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Purification of waste water resulted from oil Refining using Nanomaterials

A Thesis

Submitted to the College of Science/AL-Nahrain University as a partial
fulfillment of the requirements for the Degree of Master of Science in Chemistry

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بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

(قَالُوا سُبْحَانَكَ لا عِلْمَ لَنَا إِلا مَا عَلَّمْتَنَا إِنَّكَ أَنْتَ الْعَلِيمُ الْحَكِيمُ).

صَدَقَ اللهُ العلي العَظِيم

سورة البقرة الآية (٣٢)

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Dedication …

The spirit of the person in my life

My father's soul

And to whose feet under heaven

My mother

And to my delight, my smile

My sisters

And to all of my teachers who taught me to perform

My character

And to all, who were really brothers and sisters

My colleagues.

And finally to everyone who supported me at the University of Al-Mustansyriyah. I bow and infinite thanks and appreciation to my family, for their patience and understanding to complete my research.

Summary

Filtration of waste water accompanied with the oil from the well during the refining process, represents a real problem at some case since it may be the source of bacterial growth.

Here in this project, MgO nanoparticles has been prepared by sol- gel method to produce particles with sizes range (40nm), while Ag nanoparticles has been prepared by simple chemical method with sizes range (50.73nm).

The products were characterized by utilizing the apparatuses and techniques:

Atomic absorption spectroscopy, conductivity meter, (FTIR), X-Ray diffraction (XRD), Scanning Electron Microscopy (SEM) , Energy dispersive X-ray (EDX) , Thermo gravimetric analysis (TGA) technique.

The products utilized to coating membranes , then these coated membrane utilized to filter wastewater samples. The results showed the effectiveness of these nanoparticles in the wastewater filtration.

A part of resulting nanoparticles used for the purpose of examining its biological activity against bacteria. The results showed high effectiveness for these nanoparticles in killing bacteria.

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List of Abbreviations

AAS	Atomic absorption spectroscopy
AgNP	Sliver carbon nano tube
BOD	Biological oxygen demand
COD	Chemical oxygen demand
CMC	Critical micelle concentration
CWF	Ceramic water filter
CNT	Carbon nano tube
DNA	Deoxyribo nucleic acid
EDTA	Ethylen di amine trata acetic acid
EDX	Energy dispersive X-ray
LEDs	Light emitting diodes
MWCNT	Multe walled carbon nano tube
MEUF	Micellar enhanced ultra filtration
MB	Methylen blue
NF	Nano filtration
MWCNT	Multe walled carbon nano tube
RO	Reverse osmosis
ROS	Reactive oxygen species
SRB	Sulfate reducing bacteria
SDS	Sodium dodecyl sulfate
SWCNT	Single walled carbon nano tube
SEM	Scanning electron microscope
TFC	Thin-Film composite
TGA	Thermogravimetric analysis
TDS	Total dissolved solids
UV	Ultra violet
WWTM	Waste water treatment Method
WHO	World health organization

Chapter One

Introduction and Literature Review

1.1 Introduction

Traditional wastewater treatment process (WWTP) has been constructed to treat both local and industrial wastes to meet the effluent standards prior to discharge in the natural environment. The composition and volume of wastewater can likewise differ inside a similar area , from hour to hour, from day to day and from year to year. Contaminants in the wastewater can be characterized by their particle size as appeared in Table (1.1).

Table (1.1) Molecule measure run in sewage wastewater.

Particle size	***Dissolved***	***Colloidal***	***Suspended***	***Settleable***
(µm)	< 0.01	0.01-1.0	1-100	>100

The major pollutants found in wastewater are biodegradable organic components, volatile organic components, xenobiotics, metal ions, suspended solids, nutrients, for example phosphorous (P) and nitrogen (N), microbial pathogens and parasites. Additionally the approximate components of organic carbon found in sewage wastewater is: carbohydrates (11-18%), proteins (8-10%), free amino acids (0.5-1.5%), fatty acids (23-25%), dissolved organic acids (7-11%) and other organic compounds (25-28%). In any case, wastewater is complicated and needs detailed characterization of organic substances for an effective WWTP [1,2]. The standard techniques for evaluating organic substances are measured as biological oxygen demand (BOD), chemical oxygen demand (COD) and total organic carbon (TOC). (BOD) is the quantity of dissolved oxygen needed by microorganisms in wastewater to separate organic matter over a time of 5 days. (COD) indicates the amount of contaminants in the water that can be oxidized by a chemical oxidant. (TOC) is another measure to evaluate organic substances by combustion process.The inorganic compounds of wastewater for the most part comprise of nitrogen, phosphorous, chlorides, sulfates, bicarbonates and others. Specifically, nitrogen and phosphorous are considered important, as they are the fundamental nutrients that cause eutrophication in the recipient water if not treated appropriately.

1.2 Nanotechnology

The advantages of nanotechnology in water treatment application have been focused in three main areas: treatment and remediation, sensing detection and contamination control. It has yielded colossal advance in a many areas, for example, the manufacturing of electronics, broadcast communications and medicine. Nanotechnology also plays a major role to solve main issues of environment and water sectors[3].

1.3 Benefits of Nanotechnology in Water and Wastewater Treatment

1.3.1Water Treatment

Water purification utilizing nanofiltration technology or through adsorption and catalytic degradation processes was made conceivable by the advances accomplished and secrets revealed in the quantum world. Around the world, the requirement for clean water is increasing due to population raise, drought and pollution traditional water sources. The development of new technologies to raise the accessibility of clean water initiated 50 years ago (1960s) with the foundation of three membrane separation processes (Table 1.2): reverse osmosis (RO), ultrafiltration (UF) and microfiltration (MF) [4]. Nanofiltration membranes (Loose RO) were developed an intermediate filtration material amongst ultrafiltration and reverse osmosis [5]. Membrane processes utilizing different sorts of membrane are ending up increasingly popular for the production of drinking water from seawater, brackish water, wastewater, surface water and groundwater .

Table (1.2) Membrane type and characteristics.

Membrane type	***Pore size (nm)***	***Pressure (bar)***	***Product water***
Reverse osmosis	*>0.6*	*30-70*	***Pure water (PW)***
Nanofiltration	0.6-5	*10-0*	***(PW) and low molecular solutes***
Ultrafiltration	*5-50*	*0.5-10*	***All above and macro-molecules***
Microfiltration	*50-500*	*0.5-2*	***All above and collides***

The effect of nanotechnology on the advancement of instruments and methods for water treatment will be more pronounced soon. As scarcity of natural water threatens the progression and the social security of numerous communities around the world, it is normal that the solution will emerge from the utilization of nanoparticles to make water reusing, seawater desalination and water remediation more proficient and cost effective. For example, the utilization of nanofiltration membranes for treating water in rustic places of South Africa to provide drinking water was depicted by *Simith et al.* [6].The benefits of utilizing nanofiltration depended in the direct humanitarian advantage from utilizing nanotechnology and in the promotion of economical viabilities in country groups. Along these lines, the production of nanostructures, nanocomposites and adjusted nanostructures for water remediation will raise due to the requirement for producing clean water in quick and low energy utilization ways. Nanotechnology should to be viewed as the tool to ensure the manageability of social communities in different places and conditions. This is possible through the use of advanced filtration nanomaterials that enable desalination of seawater, reusing of polluted water and the reuse of wastewater [7].

1.3.2 Water Purification

Wastewater is any water that has been adversely polluted by organic contaminants, bacteria and microorganisms, industrial effluent or any components that weakened its initial quality. It can be sub-divided into:

1) Municipal wastewater (liquid waste released by local residences and commercial properties)

2) Industrial wastewater (liquid waste released by industrial and agricultural activities).

A portion of the factors that might influence the composition of wastewater are: land utilizes, groundwater levels, and the level of separation between stormy water and sanitary wastes. The composition of local wastewater is generally less variable than of industrial wastewater, the last being highly influenced by the kind of industrial activity required in the release of effluent water. In general, the organic composition of wastewater is assessed to comprise of proteins (50%), carbohydrates (0%), fats and oils (10%), and trace quantity (e.g. µg/L or less) of priority contaminations, surfactants, and emerging contaminants [8].

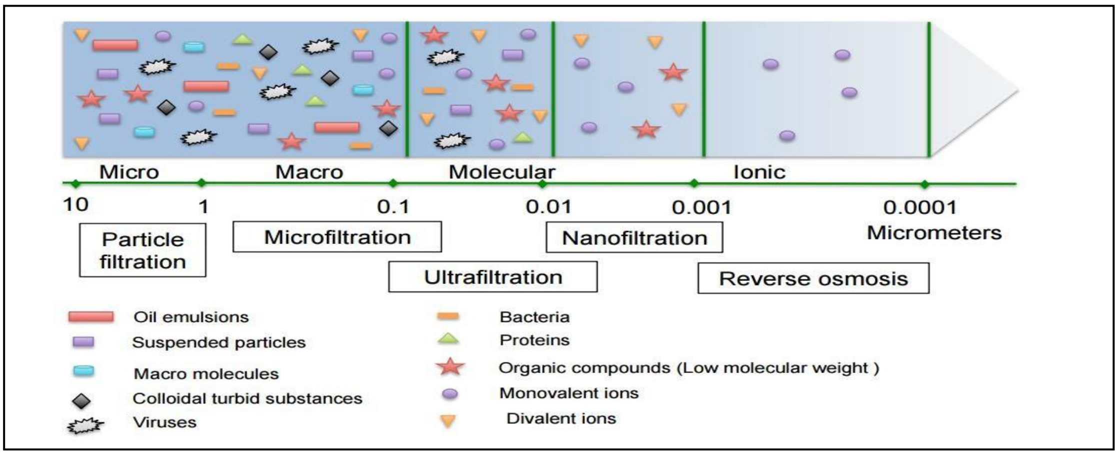


Figure (1.1) Size dependent filtration methods (scale: Micro particle range, Macro molecular range, Molecular range, Ionic range) [9].

1.4 Traditional process of disinfecting bacteria from waste water

1. Chlorination is the most common form of disinfection. Chlorine is a strong oxidant, and rapidly kills many microorganisms, especially bacteria. Because chlorine is a toxic gas, it can also be dangerous to sanitation workers. Chlorine based compounds like choloramine are often used. Although chlorine is very effective against bacteria, it is not as effective against the cysts formed by protozoans such as (*giardia lamblia* and *cryptosporidium*). Chlorine can sometimes leave residual by products in water.
2. Ozone is an unstable molecule that readily gives up one atom of oxygen providing a powerful oxidizing agent. This agent is toxic to most water borne organisms. Ozone is widely used in Europe, and is an effective method to kill cysts formed by protozoans. It also works well against almost all other pathogens.
3. Ultraviolet Light is very effective at inactivating protozoan cysts, and will also kill bacteria and viruses. However, it is not as effective in cloudy water. It is sometimes used in concert with chlorination [10].

1.5 Nanofiltration

Nanofiltration is a process based on the utilization of nano-pores present in the membrane utilized for filtering water samples with pollutants of various size ranges. Two fundamental sorts of nano-membranes are nanostructured filters and nano-reactive membranes [11]. Pre-treatment of the wastewater is one of the important regards to avoid fouling and enhancing membrane performance. Additionally membrane performance can likewise be influenced by oxidizing agents, for example, chlorine. Normally utilized films in wastewater treatment are ceramic and polymer supported membrane. A portion of the normal shapes found in the market are winding, tubular, empty fiber and edge films modules [12].

1.6 Membrane Filtration

Membrane filtration is a pressure driven process in which membrane goes about as selective barriers to limit the passage of contaminations, for example, organics, nutrients, turbidity, microorganisms, inorganic metal ions and other oxygen contaminants, and enables generally clear water to pass through [13]. With technical progresses and the constantly expanding stringency of water quality criteria, membrane processes are becoming a more great solution for the test of quality water, and water reuse [14].

The membrane process has been ordered into four general classifications depend on their pore sizes as: microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) membranes.

1) Reverse osmosis (RO): Liquid-phase pressure driven separation process in which connected trans-membrane pressure causes eclectic movement of solvent against its osmotic pressure difference.
2) Nanofiltration (NF): Pressure-driven membrane based separation process in which particles and dissolved macromolecules littler than 2 nm are rejected.
3) Ultrafiltration (UF): A separation process whereby an solution containing a solute of sub-atomic size altogether more prominent than that of the solvent particle is expelled from the solvent by the application of a hydraulic pressure weight which drives just the solvent to stream through an appropriate membrane, as a rule having a pore size in the range of 2–100 nm .

4) Microfiltration (MF): Pressure-driven membrane based separation process in which particles and dissolved macromolecules bigger than 100 nm are rejected.

1.7 Types of Nanofiltration Membranes

1.7.1 Ceramic membranes:

Have high mechanical quality, thermal steadiness, chemical compatibility, flux, long operational life and are proficient in cross stream filtration. For instance, an alumoxane NPs were utilized as a part of preparation of alumina membranes for the evacuation of manufactured dyes.These were more particular for the evacuation of dyes, for example, direct red 81, direct blue 71 and direct yellow 71 as announced before [15]. One of the industrially available ceramic membranes is membralox with a pore size of 20 nm to 0.2 μm and is for the most part made out of materials, for example, alumina, titanium, and zirconium. disadvantage involve capital and operational cost.

1.7.2 Polymeric membranes:

Have particular qualities, for example, high flux, resistance to UV, chlorine and ozone. Polymeric membranes consist of cross-linked polyamide, cellulose acetate, polysulfone materials and are utilized as a part of the treatment of wastewater. Polyamines having primary amines and carboxylic acids in their external surface have demonstrated enhanced metal ion binding with a chelating mechanism [16]. However some of the disadvantage are frequent fouling upon contact with organic pollutants , consequently leading to decreased flux rate and poor chemical stability.

1.7.3 Carbon nanotubes (CNT):

Are comprised of a hexagonal lattice of carbon and fullerene molecules. There are two sorts namely single (SWCNT) and multi walled carbon nanotubes

(MWCNT) based on the principle of hybridized carbon atom layers on their external surface. The size scope of SWCNT is between 0.3 to 3 nm, though MWCNT has diameters up to 100 nm [17]. For example, CNT's exhibited the evacuation of organic contaminants, for example, benzene, toluene, xylene and ethylbenzene was higher contrasted with the granular activated carbon. Different reviews have likewise revealed the utilization of functionalized carbon nanotubes for the evacuation of contaminants, for example, p-nitrophenol, heavy metals, trihalomethanes and dimethylbenzene [3]. *Zhang et al.* [18] reported that (MWCNT) can possibly adsorb 85% bisphenol AP from wastewater and can be reused around (8) times without influencing in efficiency. However synthesis and maintenance in utilizing carbon nanotubes for wastewater treatment is as yet a test.

1.8 Membranes functionalized with nanoparticles

The possibility of functionalizing membranes with nanoparticles is fairly new. Nevertheless a few number (principally lab-scale) tests have been performed so far with various membrane materials and nanoparticles. For example nano-TiO_2 particles can be coordinated into the polyamide top layer of the membrane to reduction membrane fouling and to raise the flux due to the super hydrophilic properties of nano-TiO_2. Be that as it may, a portion of the exploration results are contradictory [19] and the real use of such films is still in its outset. It is thus not surprising that the terminology for these membranes likewise is not consistent. Many different terms are right now being utilized, for example, nano-activated, nano-enhanced, nanoparticles-based, and nano-functionalized membranes. To avoid any ambiguity, the terminology should be standardized.

Nano-enhanced membranes (NEMs) go for joining nanotechnology with membrane technology to enhance membrane properties and thus increase their performance. There are various directions of research a few researchers are attempting to coordinate carbon nanotubes as pores in membranes [20,21]. These membranes could be utilized rather than RO membranes for the production of ultra-pure water with the benefit of an expanded flux [22]. Different methodologies include the design membranes from a CNT-mesh [20,23] or the surface functionalization of membranes with various nanoparticle sorts, for example, TiO_2, Ag, aluminum oxides, silica, CNT, zirconia or iron oxides nanoparticles to accomplish expanded flux as well as to diminish fouling, which is as yet the main test in membrane technology [19,24]. In any case, a many change in applying TiO_2 to polymeric membrane materials is that nano-TiO_2 in the surface layer not just degardes organic substances in the water phase additionally the organic membrane material.

To stay away from this issue *Keuter et al.* [25] applied nano-TiO_2 and nano-TiO_2/Ag nanocomposites to metallic microsieves. These microsieves have – because of their production process – a practically uniform pore and pore size distribution. Coating by sol–gel or chemical vapour deposition (CVD) processes offers the chance to accomplish antifouling properties by nano-TiO_2 induced photocatalytic degradation processes. Facilitate efforts are in progress to apply these functionalized membranes for direct degradation of organic components (e.g. pharmaceutical residues) in combination with UV-LEDs. TiO_2, ZrO_2 what's more, other crystalline nanoparticles may likewise be utilized as active layers of ceramic membranes. Such membranes would likewise – independently of their pore size – be arranged as nano-enhanced membrane.

