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Characterization of ammonia removal from municipal wastewater using microwave energy

Master of Science Thesis

by

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DEDICATION

To my parents for their kindness...

To my wife for her encouragement and support

To my beloved son Yahya

To my brothers and sisters

To all knowledge seekers...

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First, thanks to Allah, the compassionate the merciful, for giving me patience and strength to accomplish this research.

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ABSTRACT

Wastewater treatment is a significant issue in environment protection, that includes many topics such as ammonia removal. Microwave (MW) energy irradiation has acquired a great deal of attention in wastewater treatment matters. Recently, it was used to remove ammonia from industrial wastewater. This research investigated the characteristics of ammonia removal from domestic (municipal) wastewater hadn't been studied. In this research, two experiments were carried out using MW energy: batch reactor and continuous reactor.

The batch reactor experiment investigated the basic characteristics of using MW energy in the removal of ammonia nitrogen from municipal wastewater. Thus, three main factors were investigated: initial ammonia concentration, initial pH and radiation time. pH and radiation time showed significant influence on the removal of ammonia nitrogen. The largest removal was obtained at pH 11 in 4 min. Initial ammonia concentration had minute effect. Also, a real municipal wastewater effluent was treated by MW radiation. With the initial ammonia concentration of 67.5 mg/l at pH 11, ammonia was reduced to 5.0 mg/l.

The continuous reactor experiment showed a simulation model of a MW-based ammonia removal system. The initial ammonia concentration and detention time were the investigated affecting factors. Four initial ammonia concentrations were studied: 60, 80, 100 and 120 mg/l, and four detention times were applied: 5.26, 9, 15 and 22 min. The results showed that, for all initial concentrations, detention time of 22 min reduced ammonia nitrogen concentration to lower than 5 mg/l.

The power consumption of the system was relatively high, which will limit its use in the treatment of toxic wastewater streams. Nevertheless, the power consumption can be reduced significantly using efficient heat exchangers, which would lower down the required energy for heating. However, there are many side benefits that can be achieved when using MW energy, such as collection of ammonia gas and heating buildings.

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List of Abbreviations

APHA	American Public Health Association
AWWA	American Water Works Association
BOD	Biochemical Oxygen Demand
BRH	Bureau of Radiological Health
°C	Celsius
CaCO₃	Calcium Carbonate
cm	Centimeter
CMWU	Coastal Municipalities Water Utility
C/N	Carbon to Nitrogen ratio
COD	Chemical Oxygen Demand
DO	Dissolved Oxygen
EQA	Environment Quality Authority
EU	European Union
°F	Fahrenheit
Fe⁺³	Ferric ions
F:M	Food to Microorganism ration
FRWA	Florida Rural Water Association
g	grams
GHz	Giga Hertz
GWTP	Gaza Waste Water Treatment Plant
H⁺	Hydrogen ions
JISM	Jordanian Institution for Standards and Metrology
K	Kelvin
kW	kilo Watt
L/min	Liter per minute

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m³	Cubic meter
MAP	Magnesium Ammonium Phosphate
MCRT	Mean Cell Residence Time
mg/L	Milligram per Liter
mg-N/L	Milligram Nitrogen per Liter
ml	Milliliter
mm	Millimeter
mol/L	Mole per Liter
MW	Microwave
mW/cm²	milliwatt per square centimeter
NaHCO₃	Sodium Bicarbonate
NH₃	Ammonia
NH₄⁺	Ammonium
NH₄-N	Ammonium Nitrogen
NO₂⁻	Nitrite
NO₃⁻	Nitrate
SRT	Solids Retention Time
T_{in}	Influent Temperature
TKN	Total Kjeldahl Nitrogen
T_{out}	Effluent Temperature
TSS	Total Suspended Solids
UK	United Kingdom
USD	United States Dollar
VOC's	Volatile Organic Carbons
W	Watt
WEF	Water Environment Federation
WHO	World Health Organization

CHAPTER 1: Introduction

1.1. Background

Environmental protection is a significant matter that occupies a large area of global concern. Many issues are related to environmental protection, such as energy conservation, ecosystem reserve, water purification and wastewater treatment. Wastewater is any water that has been adversely affected in quality by anthropogenic influence. Simply, it is any water that has been used, and can't be used for the same purpose.

No one can deny that wastewater is one of the main sources of pollution which threatens environment, and should be disposed in a safe way. In addition, it usually contains various pollutants, depending on what it was used for.

In general, wastewater can be classified into two major categories, by source:

❖ Domestic, municipal or household wastewater:

This type of wastewater comes from residential sources including toilets, sinks, bathing, and laundry. It can contain body wastes containing intestinal disease organisms. There are four broad types of wastewater from the household, which can be characterized as follow:

Table (1.1) Types of wastewater from the household

Type of wastewater	Source of wastewater
Gray water	Washing water from kitchen, bathrooms, laundry, etc. without faeces and urine
Black water	Water from flush toilets (faeces and urine with flush water)
Yellow water	Urine from separation toilets and urinals (with or without water for flushing)
Brown water	Black water without urine or yellow water

(Winnerberger, 1969)

The strength of wastewater depends mainly on the degree of water pollutance, which can be categorized as strong, medium, or weak, as shown in Table (1.2). These wastewater characteristics can vary widely with local conditions, hour of the day, day of the week, season, and type of sewers (either separate or combined sewers where storm water is included).

Table (1.2) Types of wastewater from the household

Parameter	Concentration (mg/l)		
	Strong	Medium	Weak
BOD₅	400	220	110
COD	1,000	500	250
Org-N	35	15	8
NH₃-N	50	25	12
Total N	85	40	20
Total P	15	8	4
Total solids	1,200	720	350
Suspended solids	350	220	100

(Polprasert, 1996)

❖ **Industrial wastewater:**

This type is discharged by manufacturing processes and commercial enterprises. Process wastewater can contain rinse waters including such things as residual acids, plating metals, and toxic chemicals. Industrial wastewater characteristics vary according to the industry type, but, in general, ammonia concentration is higher than in the domestic wastewater.

Wastewater treatment is a process to improve and purify the water, removing some or all of the contaminants, making it fit for reuse or discharge back to the environment. Discharge may be to surface water, such as rivers or the ocean, or to groundwater that

lies beneath the land surface of the earth. Properly treating wastewater assures that acceptable overall water quality is maintained.

In many parts of the world, health problems and diseases are often been caused by discharging untreated or inadequately treated wastewater. Such discharges are called water pollution, and result in the spreading of disease, fish kills, and destruction of other forms of aquatic life.

1.2. Ammonia

Ammonia (NH_3) is one of the main contaminants presented in the municipal wastewater. It is formed by bacteria decomposition processes of organic matter (Sørensen & Jørgensen, 1993). Most of quality standards of the treated wastewater specified ammonia concentration in the treated-recharged wastewater to be less than 5.0 mg-N/L (JISM, 2006).

1.2.1. Impacts of Ammonia

Ammonia impacts are basically categorized into two substantial types:

I. Environmental impacts:

Ammonia contribute to several environmental impacts including eutrophication of surface water, soil acidification, fertilization of vegetation and changes in ecosystem.

a. Eutrophication:-

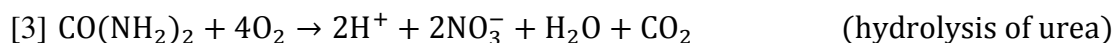
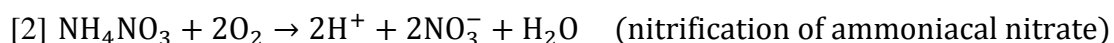
Eutrophication is a result of nutrient pollution into natural waters (creeks, rivers, ponds, or lakes). Eutrophication generally promotes excessive plant growth and decay, favors certain weedy species over others, and is likely to cause severe reductions in water quality. In aquatic environments, enhanced growth of choking aquatic vegetation or algal blooms disrupt normal functioning of the ecosystem, causing problems such as a lack of oxygen in the water, needed for fish and other aquatic life to survive. The water then becomes cloudy, colored a shade of green, yellow, brown, or red.

b. Soil acidification:-

When ammonia reaches the soil surface, it usually reacts with water in the soil and is converted into its ionic form, ammonium (NH_4^+), and absorbed to the soil. The

ammonium in the soil eventually disassociates or is nitrified into nitrite (NO_2^-) or nitrate (NO_3^-) by nitrifying bacteria, releasing hydrogen ions (H^+) into the soil. If not taken up by biomass and converted to methane, the surplus H^+ ions eventually lead to the formation of an acidic soil environment. The nitrogen left over in the soil will either be taken up by plants, stored in the soil, returned to the atmosphere, or will be removed from the soil in runoff or leaching.

For fertilizers, the most important acid forming reaction is microbial oxidation of ammoniacal fertilizers by the following reactions (Barak, 2000):



c. Changes in ecosystem:-

An ecosystem is a natural system consisting of plants, animal, and other microorganisms functioning together in a balanced relationship. Changes in ecosystems due to ammonia deposition occur through a combination of all the above mentioned processes. When changes in ecosystems occur, the natural balance of a system is disrupted and fragile plant and animal species can be replaced by non-native or N-responsive species. The disruption of an ecosystem can cause it to adapt by changing (positive or negative outcome), or a disruption may lead to the extinction of the ecosystem.

So, ammonia should be properly removed from wastewater streams to protect environment and save its components.

II. Human health impacts:

Ammonia affects human and animal health both as a gas and as a particulate. The particulate form of ammonia has broader implications for the general public, where as the gaseous form is a localized concern for the health of animals and agricultural workers. The immediate health effects of ammonia exposure can be summarized as follows:

a. Inhalation:-

Ammonia is irritating and corrosive. Exposure to high concentrations of ammonia in air causes immediate burning of the nose, throat and respiratory tract. This can cause

bronchiolar and alveolar edema, and airway destruction resulting in respiratory distress or failure. Inhalation of lower concentrations can cause coughing, and nose and throat irritation. Ammonia's odor provides adequate early warning of its presence, but ammonia also causes olfactory fatigue or adaptation, reducing awareness of one's prolonged exposure at low concentrations.

Children exposed to the same concentrations of ammonia vapor as adults may receive a larger dose because they have greater lung surface area-to-body weight ratios and increased minute volumes-to-weight ratios. In addition, they may be exposed to higher concentrations than adults in the same location because of their shorter height and the higher concentrations of ammonia vapor initially found near the ground.

b. Skin or eye contact:-

Exposure to low concentrations of ammonia in air or solution may produce rapid skin or eye irritation. Higher concentrations of ammonia may cause severe injury and burns. Contact with concentrated ammonia solutions such as industrial cleaners may cause corrosive injury including skin burns, permanent eye damage or blindness. The full extent of eye injury may not be apparent for up to a week after the exposure. Contact with liquefied ammonia can also cause frostbite injury.

c. Ingestion:-

Exposure to high concentrations of ammonia from swallowing ammonia solution results in corrosive damage to the mouth, throat and stomach. Ingestion of ammonia does not normally result in systemic poisoning.

