in the presence of Nitric oxide as a catalyst.



1.1.3 HETEROGENOUS CATALYST

Heterogeneous catalysis is a type of catalysis in which the catalyst occupies a different phase than the reaction mixture. Heterogeneous catalysts are solids that are added into gas or liquid reaction mixtures. In heterogeneous catalysis, the reactants adsorb onto binding sites on the surface of the catalyst, and the availability of these reaction sites can limit the rate of heterogeneous reactions.

Example: The synthesis of ethylene oxide, which involves the reaction between ethylene and oxygen, catalysed by silver on alumina along with several other promoters.

1.1.4 HETEROGENIZED HOMOGENEOUS CATALYST

Heterogenization of homogeneous catalytic systems can be defined as the modification of homogeneous catalysts to "break" the uniformity among the molecules of the catalyst and the products by rendering them in separable phases to allow their separation. Heterogeneous catalyst is different from the homogeneous counterpart are much more difficult to develop practically because of their complexity.

1.1.5 BIOCATALYST

Biocatalysts are generally less polluted and can be decomposed easily. They are comparatively faster than catalyst. The examples of biocatalyst include hormones or enzymes, which increase the rate of biochemical reactions.

Example: Digestive enzymes such as trypsin, pepsin etc. Note: The catalyst is the substance which increases the speed and rate of the reaction.

1.2 NANOPARTICLES

A nanoparticle or ultrafine particle is usually defined as a particle of matter that is between 1 and 100 nm in diameter. The term is sometimes used for larger particles, up to 500 nm, or fibers and tubes that are less than 100 nm in only two directions.

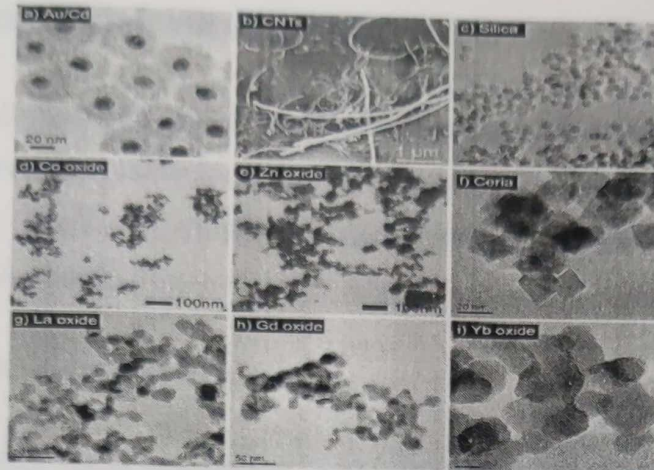


Fig 1.2 Nanoparticles

1.2.1 TYPES OF NANOPARTICLES

They are classified into different types based on their size, morphology, physical and chemical properties,

- Carbon- based nanoparticle
- Ceramic nanoparticle
- Metallic-oxide nanoparticle
- Semiconductor nanoparticle
- Polymeric nanoparticle
- Lipid-based nanoparticle

1.2.2 CARBON-BASED NANOPARTICLE

Carbon-based – (fullerenes, carbon nanotubes, graphene, carbon dots). These materials are of great interest due to their electrical conductivity, high strength, structure, electron affinity, and versatility.

1.2.3 CERAMIC NANOPARTICLE

Ceramic – These inorganic nonmetallic solids are getting great attention of researchers due to their use in applications such as catalysis, photocatalysis, photodegradation of dyes, and imaging applications.

1.2.4 SEMICONDUCTOR NANOPARTICLE

Semiconductor – Semiconductor materials possess properties between metals and non-metals and have wide bandgaps. Bandgap tuning results in significant alteration in their properties. Therefore, they are very important materials in photocatalysis, photo optics and electronic devices.

1.2.5 POLYMERIC NANOPARTICLE

Polymers – Scientists have developed many techniques to synthesize polymeric nanoparticles for a wide range of applications including surface coating, sensor technology, catalysis, and nanomedicine.

1.2.6 LIPID - BASED NANOPARTICLE

Lipids – These NPs contain lipid moieties and are used in many biomedical applications as drug carriers (the mRNA Covid-19 vaccines are using lipid nanotechnology). Lipid nanoparticles are also regarded as highly promising systems for delivering nucleic acids in gene therapy.

1.2.7 METAL - OXIDES NANOPARTICLE

Metal – They are purely made of the metal precursors. Due to well-known localized surface plasmon resonance (LSPR) characteristics, these possess unique optoelectrical properties.

1.2.8 MAGNESIUM OXIDE NANOPARTICLE

Magnesium oxide nanoparticles is widely used among the other metal oxides nanoparticles that widely being used in various fields because they have promising structure material in biological implants due to their high strength to weight ratio, low density, good functionality, recycling activity, nontoxic and hygroscopic nature. Magnesium oxide nanoparticles are adsorbing and non-toxic. They are like white powdered substance. They are highly hard, highly pure, and they have high melting point. Magnesium oxide nanoparticles can be used in as electronics, catalyst, ceramics, petrochemicals products, coating and numerous other fields. There are

few methods to synthesis by methods like sol-gel, precipitation method, and green synthesis and it is widely used as toxic Waste remediation, antibacterial materials, removal of industrial pollutants and anti-cancer activities.