1.9 Separation mechanisms in nanofiltration

Since nanofiltration membrane shows properties between those of ultrafiltration (UF) and reverse osmosis (RO), both charge and size of particle play important part in nanofiltration rejection mechanism. For the colloids and extensive molecules, physical sieving would be the predominant rejection mechanism though for the ions and lower molecular weight substances, solution diffusion mechanism and charge impact of membrane assume the significant part in separation method. *Macoun et al.* [26] displayed the nanofiltration rejection mechanisms into taking after five stage:

1) Wetted surface – water associates with the membrane through hydrogen bonding and the molecules which form the hydrogen bonding with the membrane can be transported.
2) Preferential sorption/Capillary rejection–membrane is heterogeneous and microporous, and electrostatic repulsion happens because of various electrostatic constants of solution and membrane.
3) Solution diffusion–membrane is homogeneous and non-porous, solute and solvent dissolve in the active layer of the membrane and the transport of the solvent occurs due to the diffusion through the layer.
4) Charged capillary– electric double layer in the pores decides determines rejection. Ions of same charge as that of membrane are attracted in and counter-ions are rejected because of the streaming potential.
5) Finely porous–membranes a dense material punctured by pores. Transport is determined by dividing amongst mass and pore liquid.

The properties of nanofiltration membranes lies between the non-pores reverse osmosis membranes (where the rejection is because of solution-diffusion system) and porous ultrafiltration membranes (where the rejection is by size rejection and electrostatic charge impacts). Subsequently, the rejection of uncharged particles is commanded by size exclusion, while that of ionic species is impacted by both size exclusion and electrostatic interactions. Electrostatic characterisation of nanofiltration membranes have been known as playing an important part in rejection anions, in particular, negative zeta potential on the membrane surface varies with various pH and concentration of an electrolyte solution [27].

1.10 Nanoparticles in water treatments

Sorbents are generally utilized as separation medium in water filtration to expel inorganic and organic contaminants from polluted water. Nanoparticles have two key properties that make them especially attractive as sorbents. To start with, on a mass basis, they have much larger surface areas than bulk particles. Second, they can likewise be functionalized with different compound groups to raise their affinity towards target components. Several research groups are exploiting the unique suitabilities of nanoparticles to grow high capability and selective sorbents for metal ions and anions.

Nanoparticles are characterized as small particles sized between 1 to 100 nanometers in no less than one dimension. As of now there are more than 1,317 nanotechnology-based customer products as per an examination by Nanotech-project.com [28,29].

1.10.1 AgNP synthesis

AgNP can be synthesized by different techniques including chemical the electrochemical methods, and photochemical reduction. Among all the synthetic methods, chemical reduction is most commonly utilized. However, poisonous component, for example, borohydride are typically included. Studies have concentrated on ―green synthesis approaches to avoid utilizing dangerous materials. The toluene method is broadly applied for AgNP synthesis. Environmentally begin monosaccharides and polysaccharides are utilized to lessen the $Ag(NH_3)_2^+$ complex formed by reacting $AgNO_3$ with ammonia to AgNP. Past reviews have produced AgNP with sizes running from 50-200 nm furthermore, silver hydrosols running from 20-50 nm [30-32].

For antimicrobial purposes, formation of aggregates can diminish the antimicrobial capacity of AgNP [33-35]. Stabilizers are incorporated in the AgNP producing process to guarantee their stability in a gaseous solutions. Adsorption of the stabilizing molecules onto the nanoparticle surface relies on the molecular weight, ionization, and charge density of the stabilizing molecules [36]. Stabilizing layers can expand the electrostatic and steric repulsion amongst nanoparticles and in this way improve the stability of the nanosuspension [37].

1.10.2 Environmental fate and antimicrobial properties of AgNP in different water chemistry conditions

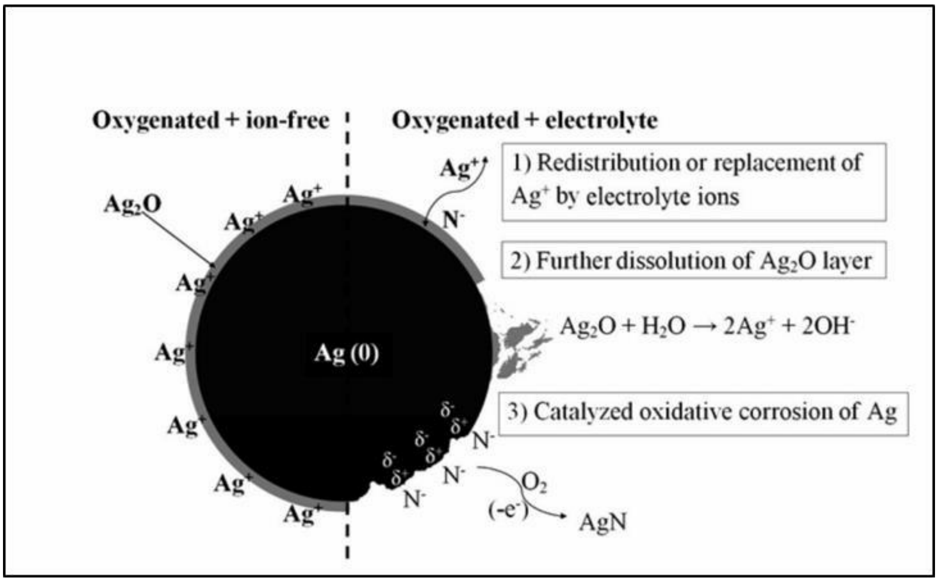
The utilization of AgNP in a wide assortment of consumer products will definitely lead to release of the nanoparticles into natural water, which are the last receptacles [38]. Subsequently, knowing the destiny and reactivity of AgNP under environmentally relevant conditions is basic to slope possible negative effects on microorganisms generally existed in aquatic ecosystems.

1.10.3 Aggregation of AgNP in different water chemistry conditions

Past reviews have demonstrated that different water chemistry conditions influence the toxicity of AgNP on microorganism groups [39]. It is broadly accepted that the aggregation of AgNP takes after the Derjaguin-Landau-Verwey-Overbeek (DLVO) hypothesis [40] which combines the impacts of the van der Waals attraction force and the electrostatic repulsion force made by the double layer of counterions [34,41]. In aqueous solution, AgNP are electrostatically stabilized when the energy barrier is formed due to the electrostatic repulsion force is in excess of the van der Waals attraction force. AgNP are steady under this condition. Be that as it may, when electrolytes are introduced into the system, the counterions in the aqueous solution neutralize the surface charges and disrupt the energy barrier, leading to aggregation [34, 41,42]. When addition of the electrolyte solution results complete expulsion of the energy barrier, quick aggregation happens and the cluster size raises regardless of the electrolyte concentration [43].

1.10.4 Dissolution of AgNPs in different water chemistry conditions

Dissolution of AgNP is an imperative parameter in determine their environmental fate. Late reviews have attempted to evaluate the dissolution of AgNP in various water modelling. For aqueous solution with various pHs, *Liu* and *Hurt* [44] and *Elzey et al.* [45] have found that lower pH can improve the dissolution of AgNPs. Figure (1.2) shows the dissolution of AgNP in the presence of DI and electrolyte solutions.



Figure(1.2):Schematic illustration of dissolution of the oxide layer coated AgNP [46].

In various ecological aqueous conditions, *Liu and Hurt* [44] explored the dissolution kinetics of AgNP and revealed that the quantity dissolved silver ions from AgNP coated with sodium citrate (average particle size: 8±1.6 nm; initial concentration: 0.05 mg/L) in diluted synthetic seawater is more than that in seawater (ionic strength: 0.7 M).

1.10.5 Antimicrobial property of AgNP in different water chemistry conditions

Antimicrobial capacity is a notable property of AgNP. AgNP can inactivate a wide spectrum of microorganisms. Past reviews have proposed three mechanisms of the antimicrobial activities of AgNP Figure (1.3):

1) Could connect to cell membrane and disrupt the permeability and respiration functions of the cell and thus kill the cells [34,47].
2) Reactive oxygen species (ROS) can be generated on the surface of nanoparticles and cause damage of DNA by exerting oxidative stress [48].
3) Silver ions released from AgNP can also cause disruption of ATP generation and DNA replication [49].

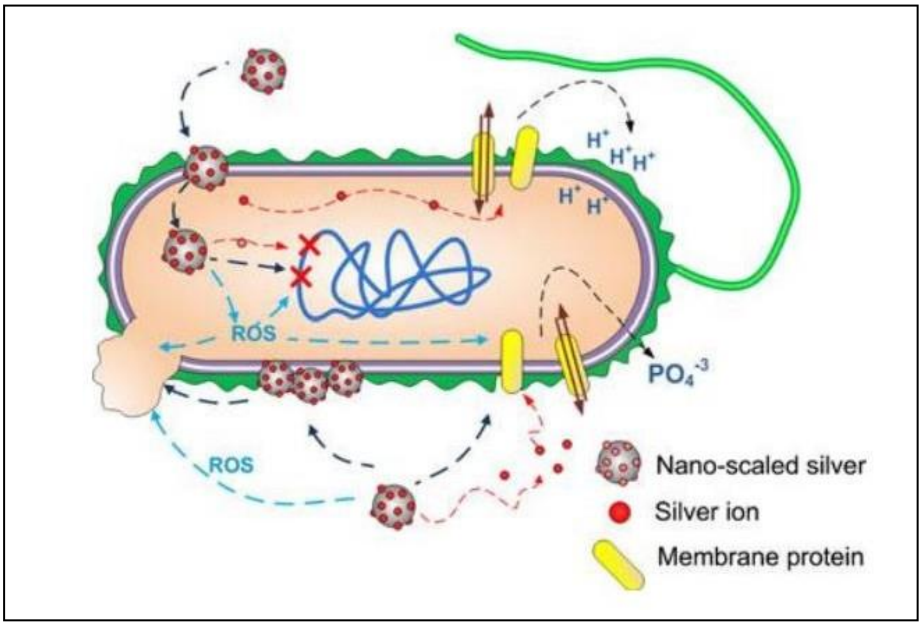


Figure (1.3) Mechanisms of interaction between AgNP and bacterial cells [46].

Past reviews have assessed the poisonous quality of AgNP toward various microorganisms in aquatic systems. *Gao et al.* [39] have reported that the lethal concentration (LC5) (µg/L) of AgNP against *Escherichia coli (E. coli)* and *Ceriodaphnia dubia (C. dubia)* are under 112.1 and 6.18 µg/L, separately.

Examines on the toxicity of AgNP against different microorganisms, for example, *Staphylococcus aureus* [50], *Leuconostoc mesenteroides* [51], *Bacillus subtilis* [28], and *Pseudomonas aeruginosa* [29] were likewise revealed. Late reviews likewise focus on viruses, a smaller microorganism, as viruses are responsible for a wide range of illness in bacteria, plants, and animals and they play critical part in aquatic food webs as active constituents of the microbial loop. A current review detailed that AgNP (particle size 21 nm) couldn't inactivate an MS2 bacteriophage in a phosphate buffer solution (PBS) even at their most highest concentration (5 mg/L) [30]. Another review demonstrates that inhibitor concentration (IC50) of AgNP on various strains of HIV-1 virus ranged from 0.19-0.91 mg/mL. However, more research is required determine the antiviral activity of AgNP in environmental relevant conditions and their antiviral mechanisms. In environmental conditions, the antimicrobial property of AgNP can be influenced by their stability in various water chemistry conditions. Environmental variables, for example, the existence of O_2, complexing agents (for example, Cl^{-}thus SO^{2-},etc), and ionic strength impact the particle size, surface chemistry, and silver dissolution of AgNP [31].

Under different oxidation conditions, *Lok et al.* [32] demonstrated that *E. coli* colony formation was not influenced by a treatment of 9.2 nm AgNP synthesized under reducing conditions contrasted with the control groups (*E. coli* without AgNP treatment). However, after air saturation, aquoeus solutions containing AgNP with O_2 for 30 min demonstrated a strong antimicrobial effect on *E. coli*.

Xiu et al. [33] showed that the toxicity of AgNP was 20 times less toxicity to *E. coli* than silver ions (EC50: 2.0±0.07 versus 0.10±0.01mg/L) and the toxicity of AgNP expanded 2.3-fold after exposure to air for 0.5 h. In these reviews, the existence of a soluble Ag_2O layer was the way to the antimicrobial property of silver.

Many reviews have explored the antimicrobial action of AgNP in various environmentally applicable conditions. *Jin et al.* [34] researched the antibacterial properties of AgNP in various synthetic electrolyte solutions and illustrated that the antibacterial activity of AgNP was much lower than with silver ions when compared on the basis of total mass of AgNP added across all water conditions. Notwithstanding, bacterial inactivation likewise relied on upon the bacteria cell sort and in addition the hardness and alkalinity of the synthetic media. Past reviews have assessed the poisonous quality of AgNP toward various microorganisms in aquatic systems.

1.11 Application of AgNP in low cost drinking water purification systems

As of now, WHO/UNICEF evaluated that 783 million individuals on the planet don't approach safe drinking water [52]. *Boschi-Pinto et al.* [53] detailed 1.87 million youth passing are because of water-borne infections. Traditional water treatment and delivery methodologies are viewed as unfeasible in these under developed areas because they require high capital investment, a high cost of support, a high quantity water source, and these need clients to pay for the treated water. Individuals need to gather their own particular water outside their homes and after that store the water in the house unit because of the absence of water supply, and pollutions could happen amid the water collection, transport, and storage, which cause a high possibility of water-borne sickness disease [54]. A point of-utilization (POU) ceramic water filter (CWF) gives a choice to purify the water. A ceramic water filter (CWF) is a basic instrument that can remove water-borne pathogens. Right now, CWFs are made by pressing and firing a mixture of mud and a burnable organic substance, for example, flour, rice husks, or sawdust before treatment with AgNP. The filter is formed utilizing a filter press, after which it is air-dried and let go in a furnace. This forms the ceramic material and burns off the sawdust, flour, or rice husk in the filters, making it permeable and penetrable to water. CWFs are detailed for as successful in evacuating over 99% of protozoa and 90-99.99% of bacteria from drinking water [55]. In any case, a high expulsion of infections is not accomplished. AgNP and silver nitrate ($AgNO_3$, Ag^+) are added to filters at all CWF processing plants to accomplish 12 higher pathogen expulsion because of their antimicrobial properties [56,57]. The silver solutions are applied to CWF either by brushing or dipping. It was accounted for that 83% of CWF production lines apply AgNP and 17% utilize Ag^+. The concentration of silver applied at CWF factories varies. Revealed quantities of AgNP applied on CWFs ranges from 32 to 96 mg per CWF [58].

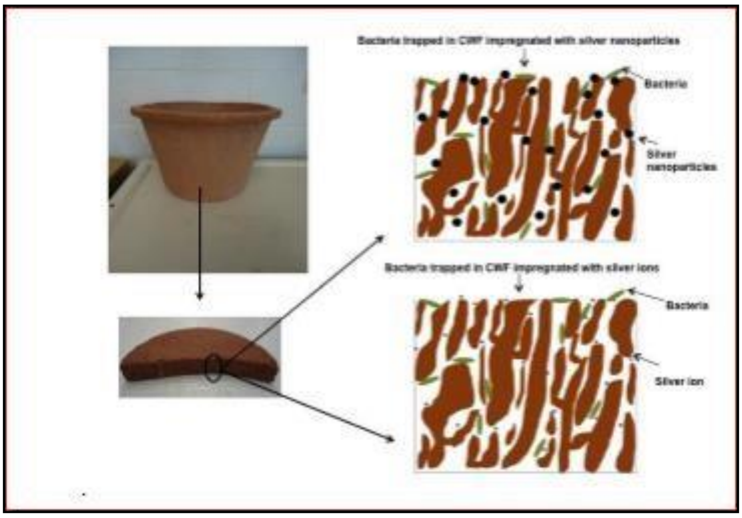


Figure (1.4) shows the bacteria trapped inside of CWFs coated with AgNP or Ag^+[46].

Two mechanisms of microorganism disinfection by CWFs were recommended:

1) CWFs can expel microorganisms by size exclusion or adsorption.
2) AgNP or Ag^+ inside of CWFs can inactivate pathogens [59].

1.12 Nano-materials as photocatalysts

Nanoparticle photocatalytic reactions depend on interaction of light energy with metallic nano-particles and are of great interest because of their expansive and high photocatalytic actions for different pollutants [60].Typically these photocatalysts are included semiconductor metals that can degrade assortment of persistent organic contaminants in wastewater, for example, dyes, cleansers, pesticides and volatile organic compound [61]. Besides, semiconductor nano-catalysts are likewise highly efficient for degradation of halogenated and non-halogenated organic components, pharmaceuticals and personal care products (PCPPs) and furthermore heavy metals in particular circumstance. Semiconductor nano-materials are required a mild operation conditions and very effective even at a little concentration.