1.3. Microwave historical background

Prior to the invention, microwave energies were purely used to detect Nazi warplanes by British bombers during World War II. However, Like many of today's great inventions, the microwave oven was a by-product of another technology. It was during a radar-related research project around 1946 that Dr. Percy Spencer, a self taught engineer with the Raytheon Corporation, noticed something very unusual. He was testing a new vacuum tube called "**magnetron**" (discussed in Chapter 2), when he discovered that the candy bar in his pocket had melted. This intrigued Dr. Spencer to carry out more experiments, until the first Raytheon commercial microwave oven was released in the year 1954. This microwave oven was almost as big as a

refrigerator. Due to its large size and steep price (around USD 5,000), its use was limited to hotels and institutions alone. The first microwave oven was used in a Boston restaurant and was tested successfully. It was only in 1967, that the first domestic microwave oven, fitted for kitchen use was introduced.

After that, microwave technique was developed considerably for various applications such as telecommunication transmissions, radar detection, radio astronomy, food production, cooking, medical applications and also environmental applications.

1.4. Statement of the problem

One of the main processes that should be applied in the wastewater treatment is ammonia removal, which is complex and expensive. Moreover, in municipal wastewater effluents, ammonia concentrations reach higher than 80 mg-N/L in some areas, like Gaza Strip (CMWU, 2008), and this costs much money and energy to be removed. Besides, ammonia removal is essential to achieve the standard level of wastewater treatment.

Several researches investigated the effect of MW radiation through different kinds of applications, like boron removal and recovery and organic pollutants degradation.

Effect of MW energy on ammonia removal was studied for only industrial wastewater, but the characteristics of ammonia removal from municipal wastewater by MW energy are still unknown, which is the main task of this research.

1.5. Objectives

The main goal of this research is to investigate the characteristics of ammonia nitrogen removal from municipal wastewater using MW radiation. Moreover, this research is supposed to achieve the following objectives:

- A. To study the effect of MW radiation on removing ammonia nitrogen from municipal wastewater, in different temperature, pH value and initial ammonia concentration conditions.
- B. To investigate the economic costs and benefits of operating a MW-based system to remove and recover ammonia from municipal wastewater.

1.6. Methodology

To achieve the objectives of the research, the following methodology was applied:

1) Literature review:

Including revision of:

- Books and websites in the field of ammonia removal methods.
- Scientific papers and reports in the field of microwave applications in wastewater.

2) Laboratory experiments:

Laboratory experiments were applied on both synthetic and real wastewater samples. The samples were heated using domestic microwave oven for different time intervals, different ammonia nitrogen concentrations (low concentrations) and different pH values. Moreover, two types of experiments were applied: batch and continuous.

3) Results and analysis:

Ammonia nitrogen concentration was measured, and the optimum condition of ammonia removal was estimated for both batch and continuous experiments, for real and synthetic wastewater.

4) Economic aspects study:

A brief feasibility study of ammonia removal-and-recovery system in a wastewater treatment plant were conducted. This was accomplished through surveying economic costs and benefits that affect the efficiency of the system, focusing on operation costs that could be caused by power consumption, and chemical additives.

The following chart illustrates the proposed methodology:

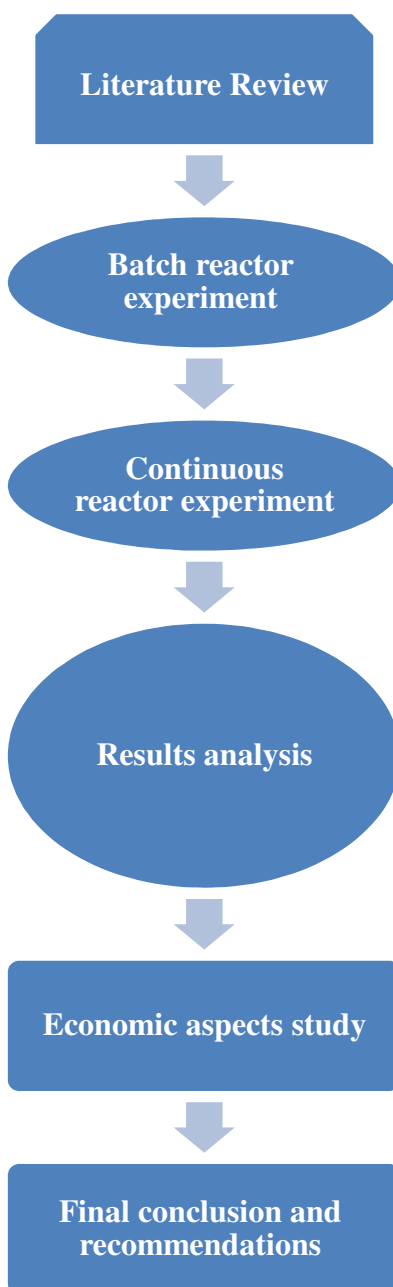


Fig. (1.1) Methodology

1.7. Thesis structure

The structure of the thesis consists of six chapters arranged carefully, to make the research clear and understandable. This section presents a concise description of these chapters.

Chapter (1) is a briefly introduction, which presents a general background about wastewater, ammonia and microwave. In addition, statement of problem, objectives, scope of work and methodology are given.

Previous researches and studies about ammonia removal techniques and standard limits are illustrated in *Chapter (2)*. This chapter gives a general overview of relevant previous researches concerning microwave technology applications in several fields including wastewater treatment.

Chapter (3) outlines the microwave batch reactor experiment, with the related results and analyses. Factors affecting ammonia removal are studied during this chapter. These factors include pH, initial concentration and radiation time.

Chapter (4) presents the microwave continuous experiment, with the achieved results and analysis. Also, related factors are investigated, including detention time, initial ammonia concentration and pH.

Chapter (5) investigates the economic aspects of using a MW-based system for removal and recovery of ammonia from municipal wastewater. This chapter explores in brief the economic costs and benefits of MW energy, including running cost, capital cost and economic benefits.

The conclusion derived from experimental results is presented in *Chapter (6)*. Finally, the recommendations for the present study and other further studies are also provided in this chapter.

CHAPTER 2: Literature review

2.1. Introduction

Nitrogen is one of the main nutrients presented in wastewater in a variety of forms, because of the various oxidation states represented, and it can readily change from one state to another depending on the physical and biochemical conditions present. The total nitrogen concentration in typical municipal wastewaters ranges from about 15 to over 50 mg/L. About 60% of this is in ammonia form, and the remainder is in organic form. (Crites, *et al.*, 2006)

Nitrogen compounds are major pollutants, which enter water bodies through aqueous wastes from several key industries (e.g. fertilizer, fish canning, refinery, and tannery), agricultural run-off and domestic wastes. (Mahne, *et al.*, 1996)

Nitrogen appears in wastewater as ammonia (NH_3 as a gas or NH_4^+ ions), nitrite (NO_2^-), nitrate (NO_3^-) and organic nitrogen (urea, fecal material). Total Kjeldahl Nitrogen (TKN) is the combination of ammonia and organic nitrogen.

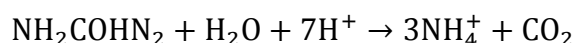
2.2. Ammonia

One of the common nitrogen compounds is "**Ammonia**". It is a colorless gas that is used in the manufacture of chemical products such as fertilizers, nitric acid and plastics and in refrigeration plants and petroleum refineries. It is present in most waters as a result of the biological degradation of nitrogenous organic matter, although it may also reach groundwater and surface waters from industrial waste discharges. (Health Canada, 1996)

Proteins and waste products from animals and humans, urea and uric acid, are decomposed to simpler organic molecules such as amino acids, which again are decomposed to ammonia. (Sørensen & Jørgensen, 1993).

Ammonia can be present in wastewater as molecular ammonia (NH_3) or as ammonium ions (NH_4^+). The equilibrium between these two forms in water and wastewater is strongly dependent on pH and temperature. At pH 7 essentially only ammonium ions are present, while at pH 12 only dissolved ammonia gas is present. (Crites, *et al.*, 2006)

While travelling through sewer pipes, the majority nitrogen contained in raw sewage (urea and fecal material) is converted from organic nitrogen to ammonia through a process called hydrolysis. The process is anaerobic and is described by the following equation:



It should be noticed that the previous equation shows the conversion of urea to ammonium, not ammonia. The ratio of ammonia (NH_3) to ammonium (NH_4^+) is affected by pH and temperature. At conditions typical for most wastewater treatment plants (pH of 6 to 7, and temperature of 10 °C to 20 °C), far more ammonium than ammonia is produced. Since ammonia and ammonium behave similarly, this fact is of no real consequence to treatment plant designers and operators.

Ammonium ions in wastewater exist in equilibrium with gaseous ammonia as shown in the equation below (Lin, *et al.*, 2009):



2.2.1. Ammonia standard limits

As nitrogen pollution has become a cause for concern, many countries and organizations had enforced stringent standards in recent years for nitrogen discharge.

Typical effluent permit limits for nitrogen compounds in wastewater effluent vary, but all are based on location of final effluent discharge. A wastewater plant that discharges to a spray field may not have a limit on nitrogen while a plant that discharges to percolation ponds may have an effluent nitrate limit of 12 mg/L. A treatment plant that discharges to a nearby stream or river may have a total nitrogen limit of 3 mg/L, or an unionized ammonia limit of 0.2 mg/L. (FRWA, 2008)

Specifically, ammonia can be normally found in fresh water, surface water or ground water. The World Health Organization (WHO) stated that the acceptable range of ammonia normally founded in water is 0.2-0.3 mg/L. In wastewater effluent, WHO recommended concentration of ammonia nitrogen to be 1.5 mg/L for potential wastewater stream. (WHO, 2008)

According to the Palestinian and Jordanian standards for treated wastewater, the acceptable concentration of ammonia nitrogen ($\text{NH}_4\text{-N}$) in the treated wastewater, for recharging purpose, is 5.0 mg-N/L. (EQA, 2008; JISM, 2006)

The organizations concerned with drinking water quality and many governments have paid a great attention for ammonia limitation in the drinking water. Table (2.1) lists some international standards of ammonia concentration in drinking water according to some international water quality guidelines. It can be noticed that all countries and organizations restricted ammonia concentration in drinking water within the range 0.3-1.5 mg/L, which indicates that ammonia is a matter of concern that should be treated well.

Table (2.1) International standards of ammonia concentration in drinking water

Country/Organization	Max allowable ammonia concentration (mg/l)	Reference
World Health Organization (WHO)	1.5	<i>WHO, 2008</i>
European Union (EU)	0.3	<i>European Commission Environment, 2000</i>
Australia	0.5	<i>Australian drinking water guidelines, 2004</i>
New Zealand	1.5	<i>Ministry of Health, New Zealand, 2008</i>
Palestine	1.5	<i>EQA, 2010</i>
Malaysia	0.5	<i>Food Act, 1983</i>
Vietnam	1.5	<i>Drinking water standards for Vietnam, 2002</i>

In order to achieve these limits, wastewater treatment plants should be improved in order to convert nitrogen compounds to less noxious forms.