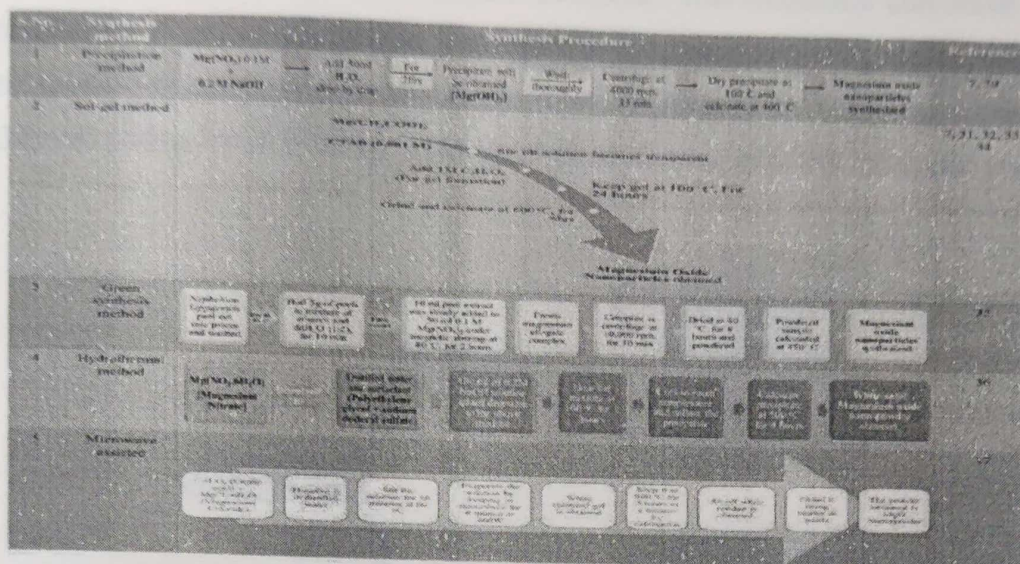


Fig 1.2.8 Synthesis of Magnesium Oxide nanoparticle

1.3 CARBON AS CATALYST

Carbon can be used as both catalyst and catalyst support for the chemical and enzymatic reactions because they have large surface area, excellent electron conductivity and chemical inertness. When the carbon is chemically mixed with nanoparticles and enzymes the catalytic activity of carbon is increased.

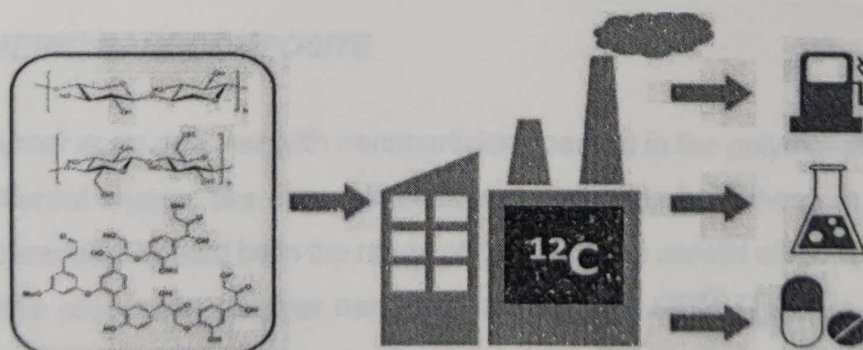


Fig 1.3 Carbon as Catalyst Source

1.4 NANOCOMPOSITES

Nanocomposites are solid materials that have multiple phase domains and at least one of these domains has a nanoscale structure. The materials can have novel chemical and physical properties that depend on the morphology and interfacial characteristics of the component materials. General form of an inorganic matrix set in the organic phase, or vice versa, from an organic matrix set in the inorganic phase.

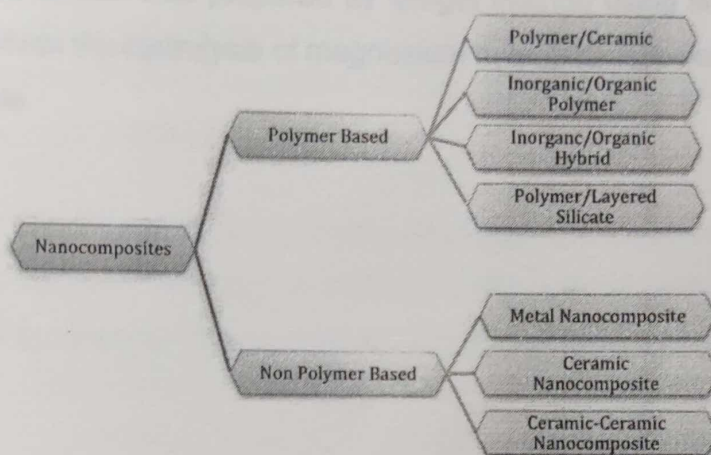


Fig 1.4 Types of Nanocomposites

1.4.1 NON-POLYMER-BASED NANOCOMPOSITE

the nanocomposite which is not consist of any polymer compounds in it is called non polymer nanocomposites. Non-polymer-based nanocomposites can be also classified as: metal/metal nanocomposites, for example Pt-Ru nanocomposites

1.4.2 POLYMERIC NANOCOMPOSITE

consists of polymer or co polymer with nanoparticles speared in the polymer phase. They are of different shapes like fiber, platelets and spheroids from those at least one of its one dimension should be in the range of 150nm. They consist of multiphase system. In plastic production polymer nanocomposites cover 95%. They are done by controlled manner of compounding.