The basic mechanism of the working of photocatalysis depends on the photo-excitation of electron in the catalyst . The radiation with light (UV in case of TiO_2) produces gaps (h^+) and excited electrons (e^-) in the conduction band. In a aqueous media, the gaps (h^+) are caught by water materials (H_2O) and produce hydroxyl radicals (^-OH) The radicals are unpredictable and powerful oxidization agents. These hydroxyl radicals on reaction oxidize the organic contaminants into water and gaseous degradation products [60].

Among different nanophotocatalysts developed up till now, TiO_2 is a one of the most broadly applied in photocatalysis because of its high reactivity under ultra violet light (k< 390 nm) and chemical stability [58]. Additionally, ZnO has likewise been broadly contemplated for its photocatalytic activity, as it contains wide band hole simply like TiO_2 [61].Various reviews have demonstrated the photocatalytic action of different synthesized catalysts. Their efficiency relies on upon various elements for example, band hole energy, molecule size, dosage, contaminants fixation and pH .For example, *Hayat et al.* [62] found that the photocatalytic degradation efficiency of ZnO diminished with high calcination temperature that raises the molecule size because of agglomeration. (CdS) is additionally an outstanding semiconductor having band hole of 2.42 eV and can be worked at wavelength <495 nm.

(CdS) nanoparticles have been attracted in big interest as photocatalyst for treatment of industrial dyes in wastewater [63].

Be that as it may, the previously mentioned catalysts are outstanding for their photocatalytic activity yet they are active just under ultra violet radiations (l <387 nm). This is because of the wide band hole vitality i.e. 3.2 eV as in should case of TiO_2. In this manner, encourage adjustments for the catalysts have been studied to expand their activities under visible light source (sunlight) for degradation of organic contaminations [64].

1.12.1 Magnesium Oxid Nanoparticles as photocatalyst

Lately, broad consideration has been paid all globally for the photocatalytic degradation of toxic organic contaminants. Particularly, the colored dye effluents produced from material and paper industries are extremely dangerous and non-biodegradable. 60–70 % of the dye effluents belonging to azo dyes make extreme organic contamination by discharging toxic and potential cancer-causing substances into the oceanic system [65]. Because of the complex aromatic structures and stability of these dyes, ordinary biological treatment process at times costly, in efficient what's more, non-destructive as a contaminants degrader [66]. The utilization of metal oxides as photocatalyst has demonstrated great utility in the complete mineralization of different environment toxins, for example, dyes, cleansers and volatile organic components [67]. Throughout the years, various metal oxides in particular TiO_2, ZnO,WO_3, Fe_2O_3 and CuO,etc., were utilized as dynamic photocatalyst materials for the degradation of organic contaminants in water and air.

Magnesium oxide (MgO) as a versatile oxide material with assorted properties like large band hole, amazing thermodynamical stability, low dielectric constant and low refractive index finds broad applications in catalysis, ceramics, dangerous waste remediation, antibacterial materials, and as an added substance in refractory, paint and superconductor items [68]. To date, photodegradation of organic dyes utilizing MgO nanoparticles is hardly revealed [69,70]. Consequently, from the perspective of environmental concern it is essential to concentrate the current research effort on the preparation of MgO nanoparticles for the degradation of organic contaminations. Up to this point, MgO nanostructures were synthesized by degradation out of $Mg(OH)_2$ or by decomposition of different magnesium precursors utilizing thermal evaporation , flame spray pyrolysis, sol–gel , compound vapour deposition , hydrothermal , and surfactant techniques [71]. The photocatalytic performance of the synthesized MgO nanoparticles was assessed by checking the photo-degradation of methyl orange (MO) and methylene blue (MB) dyes under UV light irradiation.

1.12.2 Doping/modification of photocatalysts

The use of visible light in photocatalytic treatment of wastewater is contemporary in research interest. To achieve this goal, the nano-material/semiconductor requires some modification to decrease the band gap energy form UV to visible region. There are number of available studies evaluating photocatalytic activity of modified nano-catalyst under visible light. The general methods used for modification of the catalyst include dye sensitization, doping metal impurities, hybrid nano-particles or composites using narrow band-gap semiconductors, or anions. The new metals and anions in the composite create a narrow band gap also called as impurity energy levels which upon exposure to visible light conducts electron into semiconductor for initiation of catalytic reaction. ZnO and TiO_2 nano-materials have wide band gap of 3.2

eV and have been extensively investigated for their photocatalytic activity. However, in solar spectrum both catalysts can only absorb a small portion of the UV region, which decrease their efficiency. However, modification in catalyst by loading metals on its surface can solve this problem. The modified composite material decreases the band gap energy and subsequently transfers the exited electron to semiconductor under illumination of solar radiation. Furthermore, not all conductive metals are effective for doping to improve photocatalaytic activity, e.g. Pt and Ru are ineffective for doping, while other metals such as Au, Ag and Pd showed excellent photocatalytic activities. During recent years, various doped nano-catalysts have been developed such as ZnO:Co, Ni, ZnS:Mn, ZnS:Cu, CdS:Eu, CdS:Mn, ZnSe:Mn, ZnS:Pb, and Cu. There are many dopants such as Cr, Si, Co, Mg, Mn, Fe, Al, In and Ga mare used having capability to enhance the surface area of metal oxide nano-structure. Among various dopants, anions such as nitrogen are also considered as most feasible and cost effective for industrial application [72].

1.13 Literature Review

(Suresh J. et al. in 2015); utilized that Nephelium lappaceum L. peels was viably utilized for the synthesis of magnesium oxide nanoparticles as a naturalist ligation agent. The X-ray Diffractometer (XRD) and Scanning electron microscopy (SEM) uncovered that the crystallinity and spherical morphology of the biosynthesized nanoparticles. The size of the particles were observed to be 60-70 nm as complemented from XRD and SEM analysis . The synthesized magnesium oxide powders measured by particle size analyzer (PSA) which was around 100 nm. The succeeded development of magnesium oxide nanoparticles was assured utilizing XRD, SEM-EDX and PSA examination [73].

(Rao V. et al., in 2014); detailed that the Metal oxide nanomaterials are substantial and magnificent materials, due to their distinctive properties like; chemical stability , high photograph catalytic activity, high electric permittivity and, non-poisonous nature. So they were utilized as a part of different applications like optical, electrical, electronic, disinfectant, antibacterial, environmental, semi-conductors and catalytic instruments. The work concentrated on to synthesis of Magnesium oxide (MgO) nanoparticles and its applications in the field of environments . These nanoparticles were prepared by basic appropriate chemical method like substance co-precipitation utilizing magnesium nitrate as core precursor . The synthesized metal oxide nanoparticles have been characterized by X-ray Diffractometer (XRD), Particle Size Analyzer (PSA), Scanning Electron Microscope (SEM) and Thermo Gravimetric and Differential Thermal Analyzer (TG-DTA) for thermal crystallite size, normal molecule size, morphology and warm stability individually [74].

(Sathyamoorthy R., et al., in 2013); prepared MgO nanoparticles by template-free reflux intensification approach. XRD and FTIR assured the forming of single phase MgO displaying cubic crystal structure. SEM and TEM examination assured the forming of MgO nano-flakes. The optical band hole of MgO nanoparticles evaluated from UV DRS was in the range 5.40–5.45 eV. PL spectra of MgO nanoparticles exhibited visible emission because of the forming of disorders in the band hole region of MgO. Synthesized MgO nanoparticles showed exhibited appreciable photocatalytic activity for the degradation of methyl orange and methylene blue colors under UV light irradiation. Results exhibited that the origin of photocatalytic activity in MgO arises because of the presence of high concentration of local disorders on the surface of MgO [75].

(Camtakan Z. et al., 2012); prepared MgO nanoparticles by the hydrothermal technique utilizing $MgCl_2$ and NaOH as procedures . The outcomes uncovered that the prepared MgO nanoparticles had an average diameter of around 24 nm [76].

(Sundrarajan M. et al., 2012); synthesized MgO nanoparticles by the hydrothermal technique utilizing magnesium nitrate and sodium hydroxide as precursors and dissolvable starch as a balancing out agent [77].

(Krishnamoorthy K. et al., 2012); assessed the antibacterial activity of MgO nanoparticles against the gram-negative bacteria *E. coli* and *Pseudomonas aeruginosa (P. aeruginosa),* and in addition the gram positive bacterium *S. aureus.* MgO nanoparticles showed antibacterial activity with MIC of 500 µg/mL *against E. coli* and 1000 µg/mL for *P.aeruginosa* and *S. aureus*. It was recommended that the system of the antibacterial mechanism of MgO nanoparticles might be lipid peroxidation and reactive oxygen species (ROS)because of the existence of defects of oxygen vacancy at the surface of the nanoparticles [78].

(An Y. et al., 2011); found that high MgO nanoparticle concentration resulted in greater bacterial inactivation. Many reports have demonstrated that MgO nanoparticles have better activity towards gram-positive bacteria rather than gram negative bacteria. The reason is most likely because of the difference in cell membrane structure [79].

(Emamifar A. et al., 2011; Kim et al., 2007); showed the antibacterial mechanism of silver involve inducement of oxidative stress because of production of Reactive Oxygen Species (ROS), which may bring about the degradation of the membrane structure of the cell. Release of ions from the surface of nanoparticles has been reported for to cause bacterial death due to binding to cell membrane [80].

(Jin T. and He Y., 2011); found that the higher MgO nanoparticle concentrations brought about in greater bacterial inactivation. An approximate seven log unit diminishment in *E. coli* O157: H7 was accomplished by 8 mg/mL MgO nanoparticle treatment at 24 h. At 7h, the anti *E. coli* O157: H7 activity of

MgO nanoparticles was reliant on its concentration , as in the state of low inoculum levels. The treatment with 3 mg/mL or higher MgO nanoparticles altogether diminished cell concentration to imperceptible levels after 24 h at room temperature , indicating 3 mg/ml MgO nanoparticles would be enough to kill all cells [81].

(Vecitis C. et al., in 2010);demonstrated that CNTs can kill bacteria by causing physical disorder of the cell membrane, oxidative stress , or disruption of a particular microbial process through disturbing-oxidizing a vital cellular structure/compound upon direct contact with bacterial cells [82].

(Jiang M. et al., in 2010); explored the kaolinite mud acquired from Longyan, china to eliminate heavy metal ions Pb^{+2}, Cd^{+2}, Ni^{+2} and Cu^{+2} from wastewater. The take-up is fast with most extreme adsorption being seen near of 30 min. Furthermore, kaolinite mud was utilized for expelling metal ions from real wastewater containing Pb^{+2} ,where its concentration was decreased from 160.00 mg/L to 8.00 mg/L [83].

(Brady A. et al.,Vecitis C. et al., in 2010, 2011 individually); demonstrated that the antimicrobial properties, fibrous shape, and high conductivity of CNTs enable novel CNT filters for both bacteria and infection expulsion The thin layer of CNTs successfully eliminate bacteria by size exclusion and viruses by depth filtration the retained bacteria are to a great extent inactivated by CNTs within hours. With a little intermittent voltage (23eV), multi carbon nanotubes (MWNTs) can immediately oxidize connected bacteria and viruses and lead to inactivation in seconds. The used electric potential likewise promotes viral transport to the anodic CNTs. Such CNT filters can be utilized as high performance. Point –of-use (POU) devices for water sterilization with minimum to no power requirement [84,82].

(Khiari R. et al., in 2010); studied anionic sodium carboxymethyl-cellulose (CMCNa) that was prepared from an agricultural waste date palm rachis and examined as eco-friendly flocculants combined with aluminum sulfate as coagulant for removal of turbidity in drinking water treatment [85].

(Yang B. and Xing K. in 2010); demonstrated that CNTs can firmly adsorb a numerous of polar organic compound because of the dipper contaminant-CNT interactions including hydrophobic impact, π-π interactions, hydrogen bonding, co-valent bonding, and electrostatic interactions [86].

(Nawrocki J. and Kasprzyk B. in 2010); revealed metal oxidative nanomaterials, for example, TiO_2 and CeO_2 and also carbon nanotubes have been studied as catalysts in heterogeneous catalytic ozonation methods that give fast and relatively complete degradation of organic contaminations. Both radical-mediated and non-radical-mediated reaction e pathways have been proposed [87].

(Avanzato C. et al., 2009); examined the antibacterial activity of magnesium oxide-germanium oxide composite powder. The synthesized nano-composite powder showed good bactericidal activity toward both gram-negative (*E. coli*) and in addition gram-positive bacteria (*S. aureus*), however they were more effective against gram-positive bacteria. The outcomes demonstrated that nanocomposite powders were more efficient against *S. aureus* than *E. coli* at lower concentrations. At higher concentrations (>5mg/mL), the growth of bacteria was totally inhibited (>95%) in both conditions. The minimal Inhibitory Concentration (MIC) for *S. aureus* was observed to be 0.05 mg/mL though that of *E. coli* was observed to be 0.25 mg/mL [88].

(Alyüz S. and Veli B., 2009); demonstrated that ion-exchange resin, either manufactured or natural solid resin, had the particular capacity to exchange its cations with the metals in the wastewater. Among the materials utilized in ion-exchange methods, synthetic resins were regularly favored as they were effective to nearly remove the heavy metals from the solution [89].

(Abo-Faraha S. et al. in 2009); examined the impact of ionic charge on the expulsion of Ce^{4+}, Fe^{3+} and Pb^{2+} from aqueous system by cation-exchanges resin purolite C100. They found that the metal ions adsorption sequence can be given as $Ce^{+4} > Fe^{+3} > Pb^{+2}$. Comparative outcomes foe Co^{2+}, Ni^{2+} and Cr^{3+} on an Amberlite-IRN-77 cation-exchange resin were previously gotten by other research group [90].

(Kongsuwan A. et al. in2009); investigated the utilization of acetyl (AC) from eucalyptus bark in the double components sorption of Cu^{2+} and Pb^{2+} the maximum sorption capacities for Cu^{2+} and Pb^{2+} were 0.45 and 0.53mmol/g. A main mechanism for the uptake of both heavy metals proven to be adsorption [91].

(Agoubordea L. and Navia R. in 2009); revealed zinc and copper elimination from aqueous solution utilizing brine sediments, sawdust and the mixture of both materials. The maximum adsorption capacity was observed to be 4.85, 2.58 and 5.59 mg/g for zinc and 4.69, 2.31 and 4.33 mg/g for copper, individually, utilizing an adsorbent-solution proportion of 1/40 [92].

(Landaburu J. et al. in 2009); explored the removal of zinc from synthetic wastewater by micellar enhanced ultrafiltration (MEUF) utilizing suspended dissolved solids (SDS). They found that rejection coefficients up to 99% were accomplished when the surfactant to metal molar proportion (S/M) was over 5 [93].

(Sampera E. et al. in 2009); utilized micellar enhanced ultrafiltration (MEUF) to expel Cd^{2+}, Cu^{2+}, $Pb^{2+}$and Zn^{2+} from synthetic water utilizing two anionic surfactants: sodium dodecyl sulfate(SDS) and linear alkyl benzene sulfonate

(LAS) in a lab-scale film system. The molar concentration proportion of the surfactant to metal is higher than 5 in every one of the experiments. when the initial SDS concentration was below the critical micelle concentration (CMC), metal retention higher than 90% was suddenly gotten, except for Ni^{2+}. In addition, it was demonstrated that finish evacuation of metal ions , except for Ni^{+2},could be accomplished at a liner alkylate sulfonate (LAS) concentration below critical micelle (CMC) [94].

(Dialynas E. and Diamadopoulos E. in2009); used a pilot-scale membrane bioreactor system in combination with reverse osmosis (RO) and they discovered heavy metal elimination efficiencies were very high. The major disadvantage of (RO) is the powerful utilization because of the pumping pressures, and the restoration of the membranes [95].

(Koseoglu H. and Kitis M. in 2009); explored the recovery of silver from mining wastewaters utilizing Nano Filtration (NF) or Reverse Osmosis (RO) after the silver was taken into solution as AgCN employing re-cyanidation and subsequent sedimentation or pre-filtration of wastewaters. Silver recoveries accomplished by hybrid cyanidation and membrane separation were 29-59% and 54-62% for nanofiltration and reverse osmosis membranes, individually [96].

(Chang Q. et al. in 2009b); synthesized a macromolecule heavy metal flocculant mercaptoacetyl chitosan by reacting chitosan with mercaptoacetic acid . They announced this new flocculant could not just remove turbidity, additionally evacuate heavy metals in wastewater [97].