2.2.2. Ammonia applications and uses

Ammonia is a high-value product, that has many uses in many different applications. The following are the main uses of ammonia (R.M. Technologies, 2003):

- 1- Ammonia is used in several areas of water and wastewater treatment, such as pH control, in solution form, to regenerate weak anion exchange resins, in conjunction with chloride to produce potable water, and as an oxygen scavenger in boiler water treatment.
- 2- Ammonia is used in the production of liquid fertilizer solutions which consist of ammonia, ammonium nitrate, urea and aqua ammonia. It is also used by the fertilizer industry to produce ammonium and nitrate salts.
- 3- Ammonia and urea are considered as a source of protein in livestock feeds for ruminating animals such as cattle, sheep and goats. Ammonia can also be used as a pre-harvest cotton defoliant, an anti-fungal agent on certain fruits and as preservative for the storage of high-moisture corn.
- 4- Dissociated ammonia can be used in metal treating operations as nitriding, carbonitriding, bright annealing, furnace brazing, sintering, sodium hydride descaling, atomic hydrogen welding and other applications where protective atmospheres are required.
- 5- Ammonia is used in the manufacture of nitric acid; certain alkalis such as soda ash; dyes; pharmaceuticals such as sulfa drugs, vitamins and cosmetics; synthetic textile fibers such as nylon, rayon and acrylics; and for the manufacture of certain plastics such as phenolics and polyurethanes.
- 6- The petroleum industry utilizes ammonia in neutralizing the acid constituents of crude oil and for protection of equipment from corrosion. Ammonia is used in the mining industry for extraction of metals such as copper, nickel and molybdenum from their ores.
- 7- Ammonia is a widely used refrigerant in industrial refrigeration systems found in the food, beverage, petro-chemical and cold storage industries.
- 8- The decomposition of ammonia serves as a source of hydrogen for some fuel cell and other applications.
- 9- The pulp and paper industry uses ammonia to pulp wood, and as a casein dispersant in the coating of paper.

Thereafter, developing a system capable to remove and recover ammonia from wastewater would be economically beneficial.

2.2.3. Ammonia removal methods

Development of economical and sustainable techniques for reducing the nitrogen content from wastewaters had attracted a great deal of attention. Several methods can be applied to remove ammonia from wastewater, such as biological denitrification (Sotirakou, *et al.*, 1999; Holman & Wareham, 2005), ammonia-stripping (Guštin & Logar, 2011), chemical precipitation (Quan, *et al.*, 2010) and ion exchange (Jorgensen & Weatherley, 2003). These methods vary in their pros and cons. The most commonly applied are:

A. Biological nitrification-denitrification:

Biological nitrification-denitrification is one of the most commonly used process for nitrogen removal from wastewater. Nitrification is actually a two-step process that convert ammonia/ammonium to nitrate. Bacteria, known as "Nitrosomonas", convert ammonia and ammonium to nitrite. Next, bacteria called *Nitrobacter* finish the conversion of nitrite to nitrate. The reactions are generally coupled and proceed rapidly to the nitrate form; therefore, nitrite levels at any given time are usually low. These bacteria, known as "nitrifiers", are strict "aerobes"; meaning they must have free dissolved oxygen to perform their work. Nitrification occurs only under aerobic conditions at dissolved oxygen levels of 1.0 mg/L or more. Nitrification requires a long retention time, a low food to microorganism ratio (F:M), a high mean cell residence time (measured as MCRT or Sludge Age), and adequate buffering (alkalinity). Temperature, as discussed below, also plays a role. (Wang, *et al.*, 2008)

The nitrification process produces acid. This acid formation lowers the pH of the biological population in the aeration tank and, because it is toxic to nitrifiers, can cause a reduction of the growth rate of nitrifying bacteria. The optimum pH for *Nitrosomonas* and *Nitrobacter* is between 7.5 and 8.5; however most treatment plants are able to effectively nitrify with a pH of 6.5 to 7.0. Nitrification stops at a pH below 6.0. An alkalinity of no less than 50-100 mg/L in the aeration tank is generally required to insure adequate buffering. (Zhao, *et al.*, 1999)

Water temperature also affects the rate of nitrification. Nitrification reaches a maximum rate at temperatures between 30 and 35°C (86°F and 95°F). At temperatures

of 40°C (104°F) and higher, nitrification rates fall to near zero. At temperatures below 20°C, nitrification proceeds at a slower rate, but will continue at temperatures of 10°C and less. However, if nitrification is lost in low temperature wastewater, it will not resume until the temperature increases to well over 10°C. (Crites, *et al.*, 2006)

Compared to autotrophic nitrifiers, heterotrophic nitrifiers tend to grow more rapidly with higher yield, require lower dissolved oxygen concentration and tolerate a more acidic environment. Therefore, the best environmental conditions for heterotrophic nitrifiers' growth are (Zhao, *et al.*, 1999):

- (1) low dissolved oxygen (DO) concentration,
- (2) high C/N ratio,
- (3) short solids retention time (SRT), and
- (4) acidic environment.

For every gram of ammonia oxidized to nitrate, the following occurs (Sørensen & Jørgensen, 1993):

- 4.18 g of oxygen are consumed.
- 7.14 g of alkalinity are consumed measured as calcium carbonate (CaCO_3), or 12g of alkalinity measured as sodium bicarbonate (NaHCO_3)

Biological nitrification treatment is applied through many forms, such as conventional activated sludge (Hu, *et al.*, 2003), sequencing batch reactor (Wang, *et al.*, 2010), trickling filters (Parker, *et al.*, 1997), membrane bioreactor (You & Chen, 2008), lagoon systems (Conkle, *et al.*, 2008) and oxidation ditches (Lian-feng, *et al.*, 2011).

The main disadvantage of biological method is that it does not respond well to shock loads of ammonia (Jorgensen & Weatherley, 2003), so it is suitable only for relatively low ammonia concentrations.

In addition, biological treatment systems are adversely affected by cold temperatures and changes in effluent characteristics. (Zhao, *et al.*, 1999)

B. Air stripping:

Air stripping is a process that involves the mass transfer of gas from the liquid phase to the gas phase. The removal of dissolved gases from wastewater by air stripping has received considerable attention, especially for the removal of ammonia and odorous gases and volatile organic carbons (VOC's). The removal of ammonia from

wastewater by air stripping requires that the ammonia be present as a gas. (Metcalf & Eddy, 2003).

The efficiency of the process depends on (Sørensen & Jørgensen, 1993):

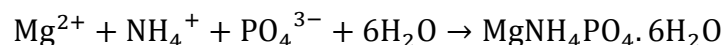
1. pH. It is needed to be higher than 11.
2. The temperature. Preferred to be in the range of 30-40 °C.
3. The quantity of air per m³. At least 3,000 m³ of air per m³ of water are required.
4. The height of the stripping tower.

Guštin and Logar (2011) studied the effect of pH, temperature and air flow rate on the removal of ammonium from an anaerobic wastewater treatment plant effluent. The researchers used ammonia stripping bench plant, which removed up to 92.8% of ammonium and 88.3% of total nitrogen from the anaerobic digestion effluent. They observed that high pH had the most significant effect on ammonia stripping, since it cause the change of ammonia/ammonium ratio in favor of ammonia. The second significant effect was caused by the flow rate of the air passing through the stripping bench plant promoting the transition of ammonia from liquid phase to gas phase. The temperature within the examined range (i.e. 353 K \approx 80°C) had the least significant effect on ammonia stripping, as mentioned by the authors.

C. Chemical precipitation:

Chemically, precipitation is the transition of a substance from the dissolved state to the non-dissolved state by the addition of other reagents that lead to the formation of precipitates. (Sørensen & Jørgensen, 1993)

The precipitation of NH₄⁺-N by forming magnesium ammonium phosphate (MAP) precipitate is the most common chemical precipitation approach used for the ammonium elimination. The basic chemical reaction to form MAP is expressed in the following equation (Zhang, *et al.*, 2009):



This method has been studied and practiced for different types of wastewater, such as tannery effluent in leather industries, digester supernatant in wastewater plants and also sludge liquor. (Li, *et al.*, 1999)

D. Ion Exchange:

Ion exchange is a reversible reaction in which a charged ion in solution is exchanged for a similarly charged ion electrostatically attached to an immobile solid particle. Practically, raw water (or wastewater) is passed through a bed of resin, which is often natural zeolite. (Davis, 2010) Ammonium exchange is accomplished using a naturally occurring zeolite; clinoptilolite (Metcalf & Eddy, 2003), which has a high affinity for ammonium ion. (Jorgensen & Weatherley, 2003)

In common practice, ion exchange method is used in water treatment for softening, where calcium, magnesium and other polyvalent cations are exchanged for sodium. However, ion exchange has been used in wastewater applications for the removal of nitrogen, heavy metals and total dissolved solids. (Metcalf & Eddy, 2003)

Jorgensen and Weatherley (2003) investigated the ammonia removal from wastewater by ion exchange in the presence of organic contaminants. The results showed that in most of the cases studied, the presence of organic compounds enhances the uptake of ammonium ion onto the ion exchangers. Also, they reported that ion exchange offers a number of advantages including the ability to handle shock loadings and the ability to operate over a wider range of temperatures.

Ion exchange process has some disadvantages in that there are substances occurring in some water (such as organic matter or Fe^{+3} ions) which can foul the resin, but in general the advantages of the process (long life of resins, cheap maintenance etc.) outweigh the disadvantages. In addition, the process is very environmentally friendly because it deals only with substances already occurring in water. (Oxy Chemicals Co. Ltd., 2006)

2.3. MW theory and heating mechanism

MW is a part of the electromagnetic spectrum occurring in the frequency range of 300 MHz to 300 GHz as shown in figure (2.1). Domestic microwave ovens operate at 2.45 GHz and industrial processing systems generally use either 2.45 GHz or 915 MHz. (Datta & Anantheswaran, 2001)

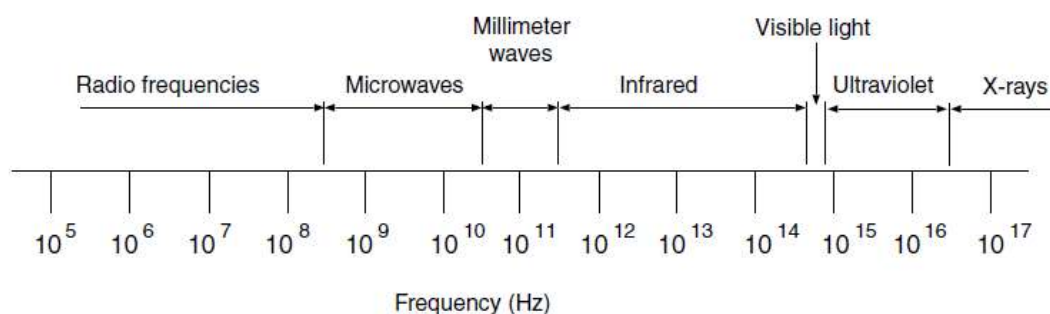


Fig. (2.1) Electromagnetic spectrum

(Datta & Anantheswaran, 2001)

The magnetron, shown in figure (2.2), is a high-power vacuum tube that generates microwaves using the interaction of a stream of electrons with a magnetic field.