1.4.3 METALLIC OXIDE NANOCOMPOSITE

Bimetallic nanoparticles being investigated in detail in the form of either of alloy or core-shell structures due to their improved catalytic properties and advancement in optical properties related to individual and differentiate metals

1.4.4 MAGNESIUM OXIDE/CARBON NANOCOMPOSITE

Magnesium oxide/carbon was prepared by sol-gel method using sucrose as binder. The method involves the hydrolysis of magnesium hydroxide followed by annealing at room temperature.

CHAPTER 2

LITERATURE SURVEY

Jagadeesan Aravind Kumar, et al., (2014) demonstrated that activated carbon can be prepared by palm shell agro waste by chemical activation method using potassium hydroxide. Nano-Magnesium oxide is placed onto palm shell. Activated carbon composite was produced by dropwise mechanism and its efficiency was tested for the removal of anthracene from the aqueous solution. Amorphous nature of composites was confirmed by X-Ray diffraction.

KR Nemade and SA Waghuley (2021) synthesized MgO using solvent spray pyrolysis technique, since reaction can be run in liquid state without further heating. MgO extensively used as catalyst. The crystallinity structure of MgO pattern was confirmed by XRD.

R Prasanth, et al., (2019) demonstrated new avenues to fight and prevent diseases using atomic scale tailoring of materials. The present investigation describes the mangrove *Rhizophora lamarckii*'s property of synthesizing magnesium oxide nanoparticles. The newly synthesized magnesium oxide nanoparticle morphology is nano hexagonal and spherical. The particles range in dimensions between 20 and 50 nm and are crystalline in nature. The functional groups of the mangrove, amine, and alkane are found to act as reductants and stabilizers.

R Dobrucka (2018) demonstrated that we can synthesis MgO nanoparticle using an alternative way to the traditional chemical method. Here the aim is to synthesis MgO nanoparticle using the *Artemisia abrotanum* herb water extract. It is characterized by UV-Visible spectroscopy, (FTIR) Fourier transform infrared spectroscopy, (XRD) X-ray diffraction, (SEM) scanning electron microscopy with EDS and (TEM) transmission electron microscopy. The crystallite structure of MgO is known XRD. The average size of the MgO nanoparticle was found in the range of 10nm using SEM. EDS conforms the signal characteristic of magnesium and oxygen. FTIR analysis confirmed the presence of active compounds responsible for the stabilization of MgO Nanoparticle.

R Wahab, et al., (2007) demonstrated the synthesis of Magnesium oxide nanoparticle by sol-gel method using Magnesium nitrate and sodium hydroxide at the room temperature. Hydrated Magnesium Oxide were heated in air at 300 and 500°C. crystallinity of the nanoparticle is obtained by X-ray diffraction. Morphology and size of the nanoparticle of the nano particle is shown at the range of 50-70nm powder composition was analyzed by the FTIR spectroscopy.

S. Abinaya, et al., (2021) obtained the nanoparticle in less hazardous process. The Magnesium Oxide nanoparticle have been effectively produced by green synthesis which is environmentally friendly, non-toxic with greater stability.

Saeid Taghavi Fardood, et al., (2018) demonstrated that they can synthesis MgO nanoparticles using Arabic gum as a bio template and magnesium nitrate as the magnesium source by the sol-gel method. This method has many advantages such as nontoxic, economic viability, ease to scale up, less time consuming and environment friendly approach for the synthesis of MgO nanoparticles without using any organic chemicals. Nanoparticles were characterized by FTIR, XRD. The average crystallite size of MgO nanoparticles was calculated using Scherrer formula and Williamson Hall equation. The XRD analysis revealed the formation of cubic phase MgO with average particle size of 14 (Scherrer formula) and 5.3 (Williamson Hall equation) nm.

• Characterization of MgO/C by FTIR, XRD and SEM

• The catalytic activity of MgO/C nanocomposites is tested by one pot synthesis of alpha, beta unsaturated ketone.

• Optimization of reaction parameters such as effect of temperature, effect of catalyst weight was determined. The optimum condition for the highest yield of alpha, beta unsaturated ketone is in pH and weight of the catalyst.

CHAPTER 3

AIM AND SCOPE

3.1 AIM

The aim of this project is to synthesis MgO/C nanocomposites from magnesium oxide, sodium hydroxide, sucrose using sol-gel method. MgO/C nanocomposites were characterized with the technique called FTIR, XRD, SEM. The catalytic activity of the synthesized MgO/C nanocomposite is confirmed by one pot synthesis of alpha, beta acetamido ketone. Optimization of reaction parameter such as effect of temperature, effect of catalyst weight was determined. The optimized condition for the highest yield of alpha, beta acetamido ketone is in pH of 5 and weight of the catalyst = 30mol%. The synthesized product was characterized by FTIR techniques.