(Kabdasli I. et al. in 2009); researched the treat ability of a metal plating wastewater containing complexed metals starting from the nickel and zinc plating process by Electrocoagulation (EC) utilizing stainless steel electrodes. Their review showed that the highest total organic carbon (TOC) reduction (66%) and also nickel and zinc removals (100%) were accomplished with an utilized current density of 9mA/cm^2 at the original electrolyte (chloride) concentration and original pH of the composite specimen used [98].

(Bhains K.and Saouza D. in2008); explored the elimination of copper ion utilizing NaOH treated rhizopusoryzae biomass in a batch reactor. The maximum copper loading capacity of the suitable and pretreated biomass was 19.4 and 43.7 mg/g, respectively [99].

(Molinari R. et al. in 2008); utilized poly-ethylen-imine (PEI) as a polymer to study the complex ion ultrafiltration method in the selective elimination of Cu^{2+} from Ni^{2+} contained in aqueous media. Preliminary tests demonstrate that ideal chemical conditions for Cu^{2+}and Ni^{2+} complex ion by the PEI were PH > 6.0 and 8.0 separately, and polymer/ metal weight proportion of 3.0 and 6.0, respectively [100].

(Murthy Z. and Chaudhari L. in 2008); detailed the use of a thin-film composite polyamide nano filtration NF membrane for the dismissal of nickel ions from aqueous wastewater. The maximum observed rejection of nickel was to be 98% and 92% for an initial feed concentration of 5 and 250 mg/L, individually. Also, they researched the binary heavy metals (cadmium and nickel) detachment ability of a commercial NF film from aqueous liquid. The most extreme watched solute dismissal of nickel and cadmium ions is 98.94% and 82.69%, separately, for an initial feed concentration of 5 mg/L [101].

(Liu F. et al. in 2008); studied the performance of various nanofiltration (NF) and reverse osmosis (RO) membranes in treating the poisonous metal effluent from metallurgical industry. They detailed that the obtained water by both NF and RO desalination satisfied the State Reutilization Qualification, but NF would be more suitible for expansive scale industrial practice [102].

(Yuan X. et al. in2008); explored the capability of ion filtration to evacuate cadmium, lead and copper from dilute aqueous solution with a plant- derived bio surfactant tea saponin. The maximum evacuation of Pb^{2+}, $Cu^{2+}$and Cd^{2+} can achieve 89.95%, 81.13% and 71%, respectively, when the proportion of collector to metal was 3:1 [103].

(Belkacem M. et al. in 2008); studied the clarification of wastewater utilizing the Electrocoagulation-electroflotation (EF) method with aluminum electrodes . The utilization of the optimized parameters on the separation of some heavy metal ions, for example, iron, nickel, copper, zinc, lead and cadmium was examined. Their review exhibited that the metal evacuation rate achieved 99% [104].

(Pan and Xing in 2008); demonstrated that carbon based nano-adsorbents have higher efficiency than activated carbon on adsorption of different organic chemicals. Its adsorption capacity fundamentally steams from the large particular surface area and the divers contaminant-CNT interactions. The available surface area for adsorption on individual CNTs is their exterior surfaces [105].

(Shivastava S. et al. , 2007); revealed the synthesized of silver nanoparticles in the range of 10-15 nm with raised stability and enhanced anti- bacterial potency. The antibacterial impact was dose dependent [106].

(Amuda Q. and Amoo I., in 2007); detailed that the combined utilization of coagulant (ferric chloride) and polyelectrolyte (non-ionic polyacrylamide) resulted in the production of sludge volume with decrease of 60% of the quantity produced when coagulant was solely utilized for the treatment of drink industrial wastewater [107].

(Kousi B. in 2007); developed an up flow fixed bed sulfate reducing bacteria (SRB) to observe for the treatment of zinc-bearing wastewater. They found that the reactor has an extensive capabilities of totally diminishing sulfates for initial concentration up to 6000 mg/L, totally expelling soluble zinc for initial concentrations up to 400 mg/L and totally evacuating total organic carbon (TOC) for initial concentrations up to 1500 mg/L [108].

(WangH. et al. 2007a); detailed the adsorption of Pb^{+2} utilizing acidified multi carbon nano tubes (MWCNTs) and found the oxygenous functional groups on MWCNTs play an important role in Pb^{+2} adsorption to form chemical complex adsorption, which accounts 75.3% of all the Pb^{+2} adsorption capability [109].

(Mohsen-Nia M. et al.,in 2007); indicated Cu^{2+} and Ni^{2+} particles were effectively expelled by the Reverse osmosis (RO) process and rejection efficiency of the two ions increased up to 99.5 % by utilizing Na_2EDTA [110].

(Polat H. and Erdogan D., in2007); achieved ion flotation to evacuate Cu^{2+}, Zn^{2+}, $Cr^{3+}$and Ag^{+} from waste waters. Sodium dodacyl sulfate (SDS) and hexadecyl-trimethyl-ammonium bromide were utilized as collectors. Ethanol and methyl isobutylcarbinol were utilized as frothers. Metal evacuation reach about 74% under ideal conditions at low pH. At essential pH it moved toward becoming as high as 90%, most likely because of the contribution from the flotation of metal precipitates [111].

(Macak J. et al. in 2007); observed TiO_2 nanotubes to be more effective than TiO_2 nanoparticles in decomposition of organic components. The higher photocatalytic action was attributed to the shorter carrier/diffusion paths in the tube walls and quicker mass transport of reactants toward the nanotube surface [112].

(Bolto B. et al., in 2007); demonstrated that the biodegradable polymers, completely shear stable, effectively accessible from reproducible farming resources and produce no secondary contamination. Also, as biopolymers are biodegradable, the sludge can be effectively degraded by microorganisms. These, they had high potential to be applied not only in foods and fermentation processes, pharmaceutical, cosmetic, downstream processing additionally in water and wastewater treatment [113].

(Ozverdi A. and Erdem M. in 2006); explored pyrite and synthetic iron sulfide to remove Cu^{+2} , Cd^{+2} and Pb^{+2}. The mechanism governing the metal evacuation process was calculated as chemical precipitation at low PH(<3) because of H_2S releasing [114].

(Gonzalez M. et al. in 2006); revealed sulfide precipitation to reuse and recapture heavy metal particles utilized nanofiltration as a second step. Results indicated sulfide precipitation was effective in decreasing the metal content and nano-filtration yielded solutions able to being directly reused in the plant [115].

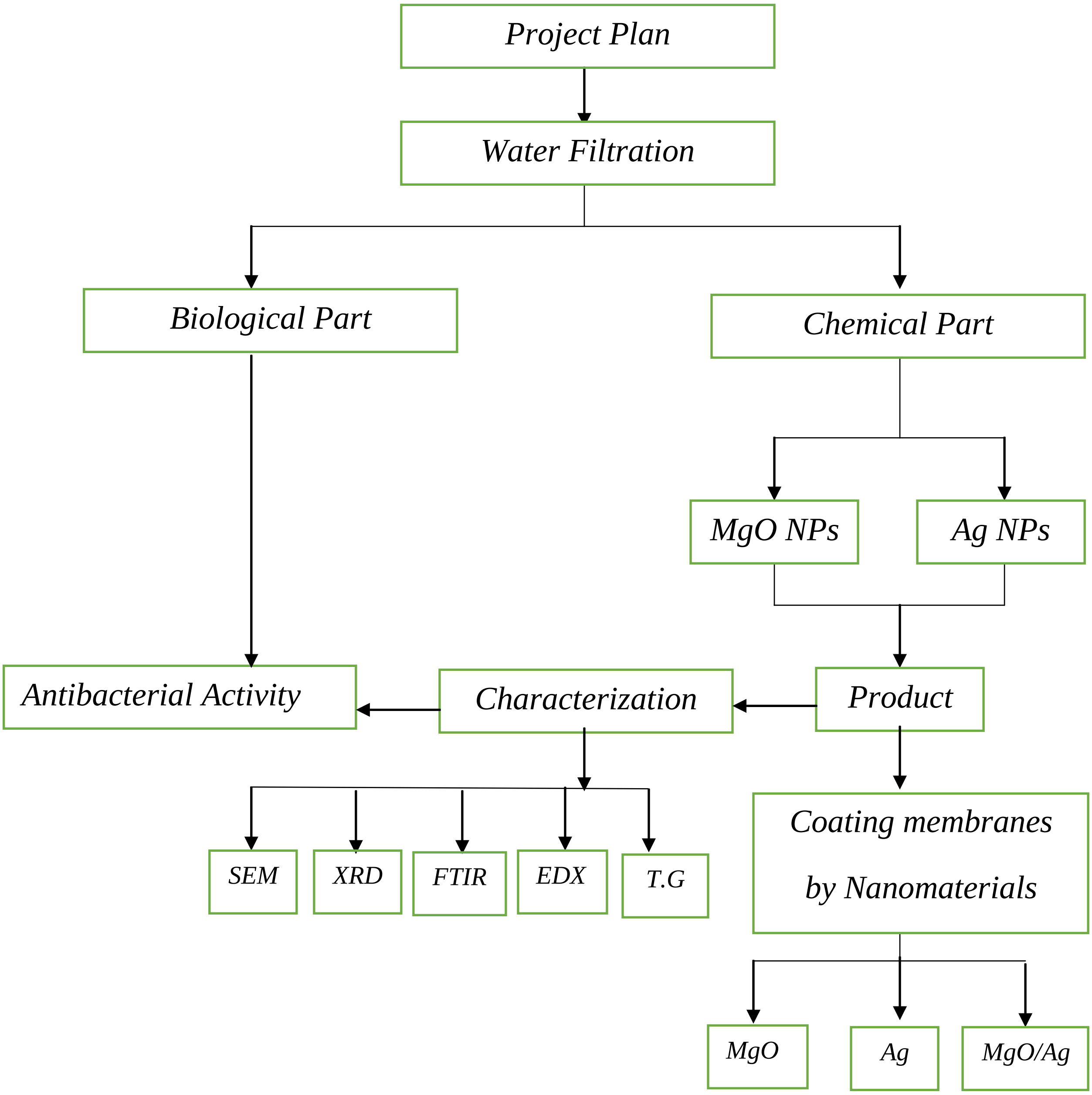
(Xu and Zhang in 2006); developed another organic heavy metal chelatordipropyldithiophosphate. The chelator can expel the concentration of lead, cadmium, copper and mercury being 200 mg/L at pH 3.6 up to more than 99.9% and the heavy metal concentrations in the wastewater after treatment are less than 1, 0.1, 0.5 and 0.05 mg/L, individually [116].

1.14 Aim of project

1) Preparation of nanomaterials (MgO and Ag).
2) Characterization of nanomaterials by (SEM, XRD, TG, EDX, FTIR).
3) Application of nanomaterials for waste water filtration.

Chapter Two

Experimental Part


Project Plan
Water Filtration
Biological Part
Chemical Part
MgO NPs
Ag NPs
Antibacterial Activity
Characterization
Product
SEM
XRD
FTIR
EDX
T.G
Coating membranes by Nanomaterials
MgO
Ag
MgO/Ag

2.1 Instruments

Table (2.1) illustrates the list of instruments used in this project, their manufacturers, model, functions and the place of the instrument.

Table (2.1) List of Instruments

Instruments	***Model***	***Function***	***Place Of Instrument***
Vacuum oven	*Gallen kamk*	*For drying the Sample*	***Lab. Collage Science of Al-Nahrian University***
Electronic Balance	*METTLER AE260Delta Range*	*Weight measurement*	=
Fourior transform Infrared Spectroscopy)FTIR(	*SHIMADZU (IR PRESTIGE 21)*	*To determine the organic functional groups present in the samples*	***Laboratory of Ibn-Sina Scientific Company***
Magnetic stirrer	*IKAMAG®REO*	*To heat and stir the solution of the sample*	***Lab. Collage Science of Al-Nahrian University***
Scanning Electron Microscopy (SEM)	*Inspect S 50 FEI company*	*For studying the morphology of the samples*	***Central Lab. University of Tahran***
X-ray Diffraction	*ADC2500*	*The chemical composition and crystal structure of the sample*	***Lab. At ministry of science and technology***
Energy Dispersive X-ray Spectroscopy	*Inspect S 50 FEI company*	*To determine the elemental constituents of sample*	***Lab. Collage Science of Al-Nahrian University***
Atomic Absorption Spectroscopy	*novAA330*	*To determine concentration of*	***Laboratory of Ibn-Sina Scientific***

		sample	
Vortex Mixers	*LABINCO L46*	*To mix the liquid*	***Lab. Collage Science of Al-Mustansiryah University***
Autoclave	*memmert*	*To sterilization of sample*	***Lab. Collage of Science Al-Mustansiryah University***
Thermal Gravimetric(TG)	*TGA PT1000*	*To determine the mass loss with respect temperature*	***Lab. Collage of education for pure science/ Baghdad university/Ibn Al-haithm***

2.2 The Chemical Material:

All reagents used during the project are given in Table (2.2).

Table (2.2) Physical Properties of Chemicals Used.

Chemicals	***Chemical Formula***	***Molar Mass***	***purity***	***Supplier***
Sodium hydroxide	$NaOH$	*40*	*99.9%*	***Scharlau company***
Ethanol	CH_2CH_2OH	*49*	*100%*	***Sigma aldrich company***
Silver nitrate	$AgNO_3$	*169.87*	*99.95%*	***BDH company***
Magnesium nitrate hexahydrate	$Mg(NO_3)_2.6H_2O$	*256.4*	*99.9%*	***BDH company***
Deionized water	H_2O	*18*	*High degree of purity*	***Lab.College science of Al.Nahrain university***

2.3 Sample Preparation

2.3.1 Synthesis of Magnesium oxide nano-particles

10.2gm (0.2M) of magnesium nitrate $Mg(NO_3)_2.6H_2O$ was dissolved in 200ml of De-ionized water.

4gm (0.5M) of sodium hydroxide solution was dissolved in 200ml De-ionized water.

Sodium hydroxide solution was added dropwise to the previous prepared magnesium nitrate $Mg(NO_3)_2.6H_2O$ solution while stirring it constantly. White precipitate of magnesium hydroxide appeared in beaker after a few minutes. The stirring was continued for 2 h ._________(1)

The precipitate was filtered and washed with De-ionized water four to five times to remove ionic impurities

Then the white precipitate divided into three parts, part one dried in furnace at 80ºC, part two dried at 60ºC and part three dried at 100ºC[117].

2.3.2 Synthesis of Nano-Sliver particles

(6.7948gm) (0.2M) of sliver nitrate $AgNO_3$ was dissolved in 200ml De-ionized water.

4gm (0.5M) sodium hydroxide was dissolved in 200 ml De-ionized water.

Sodium hydroxide was added drop by drop to prepared sliver nitrate solution while stirring it constantly . Brown precipitate of sliver appear in the beaker after few minutes._________(2)

The stirring was continued for 2 hours, the precipitate left to dried in dark place.

2.3.3 Modification of MgO nanoparticles using Ag NPs

9.2mg/ml of $Mg(OH)_2$ was added to 9.2mg/ml of Ag in order to get the following reaction:

By this reaction band gap of MgO nanoparticles was reduced using Ag.

2.4 Antibacterial activity studies:

2.4.1 Magnesium oxide, silver and MgO-Ag nanoparticles

Antibacterial activity of MgO NPs was estimated against the Gram-negative bacteria *E. coli* and Gram- positive bacteria *Staphylococcus aureus* by diffusion method. A loop full of *E. coli* and *Staphylococcus aureus* were grown overnight in nutrient Broth (NB) at 37°C for 24 h and after incubation 100µL of culture was injected in mueller hinton agar by spread plate method and hole was made to add 0.5ml of $Mg(OH)_2$, Ag and MgO-Ag nanoparticles. The antibacterial activity of the MgO, Ag and MgO-Ag nanoparticles were estimated by test the zone of inhibition of bacterial growth. And the antibacterial activity is determined as zone of inhibition in mm in diameter [118].

2.5 The process of Coating Membranes

Four fabric membranes were took and labeled as (A, B, C, D), Membrane (A) left without coating, 3.06mg/ml of $Mg(OH)_2$ (which prepared previously in (1)) was deposition on membrane (B) and left to dried in furnace for an hour at 80°C to get membrane coated with MgO nanoparticles, 3.06mg/ml of Ag (which prepared previously instep (2)) was deposition on membrane (C) and left to dried for one a day to get membrane coated with Ag nanoparticles, 3.06mg/ml of $Mg(OH)_2$-Ag was deposition on membrane (D) and left to dried for one a day to get membrane coated with MgO-Ag nanoparticles.