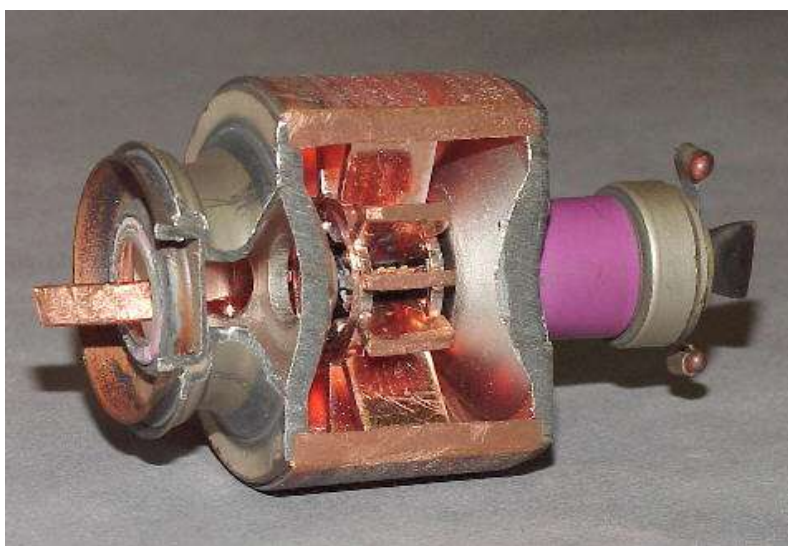


Fig. (2.2) Magnetron

2.3.1. Heating mechanisms

In microwave oven, materials (food, water, etc.) may be heated with use of high frequency electromagnetic waves. The heating arises from the interaction of electric field component of the wave with charge particle in the material. Two basic principal mechanisms involve in the heating of materials. (Dubey, 2008)

A. Dipolar Polarization mechanism:

Dipolar polarization is the phenomenon responsible for the majority of microwave heating. It depends upon nature (Polarity) of solvent and compound. An individual polar molecule has different electro-negativities that result permanent electric dipole,

and is sensitive to external electric fields which, with radiation, will attempt to align with them rapidly by rotation. However, in liquids, the instantaneous alignment is prohibited by the presence of other molecules. (Dubey, 2008)

A limit is therefore place on the ability of the dipole to respond to a field, which affects the behavior of the molecule with different frequency of electric field. For example, under low frequency irradiation, the dipole may react by aligning itself in phase with the electric field. Molecule will polarize uniformly, and no random motion result. Whilst some energy is gained in the molecule by this behavior, and some is lost in collisions, the overall heating effect is small. Under high frequency irradiation the polar molecule will attempt to follow the field, but intermolecular inertia stops any significant motion before the field has reversed, the dipole do not have sufficient time to respond the field, and so do not rotate. As no motion is induced in the molecules, no energy transfers take place, and therefore, no heating. (Dubey, 2008)

In intermediate frequency the field will be such that the molecule is almost, but not quite, able to keep in phase with the field polarity. The microwave frequency is low enough that the dipoles have time to respond to the alternating field, and therefore to rotate, but high enough that the rotation does not precisely follow the field. As the dipole reoriented to align itself with the field, the field is already changing, and a phase difference causes energy to be lost from the dipole in random collisions, and to give rise to dielectric heating, shown in figure (2.3). (Dubey, 2008)

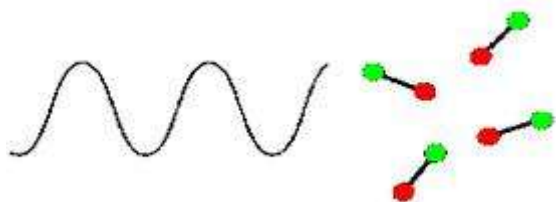


Fig. (2.3) Microwave heating by dipolar polarization mechanism

(Dubey, 2008)

B. Conduction Mechanism:

Where the irradiated sample is an electrical conductor, the charge carriers (electrons, ions, etc.) are moved through the material under the influence of electric field (E), resulting in a polarization (P). These induced currents will cause heating in the sample due to any electric resistance, as shown in figure (2.4). (Dubey, 2008)

For a very good conductor, complete polarization may be achieved in approximately within 10-18 seconds. (Dubey, 2008)

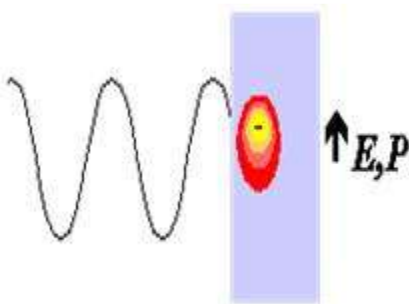


Fig. (2.4) Microwave heating by conduction mechanism

(Dubey, 2008)

2.4. Emission limits

Microwave emission limit was put by the US Bureau of Radiological Health (BRH). The BRH standards tighten the emission limit and adopt a standard that specified an emission limit of 1 mW/cm^2 at 5 cm at manufacture and 5 mW/cm^2 after sale. In 1971, the 5 mW/cm^2 emission standard was adopted in Australia by the National Health and Medical Research Council. In 1976, the International Electrotechnical Commission issued an emission standard of 5 mW/cm^2 at 5 cm. (Bangay & Zombolas, 2004)

2.5. MW applications and uses

MW is a promoted technique in environmental treatment fields. In recent years, MW was investigated for several purposes like boron removal and recovery from wastewater (Tsai & Lo, 2011), ammonia removal from industrial wastewater (Lin, *et al.*, 2009), sewage sludge stabilization (Hsieh, *et al.*, 2007), sludge-methane production (Eskicioglu, *et al.*, 2009) and contaminated soil remediation (Abramovitch, *et al.*, 2003).

This literature review focuses on the applications of MW energy in wastewater treatment only, since other applications are not a matter concern in this research.

2.5.1. Applications of MW energy in wastewater treatment

Many researches and studies were done on wastewater treatment, targeting removal or recovery several pollutants using MW energy, either alone, or coupled with oxidants and catalysts.

I. Using MW alone in wastewater treatment:

Lin, *et al.*, (2009) used MW radiation (750W output MW oven) to remove high-concentrated ammonia nitrogen from both simulated and real wastewater samples through a bench-scale study. Figure (2.5) shows a schematic diagram of the system.

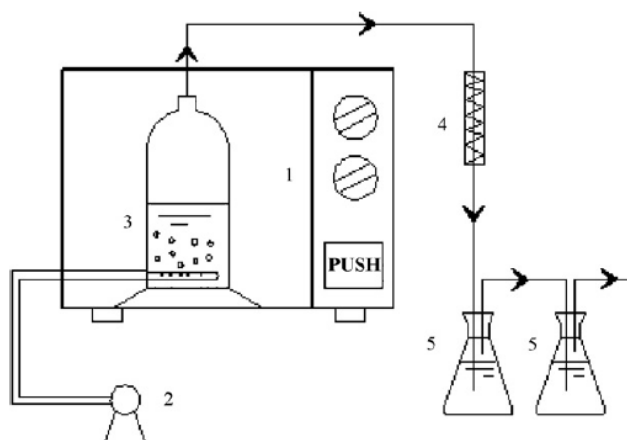


Fig. (2.5) Schematic diagram of the bench-scale MW system

(1) MW oven, (2) air compressor, (3) glass reactor, (4) condenser, (5) absorption vessel
filled with H_2SO_4

(Lin, *et al.*, 2009)

They used simulated wastewater with high ammonia nitrogen concentrations, which was similar to the industrial wastewater. The concentrations ranged from 500 mg/L to 12,000 mg/L. They investigated four factors which were thought to affect ammonia removal: radiation time, initial pH, initial ammonia concentration and aeration conditions. The experiment results showed that higher pH and MW radiation time resulted in larger ammonia removal. However, the variation in aeration conditions and initial ammonia concentration represented minute effect. For simulated wastewater, the largest removal efficiency was 98.2% obtained at pH 11 in 3 minutes with initial ammonia nitrogen concentration of 500 mg/L, as shown in Figure (2.6). While for real wastewater with initial ammonia nitrogen concentration of 5,000 mg/L and pH 11, 93% removal was achieved in 10 minutes.

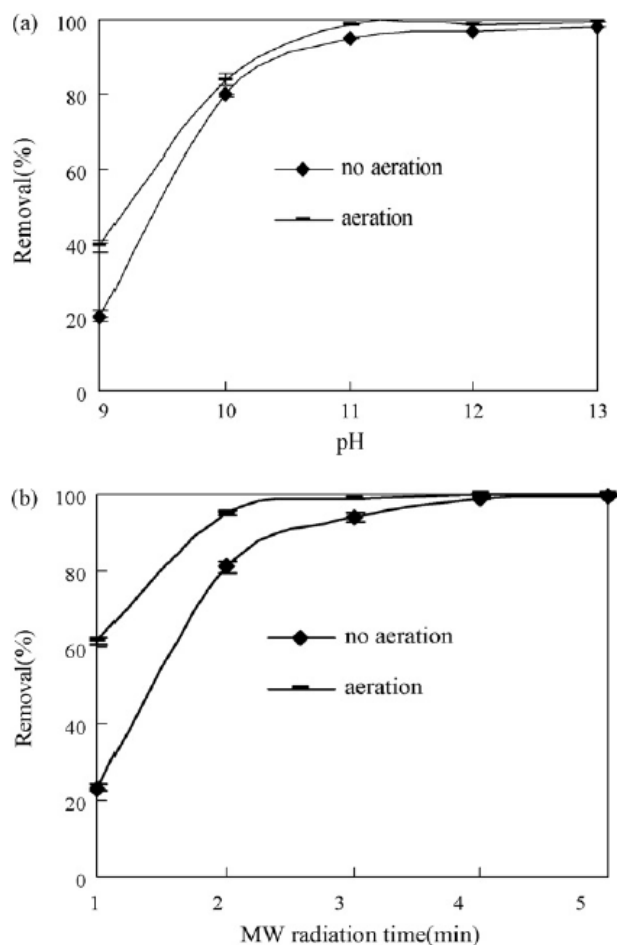


Fig. (2.6) Effect of pH and radiation time on the removal of ammonia nitrogen

(Lin, *et al.*, 2009)

Besides, the researchers applied a similar experiment using an electric cooker, as a conventional heating device, and showed that ammonia nitrogen removal by MW radiation is 225% larger than removal with conventional heating.

Based on the previous mentioned study, Lin, *et al.*, (2009) developed a continuous pilot-scale MW system (4.8kW output power) to remove ammonia nitrogen in real wastewater. They designed a MW reactor with a handling capacity of about 5m³/day. Figure (2.7) shows a schematic diagram of the system. Four factors were evaluated: ambient temperature, wastewater flow rate (2 L/min to 3 L/min), aeration conditions and initial ammonia nitrogen concentration (2400 mg/L to 11,000 mg/L). The low ambient temperature and the high flow rate reduced the effectiveness of the MW reactor. The results demonstrated that the removal of ammonia nitrogen could reach

74-84% with aeration and high ambient temperature. In general, the results of the pilot-scale study were consistent with those of the bench-scale study.

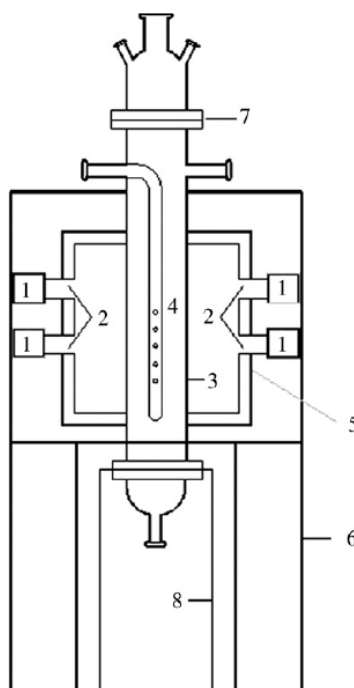


Fig. (2.7) Schematic diagram of the pilot-scale MW system

(1) Magnetron, (2) wave guide, (3) glass tube, (4) bubble tube, (5) cavity chamber, (6) shell, (7) flange, (8) bracket.