3.2 SCOPE

- Synthesizing of MgO/C by sol-gel method. Using magnesium nitrate, sodium hydroxide and sucrose.
- Characterization of MgO/C by FTIR, XRD and SEM.
- The catalytic activity of MgO/C nanocomposites is tested by one pot synthesis of alpha, beta acetamido ketone.
- Optimization of reaction parameter such as effect of temperature, effect of catalyst weight was determined. The optimized condition for the highest yield of alpha, beta acetamido ketone is in pH and weight of the catalyst noted.

CHAPTER 4

MATERIAL AND METHODOLOGY

4.1 MATERIALS:

Materials used in this reaction are magnesium nitrate, sodium hydroxide, deionized water, sucrose which were purchased from micro fine chemicals and it can be used without further purification.

4.2. CHARACTERIZATION TECHNIQUES:

4.2.1 Powder X-Ray Diffraction (XRD)

X-ray diffraction is a common technique that determine a sample's composition or crystalline structure. For larger crystals such as macromolecules and inorganic compounds, it can be used to determine the structure of atoms within the sample. If the crystal size is too small, it can determine sample composition, crystallinity, and phase purity. This technique sends x-ray beams through it. X-ray beams are chosen because their wavelength is similar to the spacing between atoms in the sample, so the angle of diffraction will be affected by the spacing of the atoms in the molecule, as opposed to using much larger wavelengths, which would be unaltered by the spacing between atoms. The x-rays then pass through the sample, "bouncing" off of the atoms in the structure, and changing the direction of the beam at some different angle, theta, from the original beam. This is the angle of diffraction. Some of these diffracted beams cancel each other out, but if the beams have similar wavelengths, then constructive interference occurs. Constructive interference is when the x-ray beams that are whole number integers of the same wavelength add together to create a new beam with a higher amplitude. The greater amplitude of the wave translates into a greater signal for this specific angle of diffraction. The angle of diffraction can then be used to determine the difference between atomic planes using Bragg's law. The distance between atomic plates can then be used to determine composition or crystalline structure.

Bragg's law: $\sin \Theta = n \lambda / 2 d$

where λ = wavelength added

θ = angle of diffraction

d = distance between atomic planes.

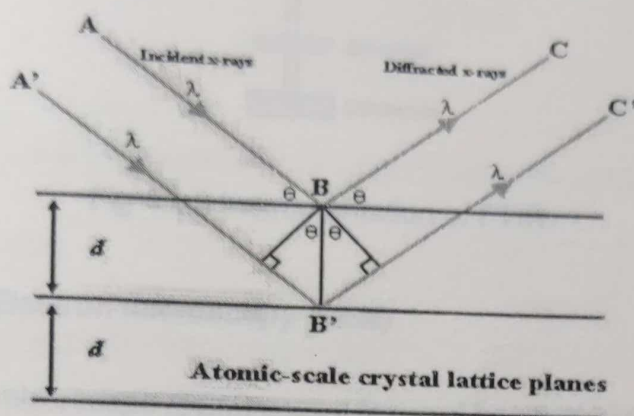


Fig 4.2.2 Bragg's law of reflection

4.2.3 Fourier Transform Infrared Spectrometer (FTIR)

FTIR is an analytical technique used to identify organic, polymeric and inorganic materials. The FTIR analysis method uses infrared light to scan test sample and observe chemical properties. The FTIR instrument sends infrared radiation of about $10,000$ to 100 cm^{-1} through a sample, with some radiation absorbed and some passes through. The absorbed radiation is converted into rotational or vibrational energy by the sample molecule. The resulting signal at the detector presents as spectrum, typically from 4000 cm^{-1} to 400 cm^{-1} , representing a molecular fingerprint of the sample. Each molecule or chemical structure will produce a unique spectral fingerprint, making FTIR analysis a great tool for chemical identification. FTIR is an established technique for quality control when evaluating industrially manufactured material, and can often serve as the first step in the material analysis process. A change in the characteristic pattern of absorbed bands clearly indicates a change in composition of the material or the presence of contamination. This technique is useful for analyzing the chemical composition of smaller particles, typically 10-50 microns. FTIR analysis is used to identify and characterize the unknown materials and identify the contamination in a material.