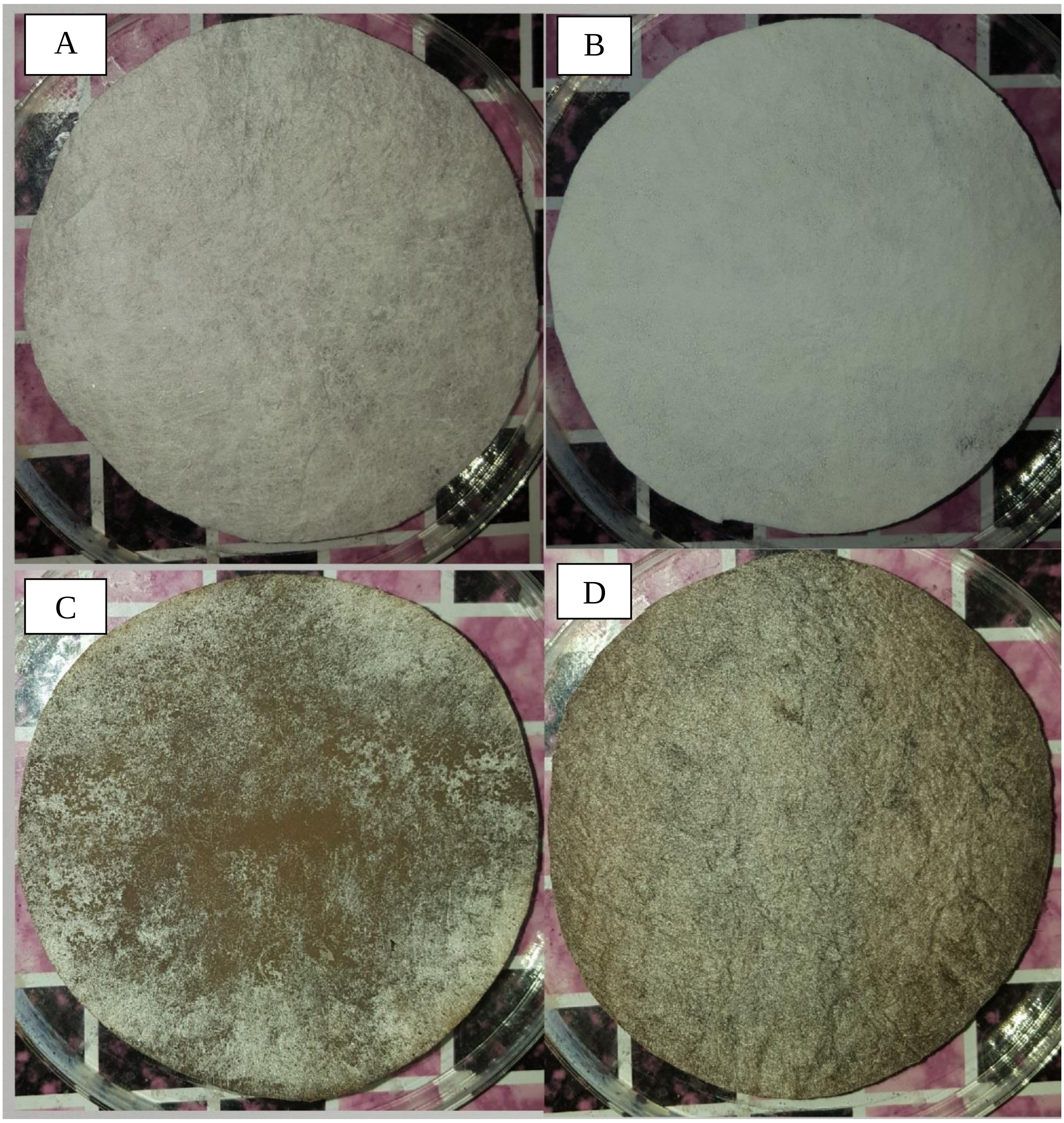


Figure (2.1): (A) membrane without coating, (B) membrane coated with MgO, (C) membrane coated with Ag, (D) membrane coated with MgO-Ag.

2.6 Filtration wastewater by coated Membranes

50ml of wastewater was took and poured into the uncoated membrane (A) and then left for an hour until the filtration process was accomplished, then the resulting water was collected for examination. The step above was repeated for the others coated membranes (B, C and D).

2.7 Determination of Water Conductivity

For testing the water samples, firstly the reagents are to be prepared. Then the conductivity meter is required to be calibrated.

2.7.1 Preparation of Reagents (Potassium Chloride Solution)

0.7456gm of potassium chloride was dissolved in 50ml of De-ionized water. The contents poured into volumetric flask , and complete the volume up to 100ml of De-ionized water .This solution is used to calibrate the conductivity meter.

Before starting experiment , the instrument was switch on for 30 minutes, so that the instrument stabilizes. Used the same electrode, the conductivity meter can measure three parameters :

1. ***Conductivity***
2. ***pH***
3. ***Total Dissolved Solids (TDS)***

50ml of water samples was taken prior and after filtration process to measure the three parameter (conductivity, pH and TDS) by soaking the electrode of conductivity meter into water samples.

2.8 Determination Membrane Efficiency

The efficiency of the membranes were measured by using it more than once , each time pH, ES and TDS were measured for each sample. At third filtration , the work of membranes has stopped , where no water drops were observed for two days.

2.9 Characteristic Equipment

2.9.1 Atomic Absorption Spectroscopy

Atomic absorption spectrometry (AAS) is an analytical chemistry method that determines the concentrations of particular element. Atomic absorption is very critical that it able to measure down to parts per billion of a gram (µg dm^{-3}) in a specimen. The method makes utilize of the wavelengths of light (i.e. amount of energy) specifically absorbed by an element [119].

Figure (2.2) Atomic absorption device. The work done at laboratory of Ibn-sina scientific company.

2.9.2 Scanning Electron Microscopy

A scanning electron microscope (SEM) is a sort of electron microscopy that gives images of a specimen by scanning it with a focused beam of electrons. The electrons interact with atoms in the specimen , producing different signals that have information about the specimen's surface composition and topography. The electron beam is scanned in a raster scan manner, and the beam's placement is combined with the detected signal to give an image. SEM can give resolution better than 1 nanometer. Specimens can be observed in high and low vacuum, in humid conditions, and at a large range of cryogenic or elevated temperatures [120].

Figure (2.3) Scanning electron microscopy (SEM) Device. The work done at University of Tahran.

2.9.3 . Fourier Transform Infrared Spectrophotometer (FTIR)

Fourier Transform-Infrared Spectroscopy (FTIR) is an analytical technique used to identify organic (and in some cases inorganic) materials. This technique measures the absorption of infrared radiation by the sample material versus wavelength. The infrared absorption bands identify molecular components and structures.

When a material is irradiated with infrared radiation, absorbed IR radiation usually excites molecules into a higher vibrational state. The wavelength of light absorbed by a particular molecule is a function of the energy difference between the at-rest and excited vibrational states. The wavelengths that are absorbed by the sample are characteristic of its molecular structure [121] .

Figure (2.4) Fourier Transform Infrared Spectrophotometer. The work done at Laboratory of Ibn-Sina Scientific Company.

2.9.4 Energy Dispersive X-ray

Energy-dispersive spectroscopy (EDS, EDX, EDXS or XEDS), sometimes called energy dispersive X-ray analysis (EDXA) or energy dispersive X-ray microanalysis (EDXMA), is an analytical instrument utilized for the elemental analysis or chemical description of a specimen. It relies on an interaction of some source of X-ray excitation and a specimen. Its characterization capacities are due in great part to the essential principle that each element has an individual atomic structure allowing an individual set of peaks on its electromagnetic emission spectrum (which is the main principle of spectroscopy) [122].

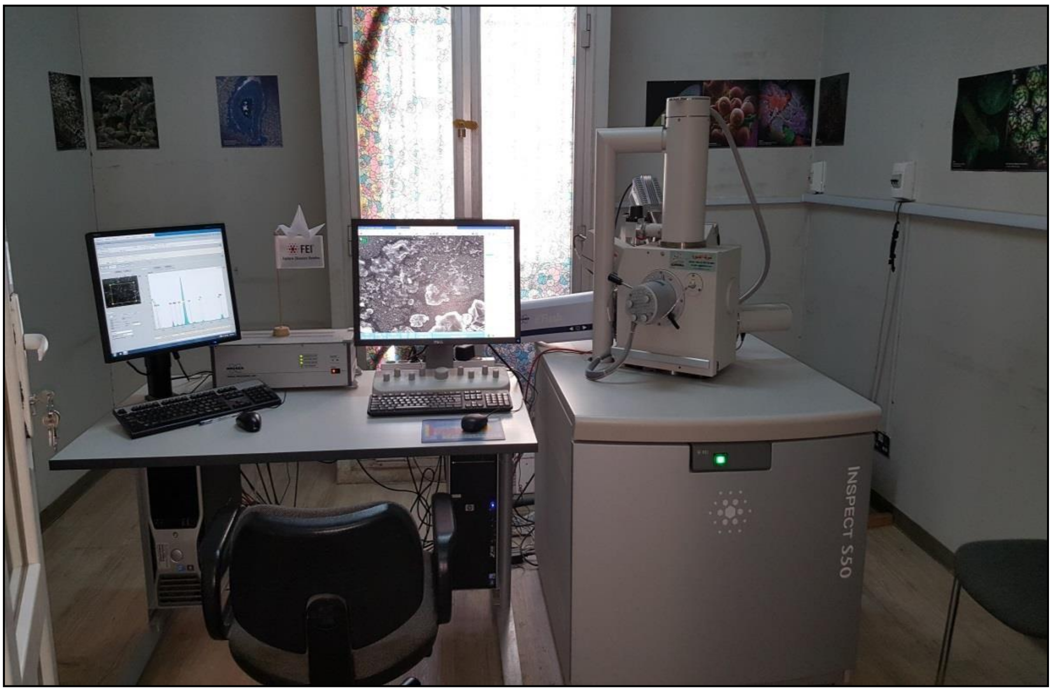


Figure (2.5) Energy Dispersive X-ray Device. Al-Nahrian University.

2.9.5 X-ray Diffraction(XRD)

Is a technique used for determining the atomic and molecular structure of a crystal, in which the crystalline atoms cause a beam of incident X-rays to diffract into many specific directions. By measuring the angles and intensities of these diffracted beams, a crystallographer can produce a three-dimensional picture of the density of electrons within the crystal. From this electron density, the mean positions of the atoms in the crystal can be determined, as well as their chemical bonds, their disorder, and various other information [122].

Figure (2.6) X-ray diffraction Device. The work done at department of science and technology.

2.9.6 Thermal Gravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) is a method of thermal analysis in which changes in physical and chemical properties of substances are determined as a function of rising temperature (with constant heating rate), or as a function of time (with constant temperature and/or constant mass loss). TGA can give information about physical phenomena, such as second-order phase transitions, like vaporization, sublimation, absorption and desorption. Likewise, TGA can give information about chemical phenomena including chemisorptions, desolvation (specially dehydration), decomposition, and solid-gas reactions (e.g., oxidation or reduction) [122].

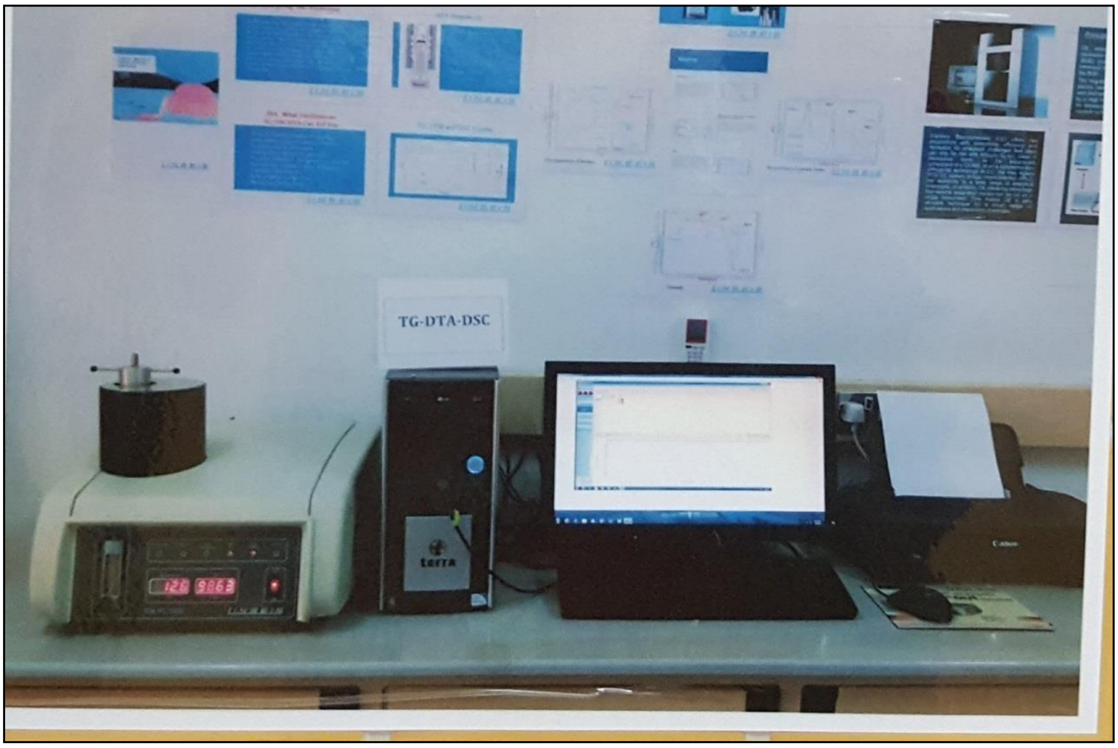


Figure (2.7) Thermal gravimetric analysis (TGA) device. The work done at Collage of education for pure science/ Baghdad university/Ibn Al-Hathim

Chapter Three

Results and Discussion

3.1 Physical and chemical characteristics:

3.1.1 Fourier Transform Infrared Spectroscopy (FTIR)

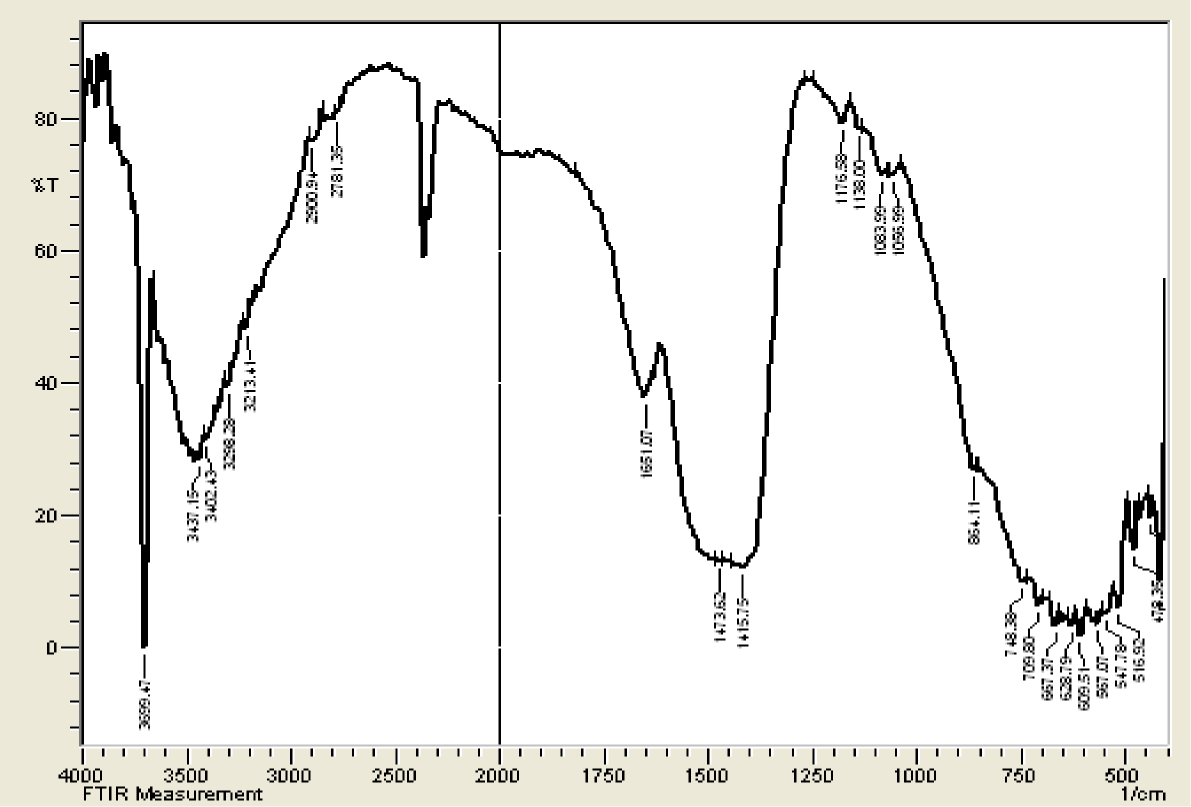


Figure (3.1): FTIR Spectra for MgO nanoparticles dried at 60ºC.