(Lin, *et al.*, 2009)

Economically, the previous study compared between MW technique and steam-stripping method used in China, and proved that the running cost of the MW technique (i.e. USD 2.88/ m³ wastewater) was a little lower than the conventional steam-stripping method (i.e. USD 3.51/m³ wastewater).

II. Combination of MW with other reagents:

MW is combined with oxidants to achieve higher reaction temperature within a shorter time frame compared to the traditional thermal method, which assists in the faster degradation of pollutants. (Remya & Lin, 2011)

Focusing on wastewater treatment applications, MW energy was used for several purposes, such as:

a. Boron removal and recovery:-

Tsai & Lo (2011) used a MW hydrothermal method to remove and recover boron from wastewater. They combined MW with calcium hydroxide (Ca(OH)₂) alone, and

Ca(OH)₂ with phosphoric acid (H₃PO₄) addition (P-addition). For the case of Ca(OH)₂ alone and the MW method, experimental results showed that boron recovery efficiency reached 90% within 10 min. For the case of P-addition and the MW method, boron recovery efficiency reached 99% within 10 min. This method is useful for concentrated wastewater, that is usually produced from heavy industries.

b. Nitrite-containing wastewater treatment:-

Li, et al., (2010) used sulfaminic acid to treat nitrite-containing wastewater enhanced with MW radiation. The study was carried on lab-scale and pilot-scale. Based on lab-scale, it was shown that 75-80% nitrite (NO₂⁻) could be removed within time as short as 4 min under 50 W MW irradiation in pH range 5-10. The pilot-scale experiments established that the chemical reduction process was able to achieve nitrite and chemical oxygen demand (COD) removal with efficiency of 80% and 20%, respectively under 3.4 kW microwave power.

c. Degradation of organic pollutants:-

Yang, *et al.* (2009) explored an advanced oxidation process based on sulfate radical (SO₄⁻), to degrade organic pollutants in wastewater. They used a MW-activated persulfate oxidation with or without active carbon. The experiment were conducted to examine whether MW heating is an effective method to activate persulfate and then to decompose biorefractory organic compounds in wastewater by using an azo dye Acid Orange 7 (AO7) (up to 1,000 mg/L) as a model compound. It was found that AO7 was completely decolorized within 5-7 min. By adding 1.0 g/L of active carbon as catalyst, 100% decolorization of AO7 (500 mg/L) was achieved within 3 min.

2.5. Advantages and disadvantages of MW technology

Lin, *et al.*, (2009) listed the basic advantages and disadvantages of the MW technique, based on a comparison with the conventional steam-stripping method, as follows:

2.5.1. Advantages

- 1) The removal efficiency of ammonia nitrogen by MW radiation is higher than that of the air-stripping method. For MW technique, the removal of ammonia nitrogen

reached 95% in lab-scale experiments, and 80% in pilot-scale experiments. Whereas, only 60% removal is reached by the air-stripping method.

- 2) MW technique is a green chemistry. It is strongly capable for water sterilization and pathogens disinfection, and it can effectively inactivate the bacteria and enzyme in wastewater.

2.5.2. Disadvantages

The main disadvantage of the MW is that its radiation consumes electric energy, and converts electric energy to heat. This means high power consumption, because the specific heat capacity of water is very high. (*Heat capacity of a substance is the amount of energy needed to raise the temperature of 1g of a substance by 1°C. For water, it takes 4.19 joules to raise the temperature of 1ml of water by 1°C*).

However, the characteristics of ammonia removal from municipal wastewater by MW energy, the cornerstone for applying MW energy in ammonia removal process, are still unknown. This research is carried out to investigate and identify these characteristics.

CHAPTER 3: Batch reactor experiment

3.1. Introduction

Ammonia nitrogen is a significant pollutant which cause hazardous effects on water resources. In this chapter, the characteristics of ammonia nitrogen removal from municipal wastewater by MW radiation were studied through a batch experiment. The experiment was done in the laboratory of the Department of Environmental Engineering in The Islamic University-Gaza, which spent around two months to be completed. Also, the water analysis laboratory in The Islamic University-Gaza helped with the ammonia nitrogen tests, especially for the real wastewater samples.

3.2. Materials and methods

Figure (3.1) shows a schematic diagram of the experimental apparatus. A domestic microwave oven (700 W, 2450 MHz, Durabrand XB2316, UK) with multiple power settings was used as the source of the MW radiation. A hole was drilled on its top cover, with copper pipe inserted to prevent MW emission (Yang, *et al.*, 2009).

In collaboration with The Palestinian Cellular Communications Company "Jawwal", using electric and magnetic field measurement device (EMR-21C, Safety Test Solutions, Germany), the radiation exposure rate of the MW oven was tested before starting experiments, to make sure that radiation leakage is not harmful. At a distance of 2cm, the whole body exposure limit, averaged over a 6 min period, was about 0.253 mW/cm^2 , which was within the recommended limit of exposure, as mentioned before in the Literature Review.

A 250-ml Erlenmeyer flask containing 100 ml of wastewater was placed in the oven and radiated under different conditions. The flask was connected to a condensing system. The temperature was measured by a thermometer and the final concentration of the ammonia nitrogen was measured using Nessler standard method (APHA, AWWA and WEF, 1989).

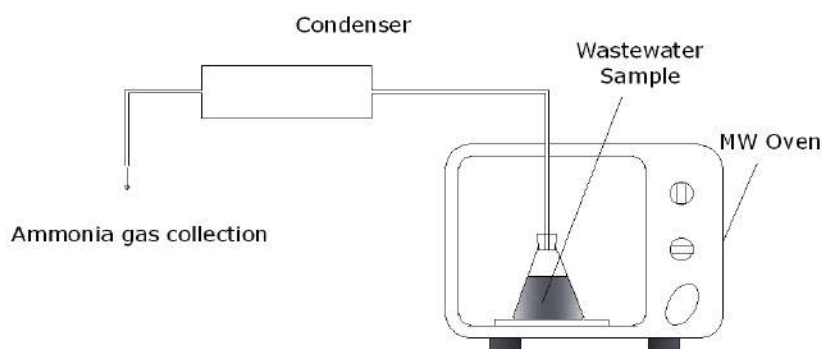


Fig. (3.1) Schematic diagram of the experimental apparatus

Synthetic wastewater solution was prepared with ammonium chloride (99.5%, analytical reagent, HiMedia Laboratories, Mumbai, India) and distilled water. The initial pH of the solution was adjusted using sodium hydroxide solution (NaOH, 2.0 mol/l, 97.5%, Chemie, Mumbai, India).

After heating each sample, the volume of wastewater slightly decreased due to evaporation of water. Thus, after heating each sample, the volume was adjusted with deionized water, using a 100-ml Erlenmeyer flask, to keep the same initial volume of the samples.

As a comparison, the same experiment was applied on real municipal wastewater samples, which were obtained from Gaza Wastewater Treatment Plant. The removal efficiencies of ammonia nitrogen were compared with those resulted from synthetic wastewater.

3.3. Results and discussion

In order to achieve the best removal of ammonia nitrogen from municipal wastewater, the affecting factors, including initial pH, radiation time and initial ammonia concentration were investigated. In this research, no aeration was applied, since it has a minute effect on ammonia nitrogen removal (Lin, *et al.*, 2009).

3.3.1. Effect of initial ammonia concentration

In this experiment, six initial ammonia concentrations were tested: 100, 85, 70, 55, 40, and 25 mg/l. Figure (3.2) indicates that ammonia removal has a clear trend. For 100 mg/l initial ammonia concentration, the difference between final ammonia concentrations for different pH values was very high. At pH 9, the final ammonia

concentration was about 67 mg/l, i.e. 33% removal efficiency. Where at pH 11, the final ammonia concentration was about 12 mg/l, i.e. 88% removal efficiency. However, for 25 mg/l initial ammonia concentration, the final ammonia concentration at pH 11 is lower than at pH 9 by about 17.5, which shows that the rate of removal was low. So, by increasing initial ammonia nitrogen concentration, the rate of ammonia removal increased.

Besides, it can be noticed that at pH 11, initial ammonia concentration had minute effect, as the final ammonia nitrogen concentration was in the range between 2.5 and 12 mg/l, which is relatively small range.

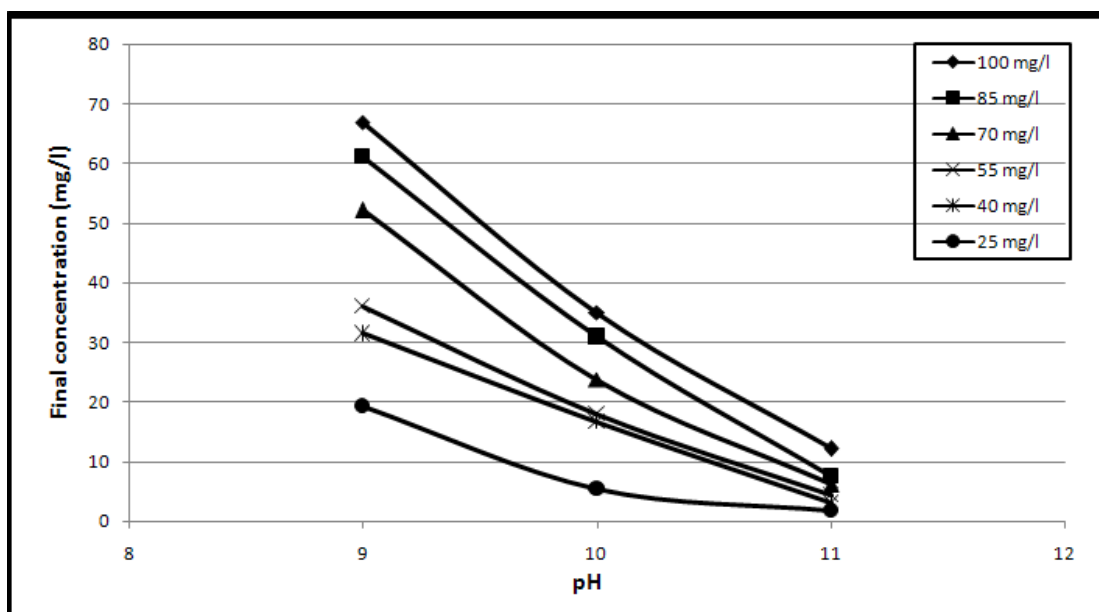


Fig. (3.2) Final ammonia concentration after 4 min radiation time at different pH values

3.3.2. Effect of initial pH

Figures (3.3), (3.4) & (3.5) illustrate the effect of initial concentration of ammonia nitrogen at pH 9, 10 and 11 respectively. Figure (3.3) shows that pH 9 resulted with low ammonia removal efficiency, and all final results were higher than 5 mg/l, the standard ammonia nitrogen concentration in treated wastewater effluent (EQA, 2010). Besides, figure (3.4) shows that pH 10 resulted with low ammonia removal efficiency, but quite better than for pH 9, especially for initial ammonia concentration of 25 mg/l, which achieved a final ammonia concentration of about 4.70 mg/l. However, figure (3.5) illustrates that raising pH value to 11 lead to higher efficiency of ammonia removal.