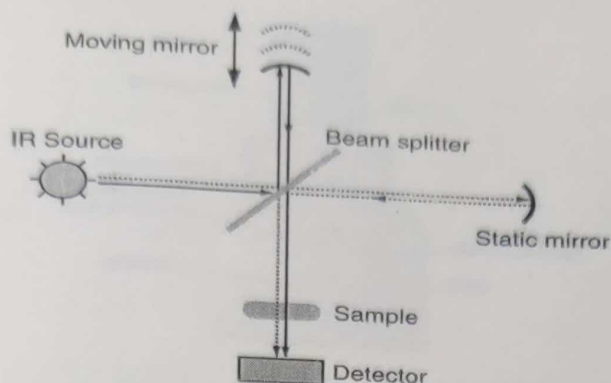


Fig 4.2.3 Instrumentation of FTIR

4.2.4 Scanning Electron Microscopy (SEM)

Scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface, the signal comes out from the electron-sample interaction reveal the information about the sample including external morphology, chemical composition, and crystalline structure and orientation of materials making up the sample. it a high-resolution image of the size and surface texture of the particle, SEM equipped with EDX can determine the main atomic composition of pf the particle, particle colours cannot be determined by SEM. SEM is one of the main testing instruments for in situ analysis of micro areas. It has high magnification, and strong stereoscopic view. Photographs taken by SEM have higher magnification and clearer definitions than those by optical microscopy. SEM can be able to measure only elements with atomic coefficients less than 5. Using SEM, we can get the selected point location images of the sample. This approach is especially useful in qualitative determining chemical composition. In SEM examination system the zones territory from around 1cm to 5 microns in width can be imaged in a filtering mode utilizing SEM methods and having amplification run from spatial goals of 50nm to 100nm.

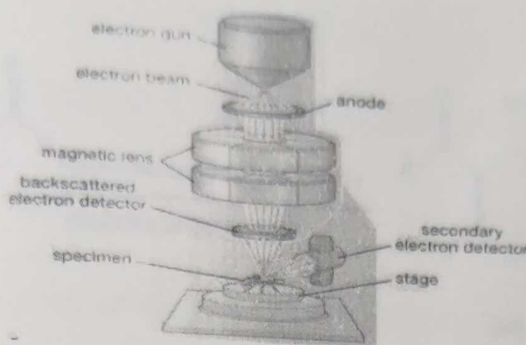


Fig 4.2.4 Scheme of Scanning Electron Microscope

4.3 METHODOLOGY:

4.3.1 *Synthesis of MgO/C nanocomposite*

3.42 g of sucrose is dissolved in 100 ml of distilled water in a Std flask. 0.1M of MgNO_3 solution is prepared by dissolving 2.56 g of MgNO_3 salt in 100 of distilled water.

Take 40ml of the prepared sucrose solution in beaker and place it in hot plate magnetic stirrer. When the solution begins to boil add 40ml of MgNO_3 solution drop by drop.

A gel like consistency is obtained, then turn off the stirrer and transfer the contents to a crucible. Place the crucible in oven (80°C) for the moisture to evaporate (Approximately 3hrs). Then, place the crucible in muffle furnace at 400°C where the reaction takes place in 7 min, an amorphous powder is obtained. Then, place the crucible at 600°C for calcination to occur.

4.3.2 *Synthesis of Alpha, Beta Acetamido Ketone*

The experimental procedure for this reaction is simple and required no inert atmosphere. The mixture of benzaldehyde, acetophenone, acetyl chloride, in the ratio 1:1:2 and acetonitrile (5ml) along with catalyst is taken in round bottom flask and refluxed at 60°C for appropriate time. The mixture was cooled and poured into ice. The solid formed was separated and dissolved in dichloromethane. The catalyst was recovered by filtration which can be reused. The organic layer was stirred with a minimum amount of diethyl ether to obtain the pure product.

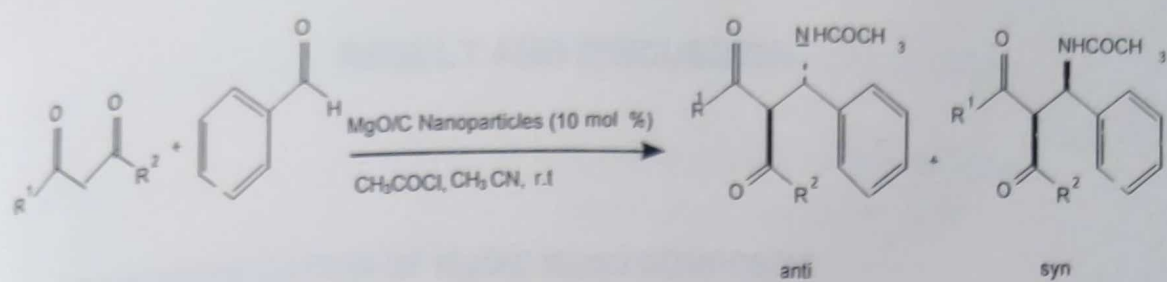


Fig 4.3.2 Synthesis of Alpha, Beta Acetamido Ketone

CHAPTER 5

RESULT AND DISCUSSION

5.1 CHARACTERIZATION OF MgO/C NANO COMPOSITE

5.1.1 FTIR Spectrum MgO/C Nano composite

The FTIR spectrum of Magnesium oxide/carbon nanocomposite are shown in Fig.5.1.1. The IR vibrational frequency obtained at 3849 cm^{-1} , 1760 cm^{-1} , 1417 cm^{-1} , 1518 cm^{-1} , and 738 cm^{-1} correspond to O-H stretching of adsorbable water molecules on magnesium oxide, C=O stretching, water and CO₂ molecule adsorption on MgO/C surface, O-H in plane bending of carboxyl group adsorbed on MgO/C and metal – oxygen bond Mg-O stretching respectively. The peak at 738 cm^{-1} is the characteristic peak for MgO/C nanocomposite.