Table (3.1): FTIR spectra for MgO nanoparticles dried at 60ºC

Wave number (cm^{-1})	***Type of bonds***
3699.47	υOH antisymmetric
3437.15	υOH (H_2O)
1415.75	υMg-OH
1651.75	δOH (H_2O)
478.35	υMg-O

Figure (3.1) Shows sharp peak at (3699.47 cm^{-1}) is attributed to the OH antisymmetric stretching vibration in $Mg(OH)_2$. The peaks at (478.35 cm^{-1}) are assigned to the Mg–O stretching vibration. The absorption peak at (1415.75 cm^{-1}) corresponds to the Mg–OH stretching vibration. The other absorption peak at

(1651.75 cm^{-1}) and (3437.15 cm^{-1}) due to the bending and the stretching vibration of water, respectively.

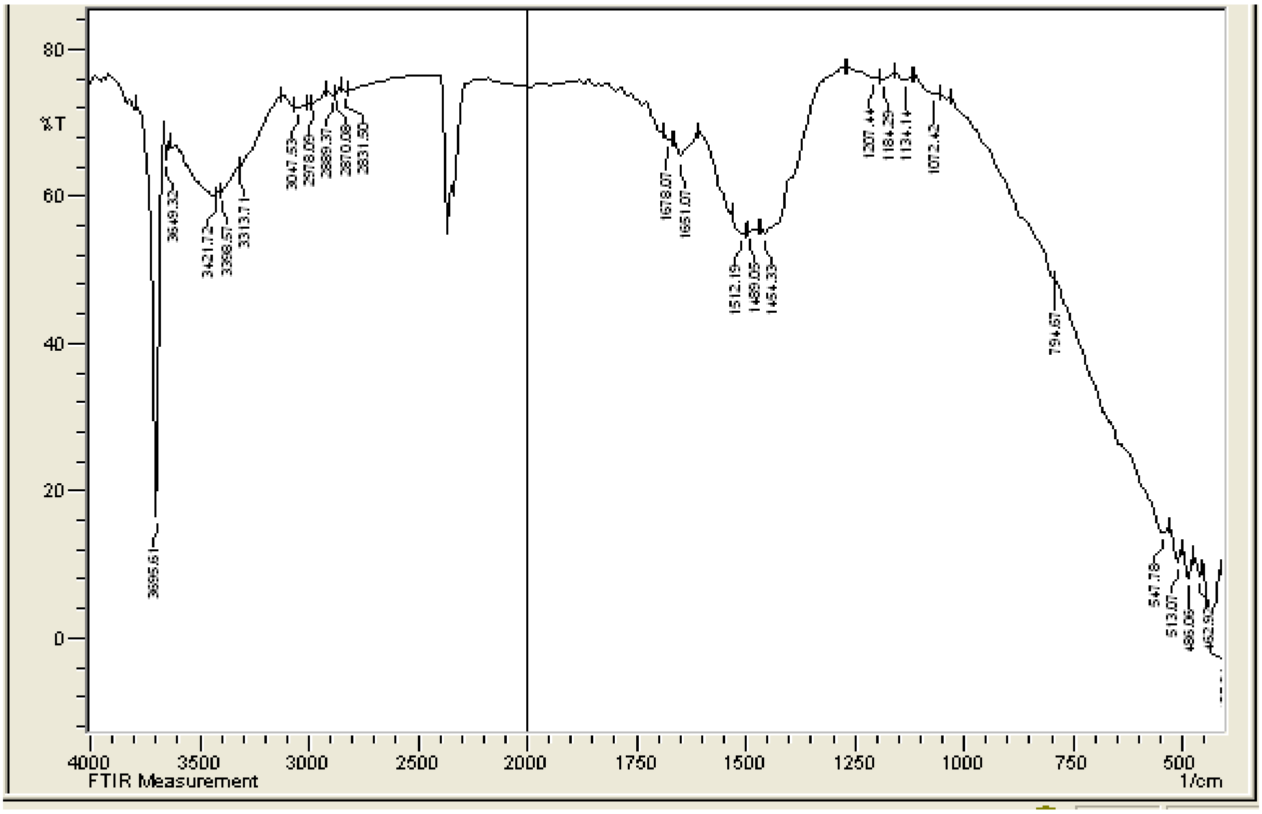


Figure (3.2): FTIR Spectra for MgO nanoparticles dried at 80ºC.

Table (3.2): FTIR spectra for MgO nanoparticles dried at 80ºC.

Wave number (cm^{-1})	***Type of bonds***
3695.61	νOH antisymmetric
3398.57	$\nu OH\ (H_2O)$
1512.19	$\nu Mg\text{-}OH$
1651.06	$\delta OH\ (H_2O)$
435.91	$\nu Mg\text{-}O$

Figure (3.2) Shows sharp peak at (3695.61 cm^{-1}) is attributed to the OH antisymmetric stretching vibration in Mg $(OH)_2$. The peak at (435.91cm^{-1})are assigned to the Mg–O stretching vibration. The absorption peaks at (1512.19 cm^{-1}) corresponds to the Mg–OH stretching vibration. The other absorption peak at (1651.07 cm^{-1}) and (3398.57 cm^{-1}) due to the bending and the stretching vibration of water, respectively.

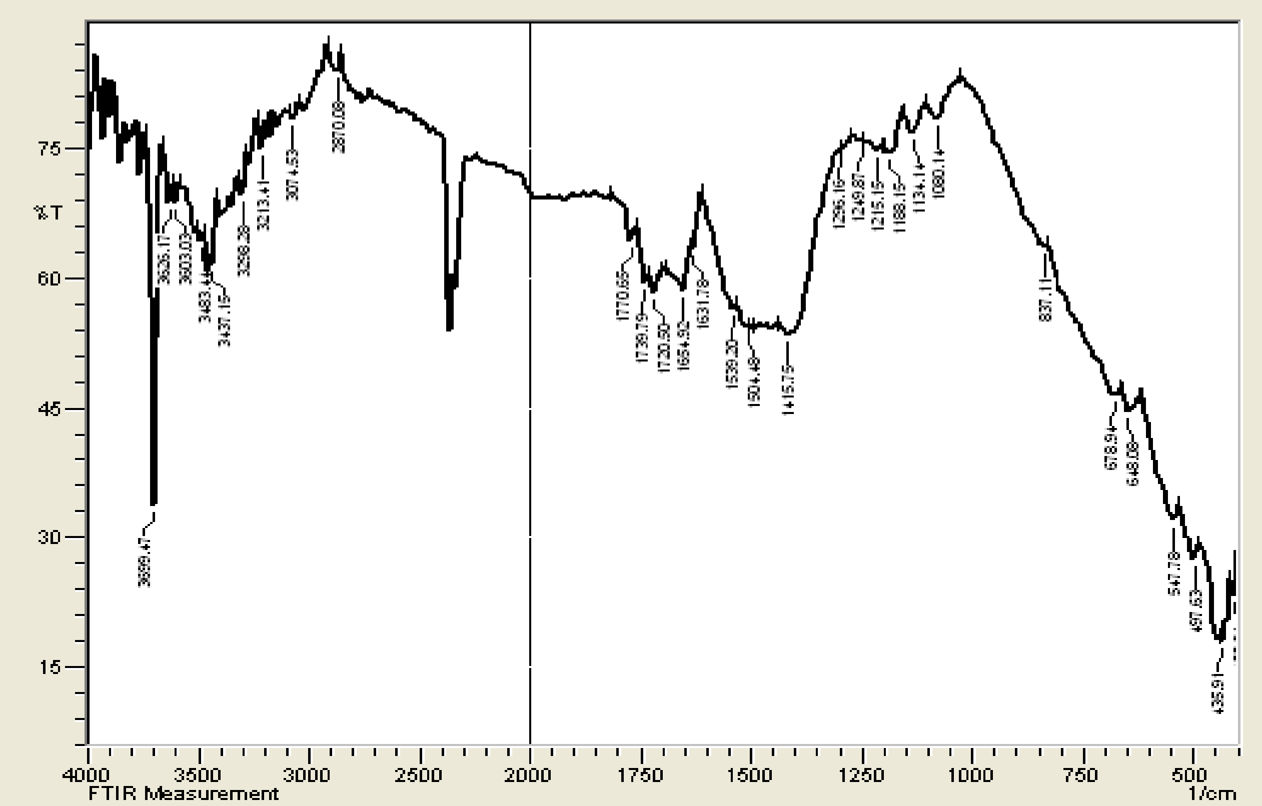


Figure (3.3): FTIR Spectra for MgO nanoparticles dried at 100ºC.

Table (3.3): FTIR spectra for MgO nanoparticles dried at 100ºC.

Wave number (cm^{-1})	*Type of bonds*
3699.47	*vOH* antisymmetric
3483.44	*vOH (H_2O)*
1504.48	*vMg-OH*
1654.92	*δOH (H_2O)*
435.91	*vMg-O*

Figure (3.3) shows sharp peak at (3699.47 cm^{-1}) is attributed to the OH antisymmetric stretching vibration in Mg $(OH)_2$. The peak at (435.91cm^{-1}) are assigned to the Mg–O stretching vibration. The absorption peak at (1504.48 cm^{-1}) corresponds to the Mg–OH stretching vibration. The other absorption peaks at (1654.92 cm^{-1}) and (3483.44 cm^{-1}) due to the bending and the stretching vibration of water, respectively. **(This work agreements with Devi Rashmi).** [123]

3.1.2 X-Ray Diffraction

The chemical composition and crystal structure of MgO nanoparticles and nano sliver are often determined by X-ray diffraction using CuKα radiation with a wavelength 1.5418 A^0 .The x-ray diffraction spectra have been analysed for MgO nanoparticles that dried at three different temperatures 60ºC, 80ºC, 100ºC. MgO NP showed peak at $2\theta = 43^o$ is corresponded to plane (211). From the peak width and intensity one can identify the smaller particle size and better crystallinity as obtained in Figure (3.4). No other peaks were observed in the spectra. **(This work agreements with Wahab and Asari).** [117]

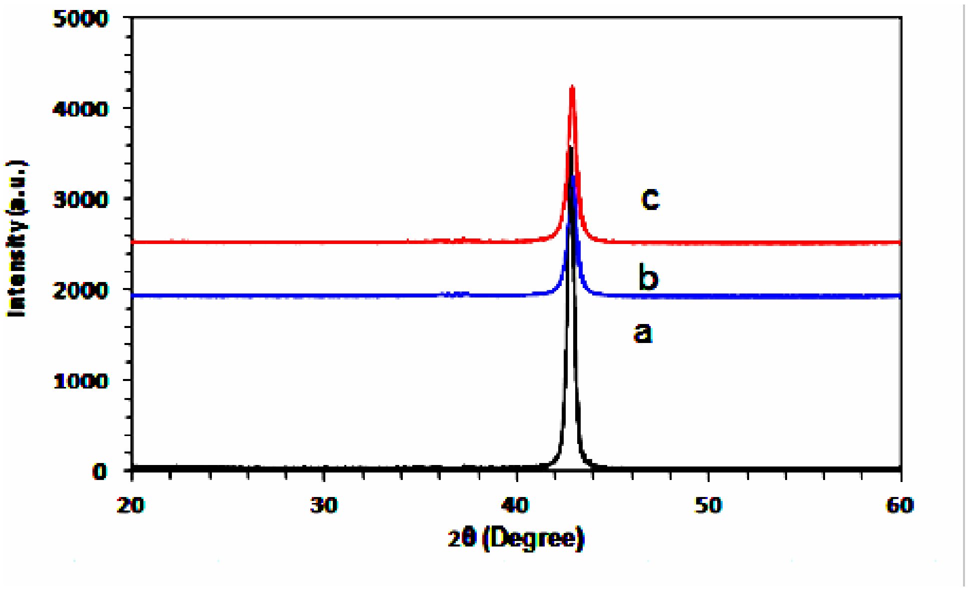


Figure (3.4): X-ray diffraction spectra of MgO nanoparticles dried at three temperatures (a) at 80ºC, (b) at 60ºC, (c) at 100ºC.

Figure (3.5) showed the peak of sliver NPs spectrum at $2\theta=38.5^{o}$ is corresponding to the plane (111). From peak width and intensity one can identify smaller particle size 50.73 nm.

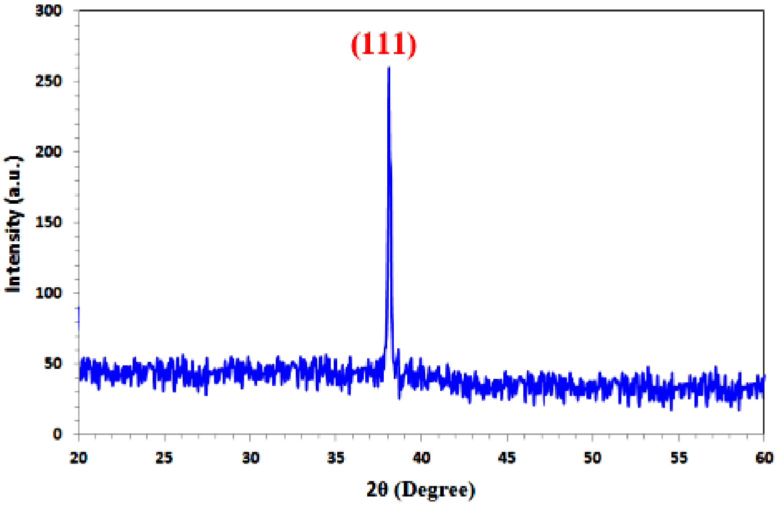


Figure (3.5): X-ray diffraction spectra of sliver nanoparticles.

3.1.3 Scanning Electron Microscopy

The particle size and morphology of synthesized nanoparticles were diagnosed using Scanning Electron Microscope. MgO and membrane coating with synthesized MgO nanoparticles were characterized in Figure (3.6 (a, b)). The particles seemed to be small spherical agglomeration , particle size for samples dried at 60ºC, 80ºC ***(Optimal temperature)*** and 100ºC were as following :

Table (3.4): Particle sizes for MgO nanoparticles dried at three different temperatures.

Temperature (ºC)	***Particle size (nm)***
60	21
80	40
100	33.56

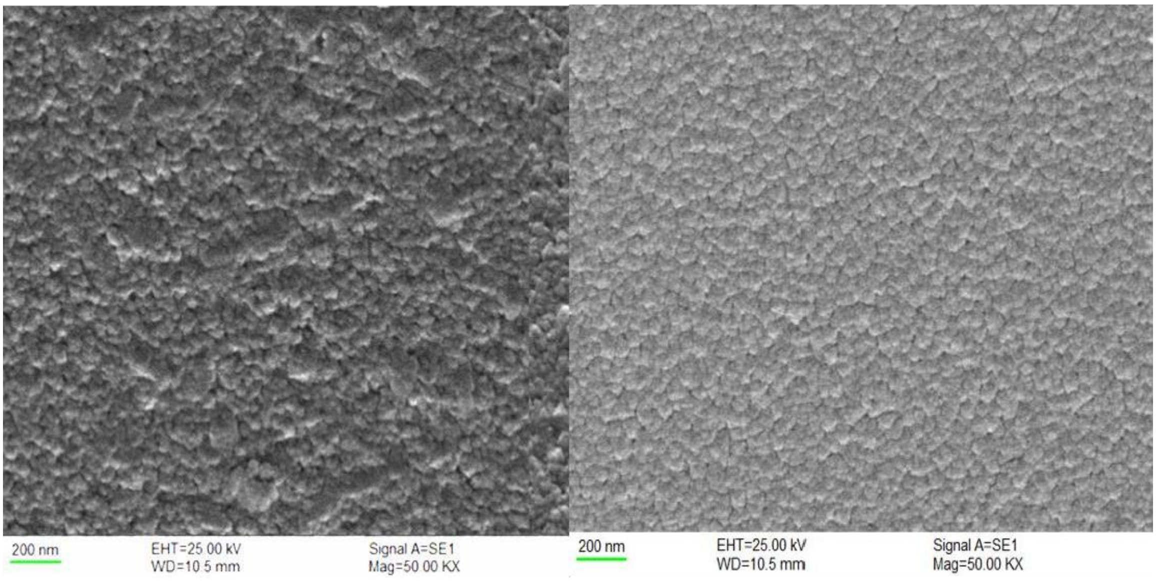


Figure (3.6): Scanning elcctron microscopy of (a) MgO nanoparticles dried at 80ºC (Optimal temperature):(left) and (b) membrane coating with MgO nanoparticles:(right).

Figure (3.7 (a, b)) shows the SEM image of sliver and membrane coating with synthesized sliver nanoparticles by chemical method. It is clear from this image that a common characteristics of the particles is spherical agglomeration in shape with particle size of (50.73nm).