Consequently, the optimal pH was found to be 11, which resulted in 88-95% ammonia removal after 4 minutes radiation, and initial concentrations of 85, 70, 55, 40 and 25 mg/l resulted with final concentrations of about 7.45, 6.10, 4.35, 2.91 and 1.77 mg/l respectively.

Moreover, the differences between ammonia removal rates are more clear at pH 11 than those at pH 10 and pH 9, which means that ammonia removal by MW at pH 11 is more applicable and effective.

Lin, *et al.* (2009) achieved 98% ammonia removal from industrial wastewater with pH 11 and after 3 min of radiation. Difference in the ammonia removal efficiencies between Lin, *et al.* and this research is referred to the quite difference between the microwaves' powers input (750 W for Lin, *et al.* and 700 W in this research). Also, Lin, *et al.* (2009) tested wastewater with high ammonia nitrogen concentration, which explain why the rate of ammonia nitrogen removal was higher than in this research.

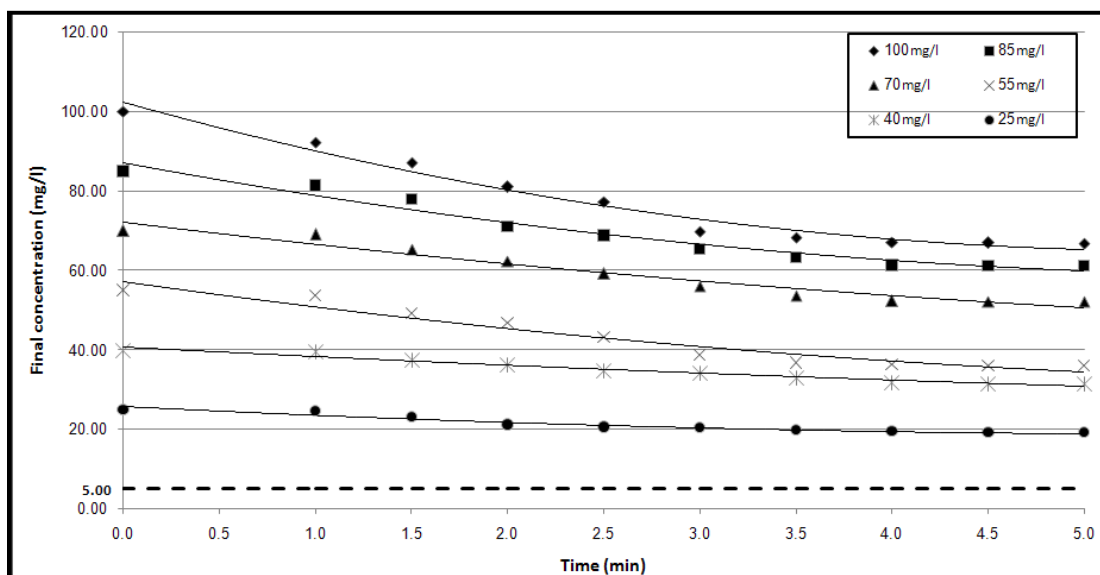


Fig. (3.3) Effect of radiation time and initial ammonia concentration on the removal of ammonia nitrogen at pH 9

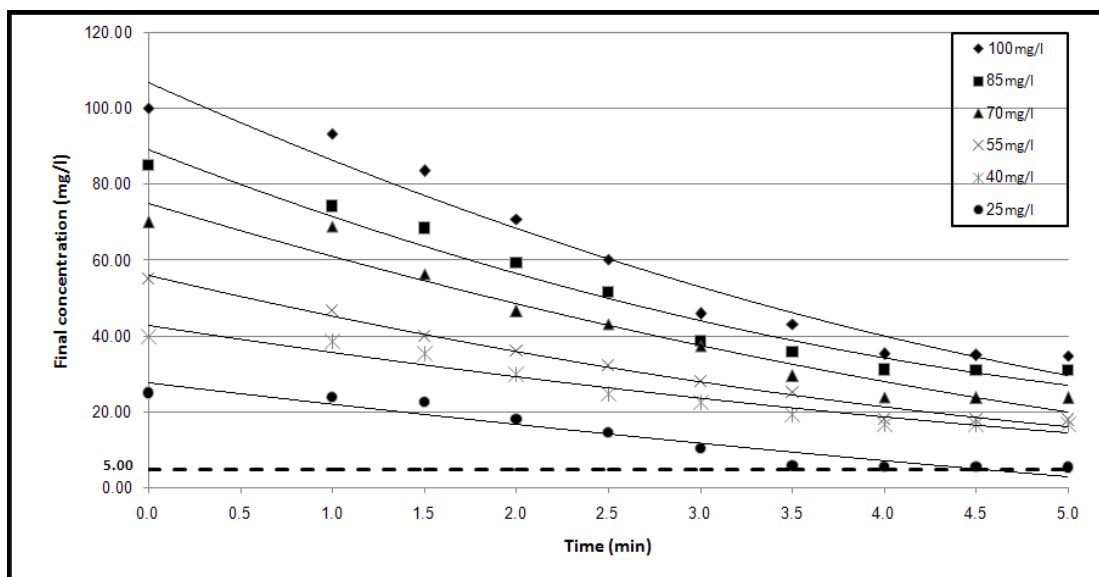


Fig. (3.4) Effect of radiation time and initial ammonia concentration on the removal of ammonia nitrogen at pH 10

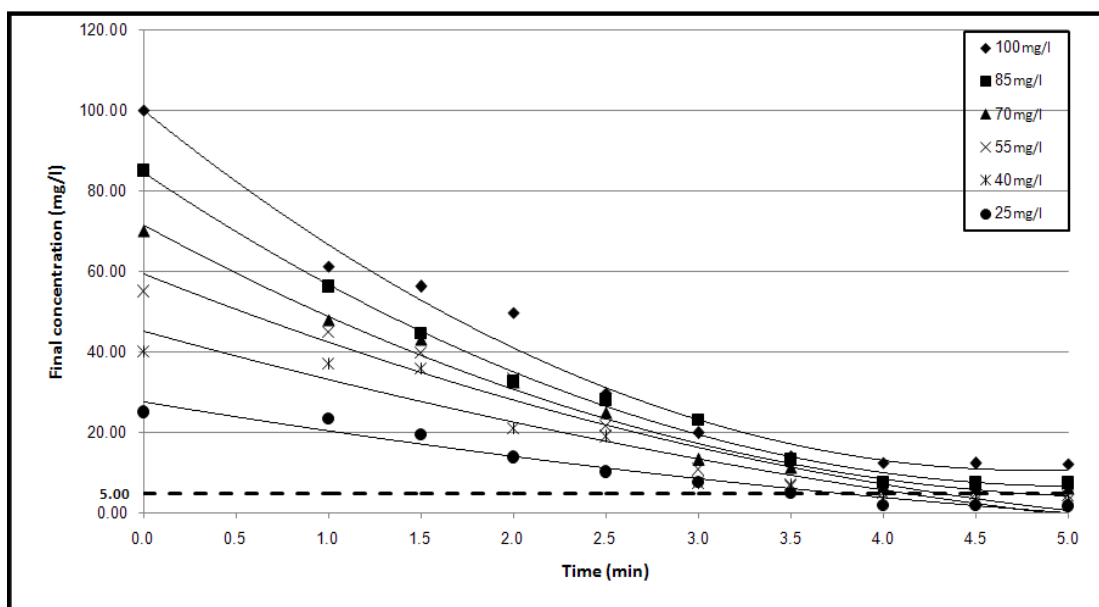
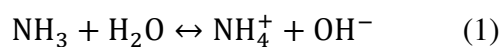


Fig. (3.5) Effect of radiation time and initial ammonia concentration on the removal of ammonia nitrogen at pH 11

There was always a pH dependant equilibrium between soluble ammonium ion (NH_4^+) and dissolved molecular ammonia (NH_3) in wastewater. (Lin, *et al.*, 2009)



$$\alpha_{\text{NH}_3} = \frac{[\text{NH}_3]}{[\text{NH}_3] + [\text{NH}_4^+]} \quad (2)$$

In reaction (2), α_{NH_3} was the distribution coefficient of NH_3 . It was the fraction of NH_3 in total ammonia. $[\text{NH}_3]$ was the concentration of NH_3 , and $[\text{NH}_4^+]$ was the concentration of NH_4^+ in water.

Figure (3.6) shows the distribution coefficients of NH_3 and NH_4^+ at different pH.

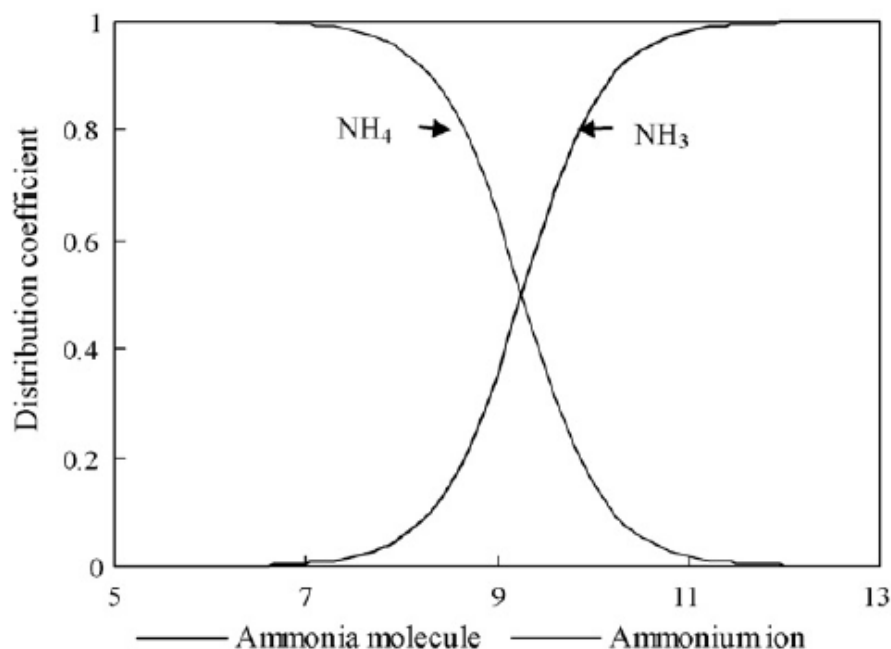


Fig. (3.6) Distribution coefficients of NH_3 and NH_4^+ at different pH levels (20°C)

(Lin, *et al.*, 2009)

Temperature has minute influence on distribution coefficients. In acidic and neutral media, ammonia nitrogen is presented as NH_4^+ . In basic solution, non-volatile NH_4^+ converts to volatile NH_3 . High pH favors ammonia volatilization by driving the equilibrium between NH_3 and NH_4^+ to molecular ammonia. (Lin, *et al.*, 2009)

At the end of radiation, pH of the solution decreased from 11 to about 9.8 due to the escape of NH_3 . When the solution was radiated by MW, the polar molecules in solution rotated fleetly (2450 million times/s), which resulted in rapid heating of the solution (Lin, *et al.*, 2009). Consequently, the molecular movement in wastewater was greatly enhanced, which was highly advantageous for the evaporation of volatile molecular ammonia from liquid to gas. When the temperature of solution rose to boiling point, molecular ammonia could be stripped from the solution by the gas bubbles produced.