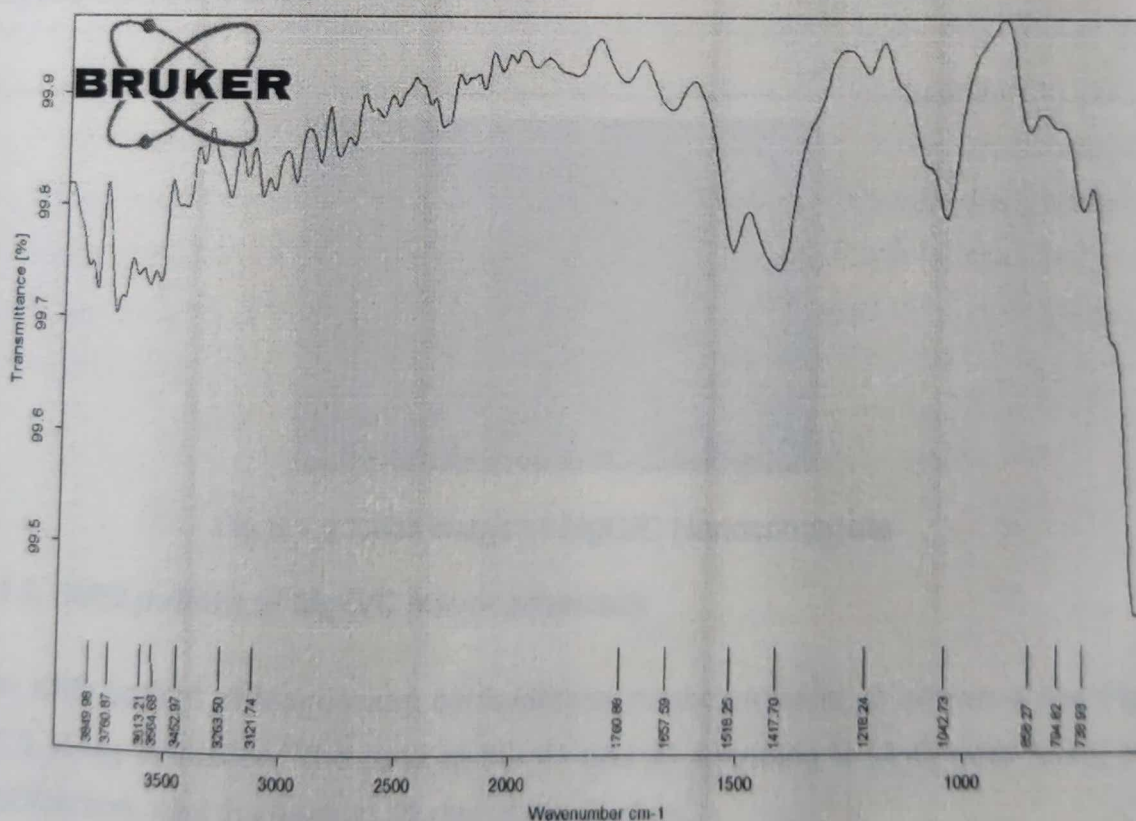


Fig 5.1.1 FTIR Spectrum of MgO/C Nanocomposite

Vibrational frequency

Functional group identification

3849 cm^{-1}

- O-H stretching of absorbable water molecule on magnesium oxide

1760 cm^{-1}

- C=O stretching

1417 cm^{-1}

- C-O

738 cm^{-1}

- Mg-O stretching

5.1.2 SEM of MgO/C Nanocomposite

The SEM image of Magnesium oxide/carbon nanocomposite is shown in the Fig. 5.1.2. the image explains that magnesium oxide/carbon nanocomposite obtained have spherical shape. The SEM image also shows the presence of agglomerates of MgO/C nanocomposite.

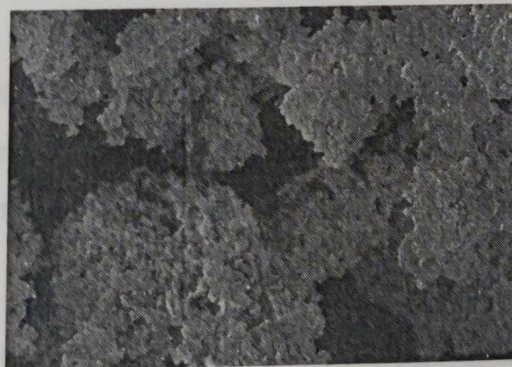


Fig 5.1.2 SEM image of MgO/C Nanocomposite

5.1.3 XRD pattern of MgO/C Nanocomposite

The XRD pattern of Magnesium oxide/carbon nanocomposite as shown in the Fig 5.1.3. X-ray diffraction. The peak at the 41 and 61 ascribing to MgO were found in MgO/carbon. And the peak at 29 describes Carbon.