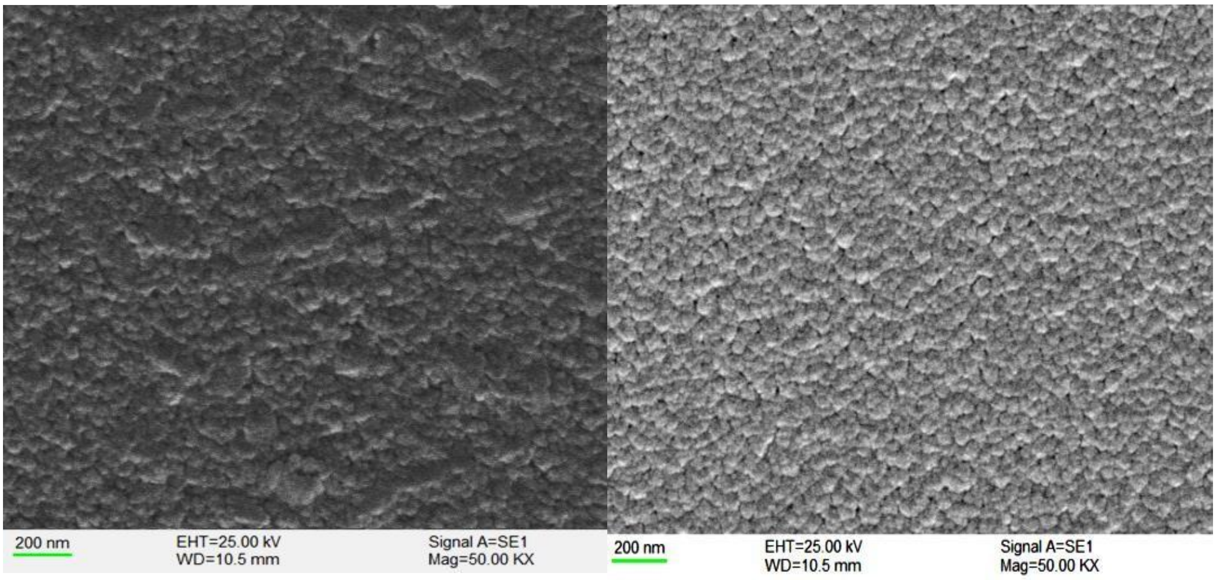


Figure (3.7): Scanning electron microscopy of (a):(left) sliver nanoparticles and (b) membrane coating with sliver nanoparticles: (right).

Figure (3.8) shows the SEM image of membrane coating with a mixture of synthesized MgO and sliver nanoparticles. It is clear from this image that a membrane is coated well, a common characteristics of the particles is spherical agglomeration has form on coated membrane, and it is visible that particles are similar in size, with particle size of (48.91nm).

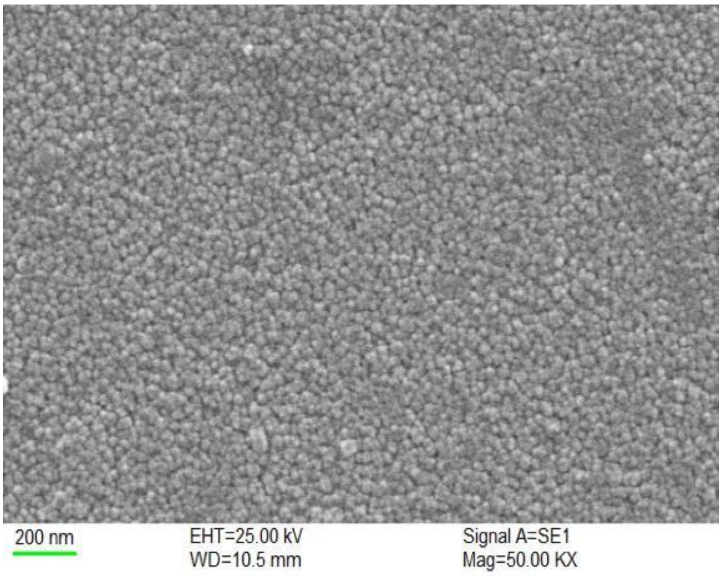


Figure (3.8): SEM image of membrane coating with a mixture of MgO and sliver nanoparticles.

3.1.4 Energy Dispersive X-ray

EDX analysis of nanoparticles were performed to determine the elemental constituents of synthesized materials, which give additional information about the purity of synthesized nanoparticles for three prepared samples that were dried at different temperatures (60, 80 and 100 ^{o}C) . Figure (3.9 (a, b and c)) presents peaks between 0.2 and 1.5 keV, which indicates the presence of MgO, at which, magnesium percentage is about (37.13, 67.83 and 47.28) while, the oxygen percentage (22.33, 23.64 and 34.13) respectively. The percentages quantities of the remaining elements are as follows: C (29.79, 8.54), Na (10.75, 8.66) and N (9.93).

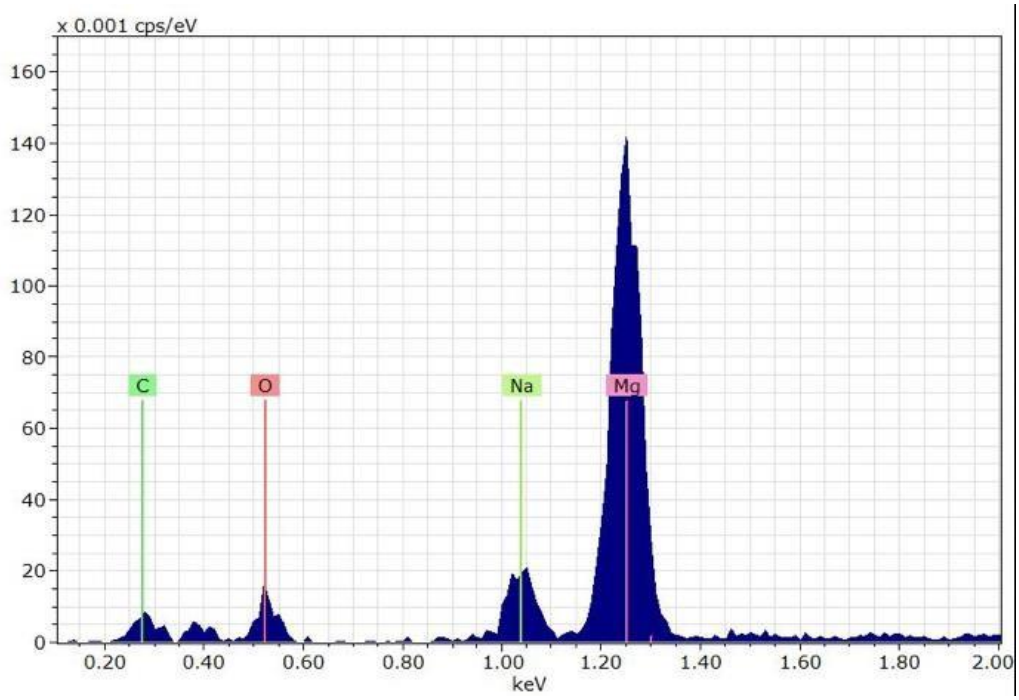


Figure (3.9) (a): EDX spectra for MgO nanoparticles dried at 60ºC.

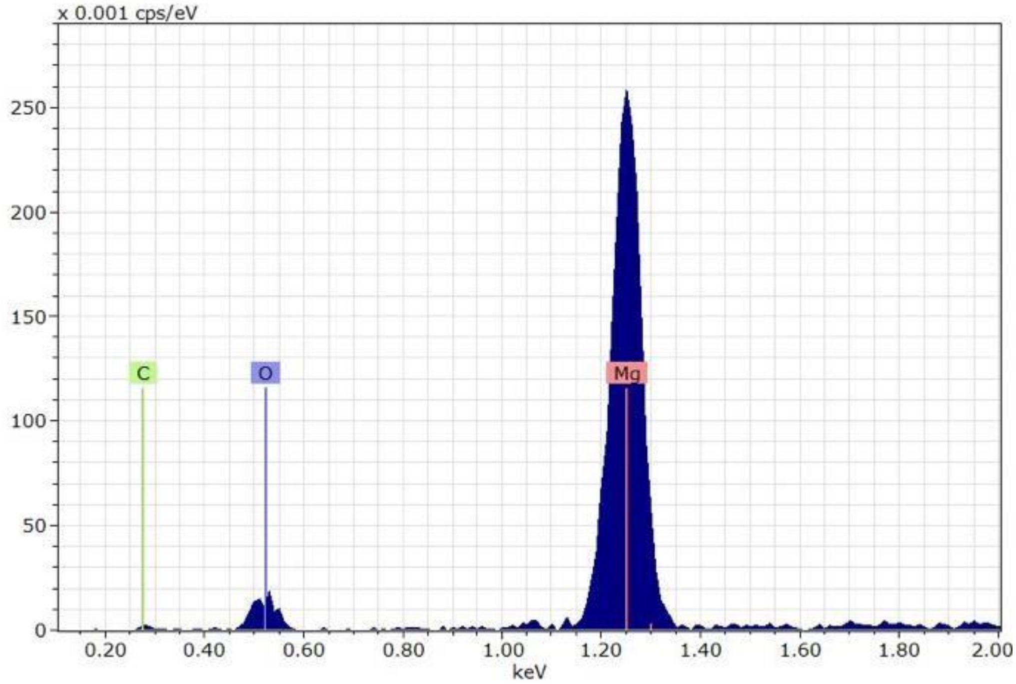


Figure (3.9) (b): EDX spectra for MgO nanoparticles dried at 80ºC.

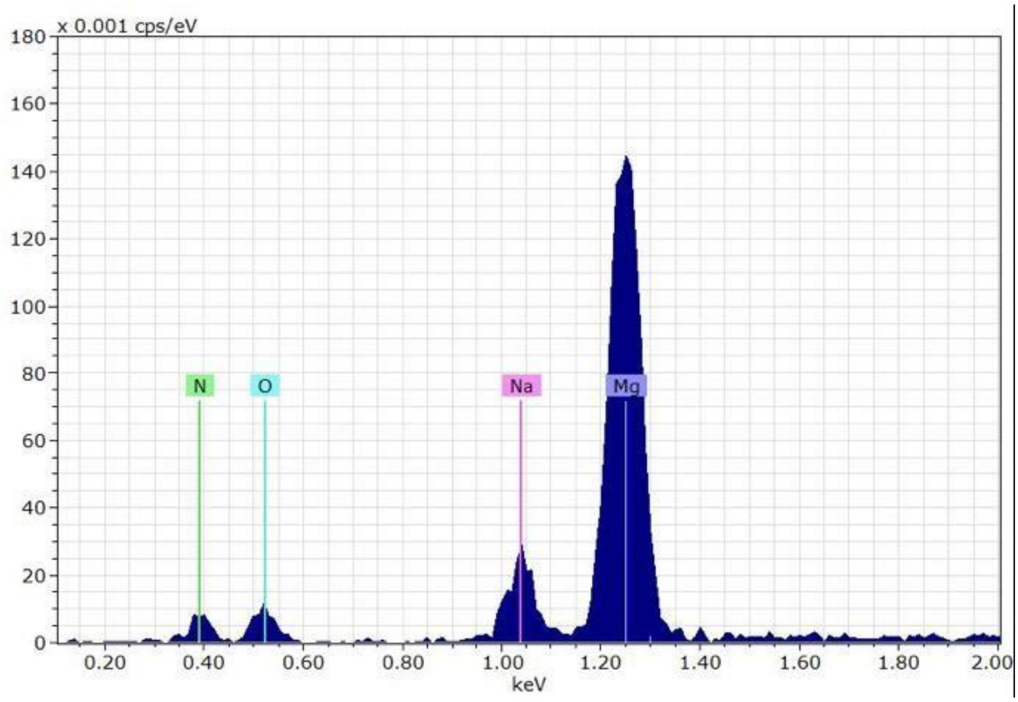


Figure (3.9) (c): EDX spectra for MgO nanoparticles dried at 100ºC.

Figure (3.10), presents peaks between 0.2 and 3.5 k eV, which indicate the presence of Ag NPs, sliver percentage was close to 100 .

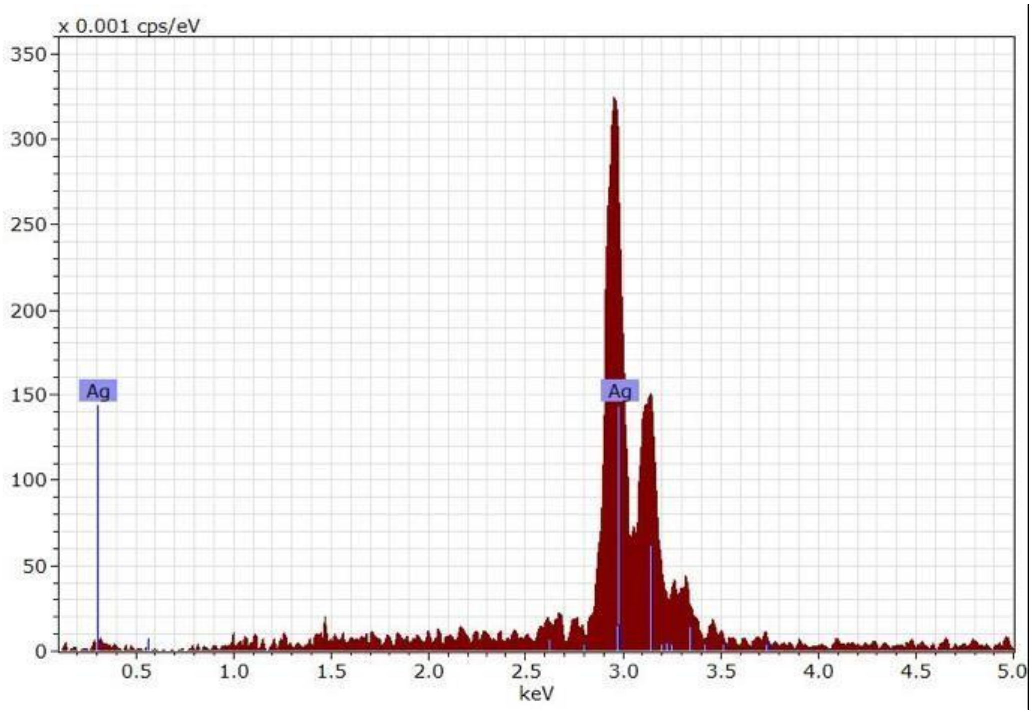


Figure (3.10): EDX spectra for sliver nanoparticles.

3.1.5 Thermal Gravimetric (TG)

Thermo gravimetric analysis is a technique by which we can measures the mass loss with respect to temperature. Figure (3.11) shows the weight loss of grown powder sample. Primary weight loss of ~ 3.5% was observed at 185 ºC for sample dried at 100 ºC , 6% for sample dried at 80 ºC which observed at 120 ºC and 4% for sample dried at 60 ºC which observed at 140 ºC due to the water evaporation. Phase transition is found to occur at about 360ºC indicating the transition from magnesium hydroxide to magnesium oxide with weight loss of ~ 70% for samples dried at 60ºC and 80ºC , while transition phase for sample dried at 100º C occur at 420ºC with weight loss 62% . **(This work agreements with Wahab and Asari).** [117]

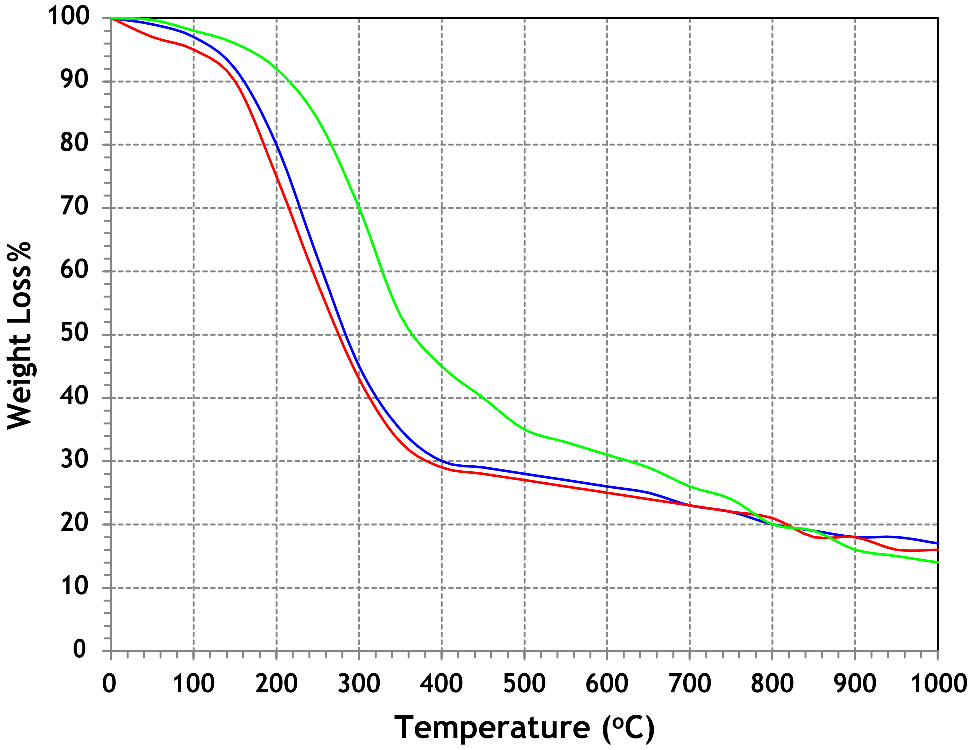


Figure (3.11): Thermal Gravimetric Spectra for MgO nanoparticles

Dried at 60 ºC, 80 ºC, 100 ºC.