3.3.3. Effect of radiation time

Table (3.1) shows the decrease of ammonia concentration with the increase of radiation time. It can be seen that for initial concentration of 55 mg/l and pH 11, 18.33% removal was achieved after 1 min radiation, while after 4 min, about 91% ammonia removal was achieved. More heat could be generated with longer MW radiation time. Thus, the solution temperature became higher, which induced more impetuous and rapid molecular motion. This benefited the escape of ammonia nitrogen from solution. After 4 min of radiation, no significant ammonia removal was achieved, and longer radiation time would result in as excessive cost, 4 min was considered to be the optimal radiation time. Also, figures (3.3), (3.4) & (3.5) show the relationship between final ammonia concentration and MW radiation time, which clears that radiation time had an obvious effect on ammonia removal.

Table (3.1) Decrease of ammonia concentration by increasing radiation time (pH = 11)

Initial concentration (mg/l)	Radiation time (min)									
	0	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0
	Percentage of removal (%)									
25	0	6.40	22.52	44.48	59.32	69.64	80.64	92.24	92.60	92.92
40	0	7.43	10.68	47.55	52.63	81.23	82.63	91.73	92.50	92.73
55	0	18.33	27.82	39.20	60.91	80.33	88.15	90.98	91.82	92.09
70	0	31.47	38.57	53.69	64.56	81.10	83.79	90.54	91.00	91.29
85	0	33.84	47.46	61.47	66.92	72.75	84.44	91.05	91.18	91.24
100	0	38.70	43.76	50.38	70.23	80.15	86.14	87.43	87.70	87.90

More details about the records of the final concentrations can be found within Appendix (A).

3.4. Treatment of real wastewater

In order to confirm the results obtained from the synthetic wastewater experiment, a similar experiment was applied on three real wastewater samples, that were collected from the effluent of Gaza Wastewater Treatment Plant (GWTP). The samples were

collected in Nov. 17th, Dec. 15th and Feb. 18th, 2011, and the initial ammonia nitrogen concentration for them were about 52.30, 67.50 and 86.72 mg/l respectively. It was noticed that ammonia nitrogen concentration in GWTP effluents reaches above 80 mg/L, which is higher than the standard level (EQA, 2008).

The samples' volume was 100 ml, which were filtered carefully by filter paper (120mm radius), to remove any suspended solids that may interfere final testing results. The samples were heated at pH 11, as it was found to be the optimum pH, which resulted from treating synthetic wastewater.

Figure (3.7) shows the results obtained from treating the real wastewater samples. It could be seen that after 4 min of radiation, the final ammonia concentration became relatively constant, and more heating resulted with irrelevant ammonia reduction. Therefore, 4 min radiation was estimated to be the optimum radiation time.

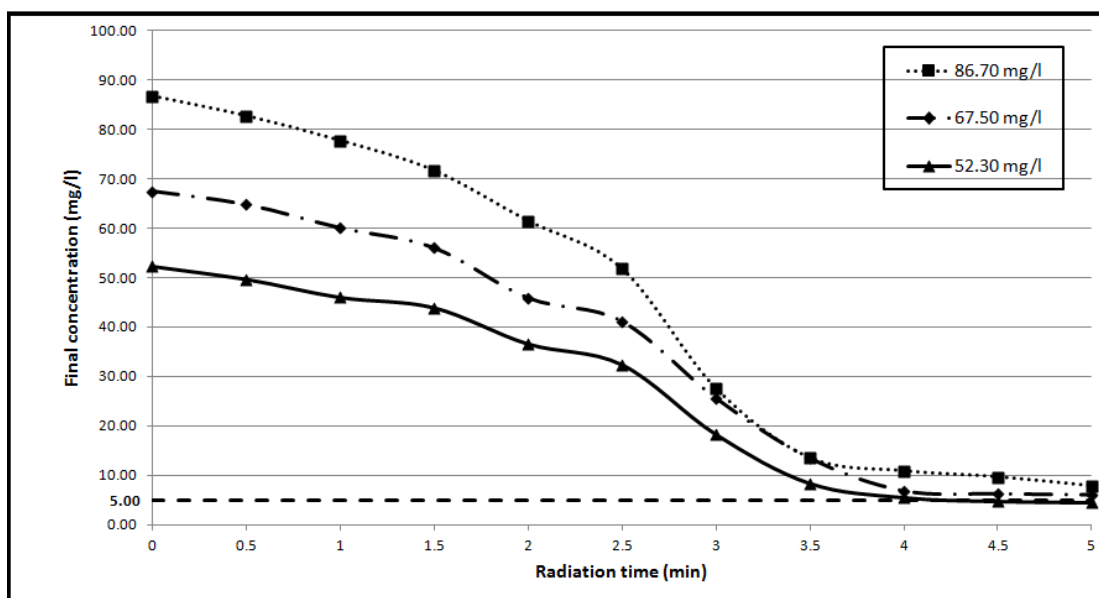


Fig. (3.7) Real municipal wastewater treatment results (pH 11, MW power = 700W)

Table (3.2) presents the final ammonia concentration at 4 min radiation. It could be noticed that the results of real wastewater experiment were consistent with those of synthetic wastewater experiment, which means that high accuracy of results were achieved.

Table (3.2) Final ammonia concentration after 4 min radiation

Initial concentration (mg/l)	Final concentration (after 4 min) (mg/l)	Percentage of removal (%)
52.30	4.45	91.50
67.50	6.08	90.10
87.60	7.80	91.1

CHAPTER 4: Continuous reactor experiment

4.1. Introduction

The main goal of the continuous reactor experiment is to simulate a real MW-based ammonia removal process, in order to compare such a system with conventional ammonia removal methods. The basic idea of this experiment is to build a small-scale lab model of a continuous flow MW reactor, which has similar process of a real one.

4.2. Materials and methods

Figure (4.1) presents a schematic diagram of the experimental apparatus. The source of the MW energy is the same domestic microwave oven that was used in the batch experiment. Another two holes were drilled in its side cover; one of them as inlet and the other as outlet for the flowing wastewater, with copper pipes inserted to prevent MW emission. The wastewater were pumped into a 300 ml glass tube fixed inside the MW oven. In this continuous experiment, the effects of two operating conditions, including wastewater flow rate and initial ammonia concentration were investigated. To provide a constant flow rate of wastewater, a peristaltic pump (SP311, VELP Scientifica) with multiple flow rate setting was connected to a small tank containing wastewater, to pump wastewater into the microwave oven. The temperature was measured by a thermometer and the final concentration of the ammonia nitrogen was measured using Nessler standard method (APHA, AWWA and WEF, 1989).

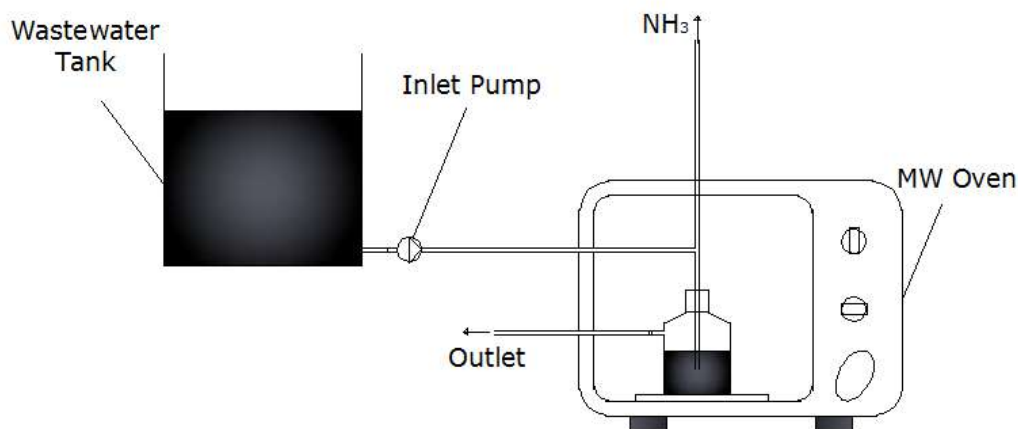


Fig. (4.1) Schematic diagram of the experimental apparatus

According to the results of the batch experiment in Chapter 3, the initial pH of the wastewater was adjusted to 11, since it is the best for using MW energy to remove ammonia nitrogen from wastewater. The pH decreased due to the escape of NH_3 during radiation process.

In order to investigate the effect of detention time on ammonia removal efficiency, five samples were taken within each experiment.

After heating each sample, the volume of wastewater decreased about 10-26% of the initial volume due to evaporation of water. Thus, after collecting each sample, the volume was adjusted with deionized water, to keep the same initial volume of the pumped wastewater; i.e. 300 ml.

4.3. Results and discussion

In this experiment, the effect of initial ammonia concentration and detention time were investigated. In order to match ammonia concentrations in real wastewater streams, four initial concentrations of ammonia nitrogen were tested: 60, 80, 100 & 120 mg/l. The peristaltic pump had flow rate settings ranked from 1 to 10. To investigate the optimum flow rate, four flow rate settings were selected to pump wastewater: 10, 7, 5 & 3 with flow rates of 57, 33.3, 20 & 13.5 ml/min respectively. Lower flow rate settings were too small to be applied in this experiment.

4.3.1. Effect of detention time

Detention time of wastewater into the MW reactor increases with the decrease of wastewater flow rate. Thus, the detention time of wastewater into the MW reactor was calculated according to equation (4.1):

$$\theta \text{ (min.)} = \frac{V(\text{ml.})}{q(\text{ml/min.})} \quad \dots \quad (4.1)$$

where θ is the detention time, V is the reactor volume and q is the flow rate. Table (4.1) shows the detention time for each flow rate case.

Table (4.1) Detention time for each flow rate case

Flow rate (ml/min)	Reactor volume (ml)	Detention time (min)	Evaporated water/detention time period (%)
57	300	5.26	10
33.3	300	9	13.5
20	300	15	17.4
13.5	300	22	26

Figures (4.2), (4.3), (4.4) and (4.5) show the relationship between MW radiation time units and final ammonia concentration for 60, 80, 100 and 120 mg/l initial ammonia concentration, respectively, then attained a plateau after 3 radiation time units, indicating that the system was steady-state after 3 radiation time units, and the final ammonia concentration became approximately constant.