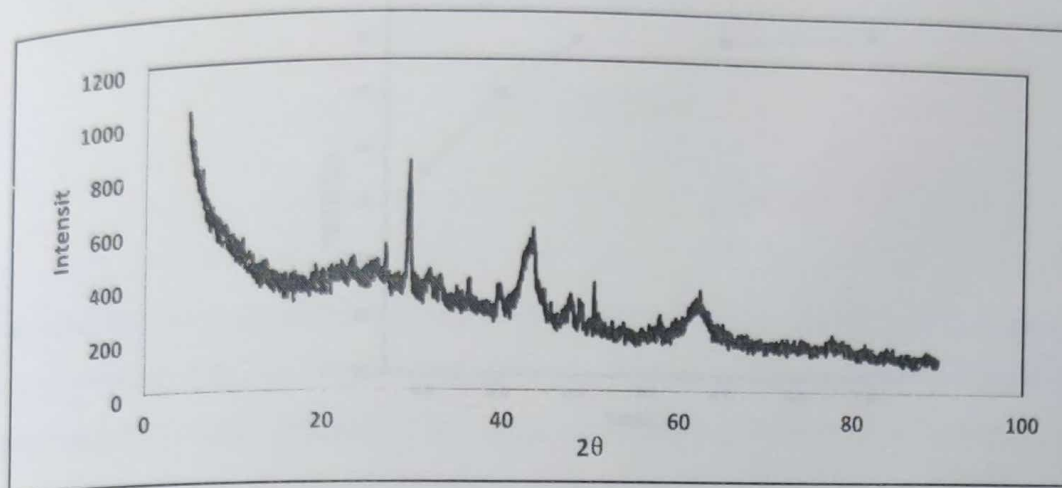


Fig 5.1.3 XRD pattern for MgO/C nanocomposite

5.1.4 Catalytic studies

In one-pot synthesis, four-component condensation of benzaldehyde, acetophenone, acetyl chloride and acetonitrile were carried out and MgO/C composites at 60°C. Acetonitrile acts as reactant and also as solvent. The optimized feed molar ratio of 1:1:2 (benzaldehyde: acetophenone: acetyl chloride), 5ml (acetonitrile) was maintained at 60°C for all the catalytic runs. The alpha, beta acetamido ketone conversion over the catalytic systems at various catalysts loading is shown in the figure 5.1.4. Without the use of catalysts, no observable conversion was obtained. The alpha, beta acetamido ketone conversion increases with increase in catalyst loading upto 30 mol%. Further increase in catalyst loading does not increase the rate of the reaction. Thus 30 mol% is considered to be the optimized catalyst loading.

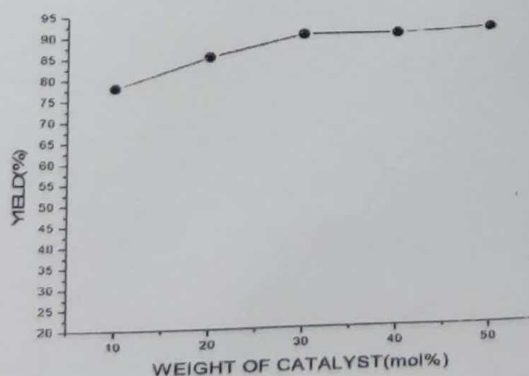


Fig 5.1.4. Effect of catalyst loading on alpha, beta acetamido ketone conversion over MgO/C composite.

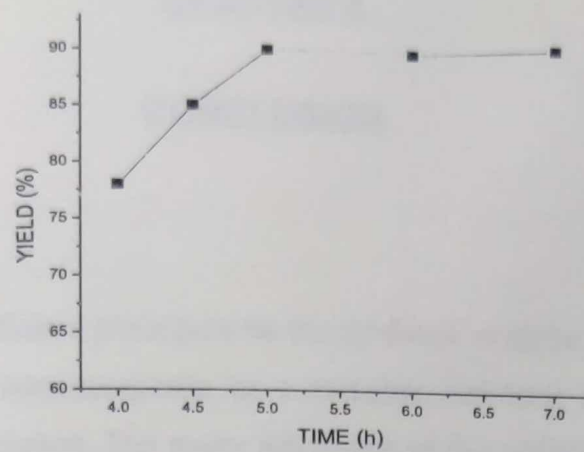


Fig 5.1.5 Effect of time on alpha, beta acetamido ketone conversion over MgO/C.

CHAPTER 6

CONCLUSION

We have reported an efficient procedure for the synthesis of alpha, beta acetamido ketones using MgO/C nanocomposite as a reusable, non-toxic and inexpensive heterogeneous nano catalyst. The major advantage of this method is the ease of work-up; i.e., the products can be isolated without chromatography. The method also offers some other advantage such as clean reaction, low loading of catalyst, high yields of products, short reaction times and use of various substrates, which make it a useful and attractive strategy for the synthesis of alpha, beta acetamido ketones.