3.1.6 Atomic Absorption Analysis

Two types of waste water samples were taken from same field to determine the concentration of heavy metals, the first sample contains only oil and water while the second sample consist of oil, soil and water. The analytical tests (table (3.5) & (3.6)) included the metals: Na, K, Mg, Ca, Hg, Pb, Co, Ni, Zn and Cu. This agreements with other work ***(Abudul-Sattar Al-Hayani)*** [124] .

Table (3.5): Illustrates the concentration of metals prior filtration for sample (1).

Type of Metals	***Conc. [mg/L]***
Na	3.2585
K	Nil.
Mg	0.2845
Ca	0.3828
Hg	Nil.
Pb	Nil.
Co	0.0163
Ni	Nil.
Zn	0.0937
Cu	Nil.

Table (3.6): Illustrates the concentration of heavy metals prior filtrtion for Sample (2).

Type of Metls	***Conc. [mg/L]***
Na	107
K	2.7227
Mg	37.2196
Ca	61.26
Hg	3.262
Pb	Nil.
Co	0.0215
Ni	0.0030
Zn	0.1563
Cu	Nil.

3.1.7 Conductivity Meter Analysis:

This analysis prescribed the measurements of the medium properties such as: Potential of Hydrogen (pH), Electrical Conductivity (EC) which reflects the existence of salts and Total Dissolved Solids (TDS). Measurements for wastewater prior filtration are shown in tables ((3-7)& (3-8)). The analysis has a good agreement with ***(Abudul-Sattar Al-Hayani)*** [124].

Table (3.7): Illustrates pH, EC and TDS measurements for Sample1.

Potential of Hydrogen	*Electrical Conductivity (μc/cm)*	*Total Dissolved Solids (mg/L)*
8.6	*119*	*80*

Table (3.8): Illustrates pH, EC and TDS measurements for Sample2.

Potential of Hydrogen	*Electrical Conductivity (μc/cm)*	*Total Dissolved Solids (mg/L)*
7.8	*343*	*230*

3.1.8 Biological examination for waste water

Two types of wastewater samples has been examined to detected presence of *E. coli* and *Staphylococcus aureus* bacteria the results showed no bacteria grown.

3.2 Application of the synthesized nano-membrane on the wastewater

The treatment of wastewater established by using synthesized nano-membranes to remove heavy metals and oil. The resulted water examined after filtration process by using atomic absorption instrument to ensure the efficiency of membranes work and electro conductivity meter, the results was as follows:

-Atomic Absorption measurements

- Concentration of heavy metals after filtration for Sample1 (which contain water and oil as mentioned previously) were as follow:

Table (3.9): Illustrates concentration of metals prior and after filtration by using membrane coating with MgO NPs.

Element	***Conc. (mg/L) prior filtration***	***Conc. (mg/L) after filtration***
Na	3.2585	203.33
K	Nil.	Nil.
Mg	0.2845	42.2
Ca	0.3828	0.175
Hg	Nil.	Nil.
Pb	Nil.	Nil.
Co	0.0163	0.01
Ni	Nil.	Nil.
Zn	0.0937	0.02
Cu	Nil.	Nil.

Table (3.10): Illustrates concentration of metals prior and after filtration by using membrane coating with Ag NPs.

Element	***Conc. (mg/L) prior filtration***	***Conc. (mg/L) after filtration***
Na	3.2585	343.32
K	Nil.	Nil.
Mg	0.2845	0.1
Ca	0.3828	0.2117
Hg	Nil.	Nil.
Pb	Nil.	Nil.
Co	0.0163	Nil.
Ni	Nil.	Nil.
Zn	0.0937	Nil.
Cu	Nil.	Nil.

Table (3.11): Illustrates concentration of heavy metals prior and after filtration by using membrane coating with a mixture of MgO/Ag NPs.

Element	*Conc. (mg/L) prior filtration*	*Conc. (mg/L) after filtration*
Na	3.2585	413.31
K	Nil.	Nil.
Mg	0.2845	116.6
Ca	0.3828	0.1539
Hg	Nil.	Nil.
Pb	Nil.	Nil.
Co	0.0163	Nil.
Ni	Nil.	Nil.
Zn	0.0937	Nil.
Cu	Nil.	Nil.

- Concentration of heavy metals after filtration for Sample2 (which contain water, oil and soil) were as follow:

Table (3.12): Illustrates concentration of heavy metals prior and after filtration by using membrane coating with MgO NPs.

Element	*Conc. (mg/L) prior filtration*	*Conc. (mg/L) after filtration*
Na	107.7227	230
K	2.7227	0.9257
Mg	37.2196	50
Ca	61.29	1.25
Hg	3.262	0.017
Pb	Nil.	Nil.
Co	0.0215	Nil.
Ni	0.003	0.0022
Zn	0.1563	Nil.
Cu	Nil.	Nil.

Table (3.13): Illustrates concentration of metals prior and after filtration by using membrane coating with Ag NPs.

Element	***Conc. (mg/L) prior filtration***	***Conc. (mg/L) after filtration***
Na	107.7227	180
K	2.7227	0.8378
Mg	37.2196	11.53
Ca	61.29	1.062
Hg	3.262	0.016
Pb	Nil.	Nil.
Co	0.0215	Nil.
Ni	0.003	0.0027
Zn	0.1563	Nil.
Cu	Nil.	Nil.

Table(3.14): Illustrates concentration of metals prior and after filtration by using membrane coating with a mixture MgO/Ag NPs.

Element	***Conc. (mg/L) prior filtration***	***Conc. (mg/L) after filtration***
Na	107.7227	383.4
K	2.7227	10.4
Mg	37.2196	78.8
Ca	61.29	0.687
Hg	3.262	0.002
Pb	Nil.	Nil.
Co	0.0215	Nil.
Ni	0.003	Nil.
Zn	0.1563	Nil.
Cu	Nil.	Nil.

From above atomic absorption spectroscopy results , the heavy metals concentrations were diminished after filtration process by three coated membranes MgO, Ag and MgO-Ag due to release of reactive oxygen species (R.O.S)

-Conductivity meter measurements

- pH, EC and TDS measurements after filtration for sample1(which contain water and oil as mentioned previously) were as follow:

Table (3.15):Illustrates pH, EC and TDS measurements for wastewater filtered by membrane without coating.

Potential of Hydrogen	***Electrical Conductivity (μc/cm)***	***Total Dissolved Solids (mg/L)***
8.6	119	80

Table (3.16): Illustrates pH, EC and TDS measurements for wastewater filtered by membrane coated with MgO NPs.

Potential of Hydrogen	***Electrical Conductivity (μc/cm)***	***Total Dissolved Solids (mg/L)***
9.5	910	610

Table (3.17): Illustrates pH, EC and TDS measurements for wastewater filtered by membrane coated with Ag NPs.

Potential of Hydrogen	***Electrical Conductivity (μc/cm)***	***Total Dissolved Solids (mg/L)***
9.5	925	620

Table (3.18): Illustrates pH, EC and TDS measurements for wastewater filtered

by membrane coated with a mixture of MgO/Ag NPs.

Potential of Hydrogen	*Electrical Conductivity (µc/cm)*	*Total Dissolved Solids (mg/L)*
9.5	1940	1300

- pH, EC and TDS measurements after filtration for sample2 ((which contain water, oil and soil as mentioned previously) were as follow:

Table (3.19): Illustrates pH, EC and TDS measurements for wastewater

filtered by membrane without coating.

Potential of Hydrogen	*Electrical Conductivity (µc/cm)*	*Total Dissolved Solids (mg/L)*
7.8	343	230

Table (3.20): Illustrates pH, EC and TDS measurements for wastewater filtered by membrane coated with MgO NPs.

Potential of Hydrogen	*Electrical Conductivity (µc/cm)*	*Total Dissolved Solids (mg/L)*
9.2	1104	740

Table (3.21): Illustrates pH, EC and TDS measurements for wastewater filtered by membrane coated with Ag NPs.

Potential of Hydrogen	*Electrical Conductivity (μc/cm)*	*Total Dissolved Solids (mg/L)*
9.3	791	530

Table (3.22): Illustrates pH, EC and TDS measurements for wastewater filtered

by membrane coated with MgO/Ag NPs.

Potential of Hydrogen	*Electrical Conductivity (μc/cm)*	*Total Dissolved Solids (mg/L)*
9.2	1552	1040

3.3 Determination membranes efficiency

This step have been repeated filtration process by the same membranes which used previously and then we measured the PH, EC and TDS for resulted water by conductivity meter, the results were as follow:

- Second filtration process by same membranes for (Sample1)

Table (3.23): Illustrates pH, EC and TDS measurements for wastewater filtered by membrane without coating.

Potential of Hydrogen	*Electrical Conductivity (μc/cm)*	*Total Dissolved Solids (mg/L)*
7.8	343	230

Table (3.24): Illustrates pH, EC and TDS measurements for wastewater filtered by membrane coated with MgO NPs.

Potential of Hydrogen	*Electrical Conductivity (µc/cm)*	*Total Dissolved Solids (mg/L)*
9.6	1710	850

Table (3.25): Illustrates pH, EC and TDS measurements for wastewater filtered by membrane coated with Ag NPs.

Potential of Hydrogen	*Electrical Conductivity (µc/cm)*	*Total Dissolved Solids (mg/L)*
9.5	1030	810

Table (3.26): Illustrates pH, EC and TDS measurements for wastewater filtered by membrane coated with MgO/Ag NPs.

Potential of Hydrogen	*Electrical Conductivity (µc/cm)*	*Total Dissolved Solids (mg/L)*
9.2	2240	1760

-Second filtration process by same membranes for (Sample2)

Table (3.27) Illustrates pH, EC and TDS measurements for wastewater filtered by membrane without coating.

Potential of Hydrogen	*Electrical Conductivity (µc/cm)*	*Total Dissolved Solids (mg/L)*
7.8	343	230

Table (3.28): Illustrates pH, EC and TDS measurements for wastewater filtered by membrane coated with MgO NPs.

Potential of Hydrogen	*Electrical Conductivity (µc/cm)*	*Total Dissolved Solids (mg/L)*
9.7	1820	870

Table (3.29): Illustrates pH, EC and TDS measurements for wastewater filtered by membrane coated with Ag NPs.

Potential of Hydrogen	*Electrical Conductivity (µc/cm)*	*Total Dissolved Solids (mg/L)*
9.6	1050	830

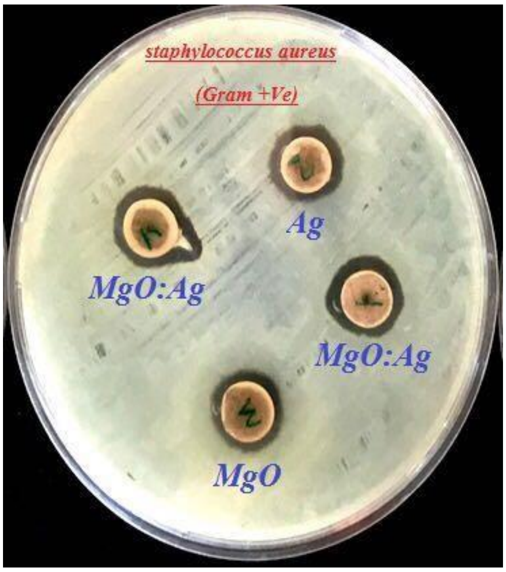
Table (3.30): Illustrates pH, EC and TDS measurements for wastewater filtered by membrane coated with MgO/Ag NPs.

Potential of Hydrogen	*Electrical Conductivity (µc/cm)*	*Total Dissolved Solids (mg/L)*
9.6	2110	1780

During the third filtration process, no drops of water were observed which indicate that the membranes stopped working.

3.4 Application produced nanoparticles on Bacteria

Synthesized nanoparticles were examined to test it’s biological effectiveness on *E.coli* and *Staphylococcus aureus* bacteria which taken from wounds and burns, the culture results was as follows:



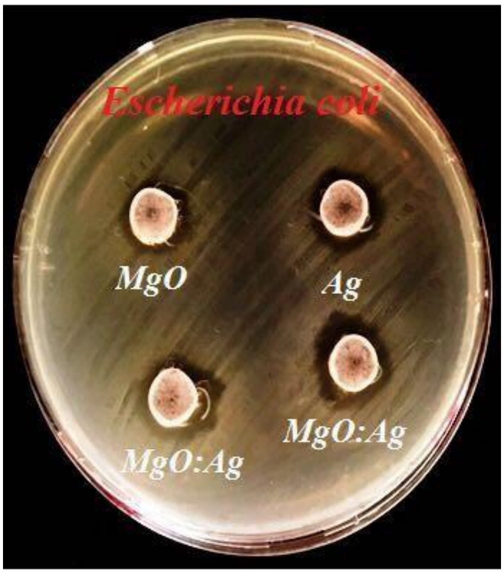


Table (3.31) Shows the inhibition zone measurements for Satphylococcus aureus.

Samples	***Diameter (mm)***
MgO dried at 60 °C	8
MgO dried at 80 °C	15
MgO dried at 100 °C	14
Ag	17
MgO-Ag	20

Table (3.32) Shows the inhibition zone measurements for Escherichia Coli.

Samples	***Diameter (mm)***
MgO dried 60° C	7.5
MgO dried 80°C	16
MgO dried 100 °C	13
Ag	20
MgO-Ag	22

Conclusions and Recommendations

4.1 Conclusions

1. Synthesis of MgO have been done using sol-gel method and Ag NPs have been done using simple chemical method .

2. The products were characterized by various devices and techniques:, FTIR, (XRD), (SEM), EDX and TGA.

3. The water samples were characterized by Atomic absorption spectroscopy (AAS) and Conductivity meter.

4.The activity of the products was examined to remove the heavy metals from waste water by filtration process.

5.The biological activity of the products has been examined in order to inhibit the growth of E.coli and Staphylococcus aureus bacteria.

4.2 Recommendations

1- To study different shapes and sizes of nanomaterials can use it as composite to enhance the chemical and biological activity of MgO and Ag.

2- To study the possibility of using synthesized Ag/MgO as medicine treatment for some diseases such as treatment of dental caries.

3. Study possibility the of using synthesized MgO NPs as ceramic dental cavities.

References

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ملخص

يشكل الماء المصاحب للنفط خلال عمليات التكرير مشكلة حقيقة حيث يعتبر مصدر لنمو البكتريا.

في هذا المشروع البحثي تم تحضير مادة اوكسيد المغنيسيوم باستخدام طريقة الجل-محلول حيث تم الحصول على دقائق حجمها (40 نانومتر)، بينما تم تحضر مادة الفضة النانوية باستخدام الطريقة الكيميائية البسيطة و تم الحصول على دقائق ذات حجم (50.73 نانومتر).

تم تشخيص المواد المحضرة باستخدام الاجهزة التالية:

جهاز المطيافية الذري، مطيافية الاشعة تحت الحمراء، انحراف الاشعة السينية، مجهر المسح الالكتروني، جهاز قياس التوصيلية، تحليل الثقل النوعي الحراري، جهاز تشتت الاشعة السينية.

استخدمت المواد النانوية الناتجة في عملية طلاء الاغشية، بعد ذلك استخدمت هذه الاغشية في عملية تنقية المياه الآسنة المصاحبة للنفط، اظهرت النتائج فعالية هذه المواد النانوية في عملية التنقية والترشيح.

جزء من المواد الناتجة استخدمت لغرض فحص فعاليتها الحيوية ضد البكتريا. وقد اظهرت النتائج فعالية عالية لهذه المواد في قتل البكتريا.

جمهورية العراق
وزارة التعليم العالي والبحث العلمي
جامعة النهرين
كلية العلوم
قسم الكيمياء

تنقية المياه الآسنة الناتجة من آبار النفط خلال عملية التكرير باستخدام مواد نانوية

رسالة

مقدمة الى كلية العلوم \ جامعة النهرين
كجزء من متطلبات نيل شهادة الماجستير في علوم الكيمياء
من قبل
ساره عدنان هادي

اشراف
الاستاذ المساعد الدكتور
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