REFERENCES

1. A Almonstasser, A Praveen and Azam. "Synthesis, Characterization and antibacterial activity of Magnesium oxide (MgO) nanoparticles". IOP Science 16-18 August 2018. (Vol 577).
2. Andrew L. Shannon, Anne Woolridge. "Medical Waste". 2011. Pages 329- 339.
3. Edmond Lam and John H.T. Luon. "Carbon materials as catalyst supports and catalysts in the transformation of biomass to fuel and chemicals". American chemical society. 25 August 2014. Pages 1-64.
4. Gaurav Sharma, Rajgovind Roni and Nakuleshwar Dut Jasuja. "Phyto assisted Synthesis of Magnesium oxide with *Swertia Chiravatia*". 2017.
5. J Jamie Sadhwani Alonso, Nenna El Korin, Melian-Martel B. Del RioGamero. "Removal of ciprofloxacin from seawater by reverse osmosis". Journal of Environment Management. July 2018. (Vol 217) Pages 337-345.
6. J Suresh, R Yuvakkumar, M Sundrarajan, Sun Ig Hong. "Green synthesis of magnesium oxide nanoparticles". Advanced Material Research. May 2014. (Vol – 2) Pages 141-144.
7. Javaid Akhtar, Nor Aishah Saidina Amin, Khurram Shahzad. "A review on removal of pharmaceuticals from water by adsorption". Desalination and water treatment. 25 June 2015. (Vol 57) Pages 12842-12860.
8. Jozsef Hari, Bela Pukanszy. "Nanocomposite: Preparation, Structure, and Properties". Applied Plastic Engineering Handbook. 2011. Pages 109-142.
9. K. Mageshwari, S.S Mali, R. Sathyamoorthy, P.S Patil. "Synthesis of MgO nanoparticles for effective photocatalytic application". Powder technology. November 2013. (Vol 249) Pages 456-462.
10. KR Nemade, SA Waghuley. "Synthesis of MgO nanoparticles by solvent mixed spray pyrolysis technique for optical investigation". International Journal of Metals. 2014.
11. L. Umaralikhan, M. Jamal Mohamed Jaffer. "Green synthesis of MgO nanoparticles and its antibacterial activity". Iran J Sci Technology Trans Sci 07 June 2016. (Vol - 42) Pages 477-485.

12. Lan She, Jing Li, Ying Wan, Xiangdong Yao, Bo Tu and Dongyuan Zhao. "Synthesis of ordered mesoporous MgO/carbon composites by a one-pot assembly of amphiphilic triblock copolymers". *Journal of Material Chemistry*. 08 November 2010.
13. Mohammad Moslem Imani and Mohsen safaei. "Optimized synthesis of Magnesium oxide Nanoparticles as Bactericidal Agents". *Journal of Nanotechnology*. 01 April 2019. (Vol 2019).
14. Omar Fawzi Suleiman Khasawneh, puganeshwary Palaniandy. "Occurrence and removal of pharmaceuticals in wastewater treatment plants". *Process Safety and Environmental Process*. June 2021. (Vol 150), Pages 532-556.
15. Palanisamy. G and Pazhanivel. T. "Green synthesis of MgO nanoparticles for antibacterial activity". *International Research Journal of Engineering and Technology*. September 2017. (Vol 04).
16. Renata Dobrucka. "Synthesis of MgO nanoparticles using *Artemisia abrotanum* herba extract and their antioxidant and photocatalytic properties". *Iranian journal of science and technology*. 02 September 2016. (Vol 42), Pages 547-555.
17. Rizwan Wahab, SG Ansari, Mushtaq A Dar, Young Soon Kim, Shik Shin. "Synthesis of magnesium oxide nanoparticles by sol-gel process". *Material science forum*. October 2007. (Vol 558-559), Pages 983- 986.
18. S. Abinaya, Helen P. Kavitha, M. Prakash, A. Muthukrishnaraj. "Green synthesis of magnesium oxide nanoparticles and its application". *Sustainable Chemistry and Pharma*. April 2021 (vol 19).
19. Sally Gaw, Kevin V, Thomas, and Thomas H. Hutchinson. "Sources, impacts and trends of pharmaceuticals in the marine and costal environment". *the royal society*. 19 November 2014.
20. Sang D. Kim, Jaeweon Cho, In S. Kim, Brette J. Vanderford, Shane A. Snyder. "Occurrence and removal of Pharmaceuticals and endocrine disruptors in South Korean surface, drinking and waste waters". *Water Research*. March 2007. (Vol 41) Pages 1013-1021.
21. Taimur Athar. "Synthesis of MgO nanoparticles via Sol-Gel method". *American scientific*. 2013. (Vol 2) Pages 493-496.
22. Vania Calisto, Catarina Í.A. Ferreira, Joao A.B.P. Oliveira, Marta Otero, Valdermar I. Esteves. "Adsorptive removal of pharmaceuticals from water by

- commercial and water-based carbons". *Journal of Environmental Management*. 1 April 2015. (Vol 152) Pages 83-90.
23. Wei-Tehjiang, Po-Hsiang Chang, Ya-Siang Wang, Yolin Tsai, JiinShuhjjean, Zhaohuuli. "Removal of ciprofloxacin from water Birnessite". *Journal of Hazardous Material*. 15 April 2013 (250-251). Pages 362-369.
24. Yi Yang, Yong Sik Ok, Ki-Hyun Kim, Eilhann E. Kwon, Yiu Fai Tsang. "Occurrence and removal of pharmaceuticals and personal care products (PPCPs) in drinking water and water/sweage water treatment plants". *Science of the total Environment*. 15 October 2017. Pages 303-320.
25. Yuchao Shao, Dong-Yang Zhao, Weijing Lu, Yuyang Long, Weicheng Zheng, Zhong-Ting Hu. "MgO/Carbon nanocomposites synthesized in molten salts for catalytic isomerization of glucose to fructose in aqueous media". *Green Chemical Engineering*. 26 December 2022. (Vol) Page 359-366.