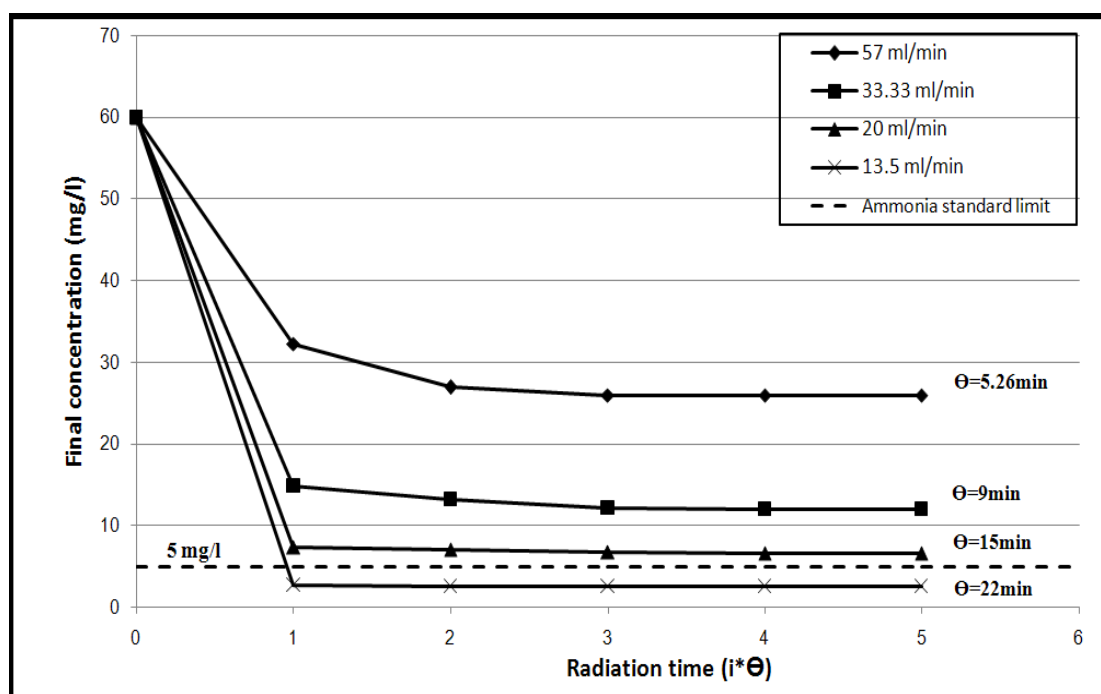


Fig. (4.2) Ammonia nitrogen removal under different flow rate cases, with initial ammonia concentration = 60 mg/l

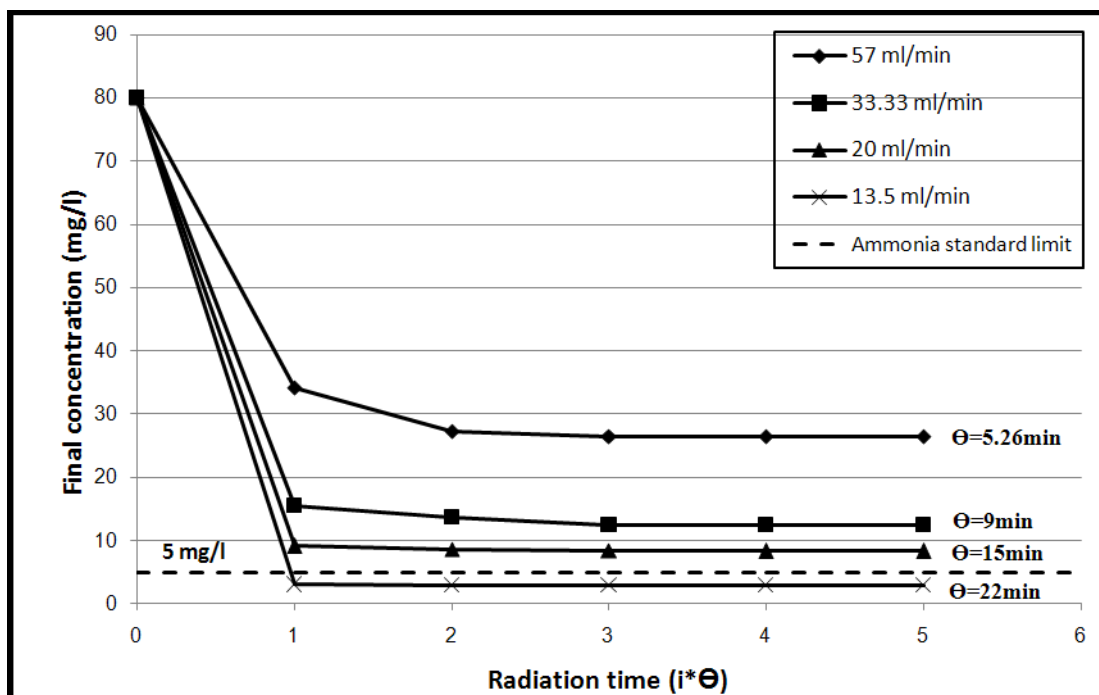


Fig. (4.3) Ammonia nitrogen removal under different flow rate cases, with initial ammonia concentration = 80 mg/l

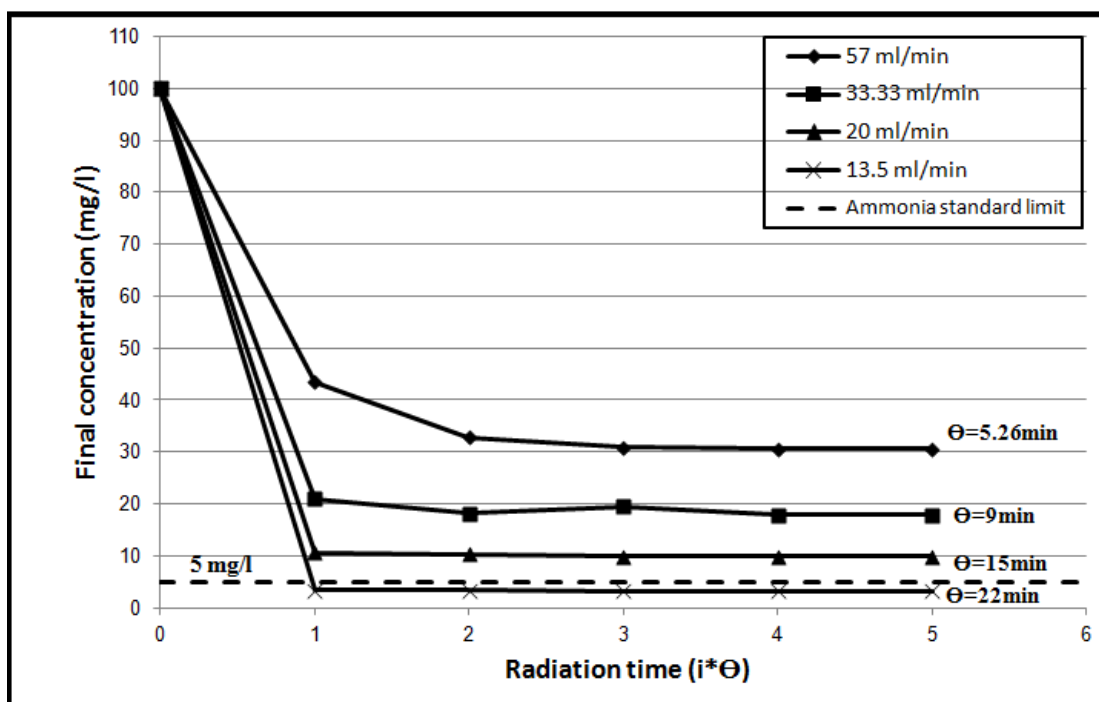


Fig. (4.4) Ammonia nitrogen removal under different flow rate cases, with initial ammonia concentration = 100 mg/l

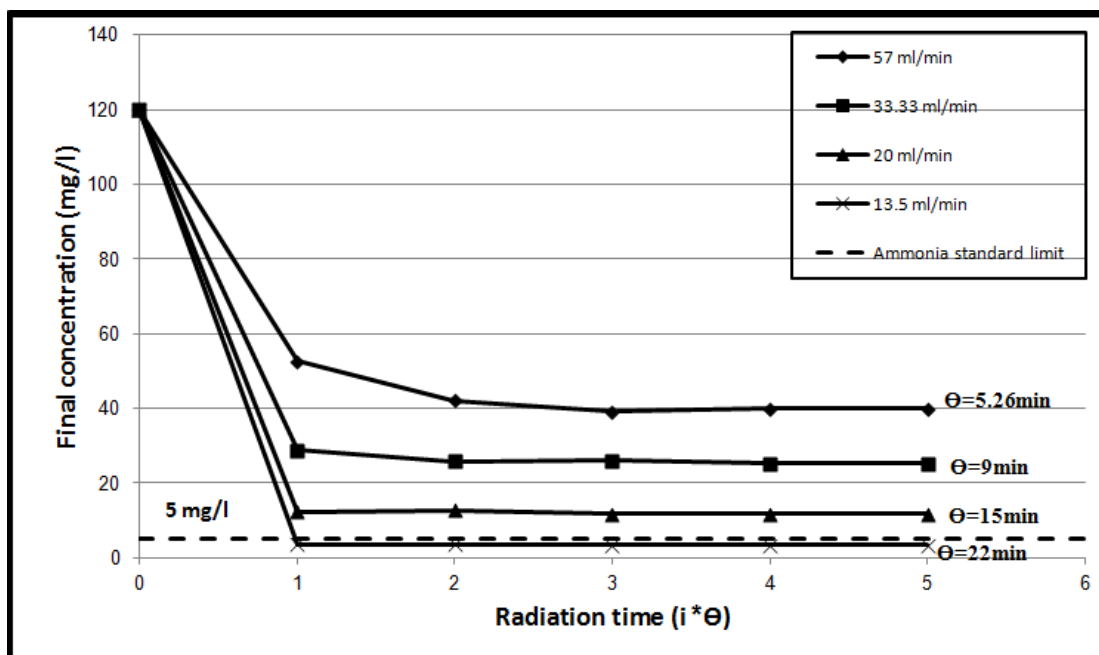


Fig. (4.5) Ammonia nitrogen removal under different flow rate cases, with initial ammonia concentration = 120 mg/l

From the previous figures, it could be noticed that, for all initial concentration cases, a flow rate of 13.5 ml/min resulted with final ammonia concentrations between 3.47 and 2.60 mg/l, which were less than 5 mg/l, and the 20 ml/min flow rate achieved ammonia removal results between 11.75 and 6.70 mg/l, just above the 5 mg/l limit. But, the steady state results for the flow rates 33.33 ml/min and 57 ml/min were not significant, and hence they were not efficient. Table (4.2) demonstrates the final ammonia concentrations after steady-state for each flow rate case with initial ammonia concentration of 120 mg/l.

Table (4.2) Final ammonia concentration after steady-state

Initial ammonia concentration (mg/l)	Flow rate (ml/min)	Final ammonia concentration (mg/l)	Ammonia removal efficiency (%)
120	57	40.42	66.68
	33.3	25.45	78.82
	20	11.75	90.21
	13.5	3.47	94.22
100	57	30.57	69.43
	33.3	17.86	82.14
	20	9.92	90.08
	13.5	3.16	94.73
80	57	26.54	66.83
	33.3	12.40	84.50
	20	8.31	89.62
	13.5	2.91	95.14
60	57	26.04	56.60
	33.3	12.09	79.85
	20	6.70	88.84
	13.5	2.60	95.66

4.3.2. Effect of initial ammonia concentration

In this experiment, ammonia concentrations that are most presented in real municipal wastewater were synthesized (60, 80, 100 & 120 mg/l). For each initial concentration, the four flow rate cases were applied, and the optimum concentration was investigated. Figures (4.6), (4.7), (4.8) and (4.9) illustrate the variation of ammonia removal with the variation of initial ammonia concentration.

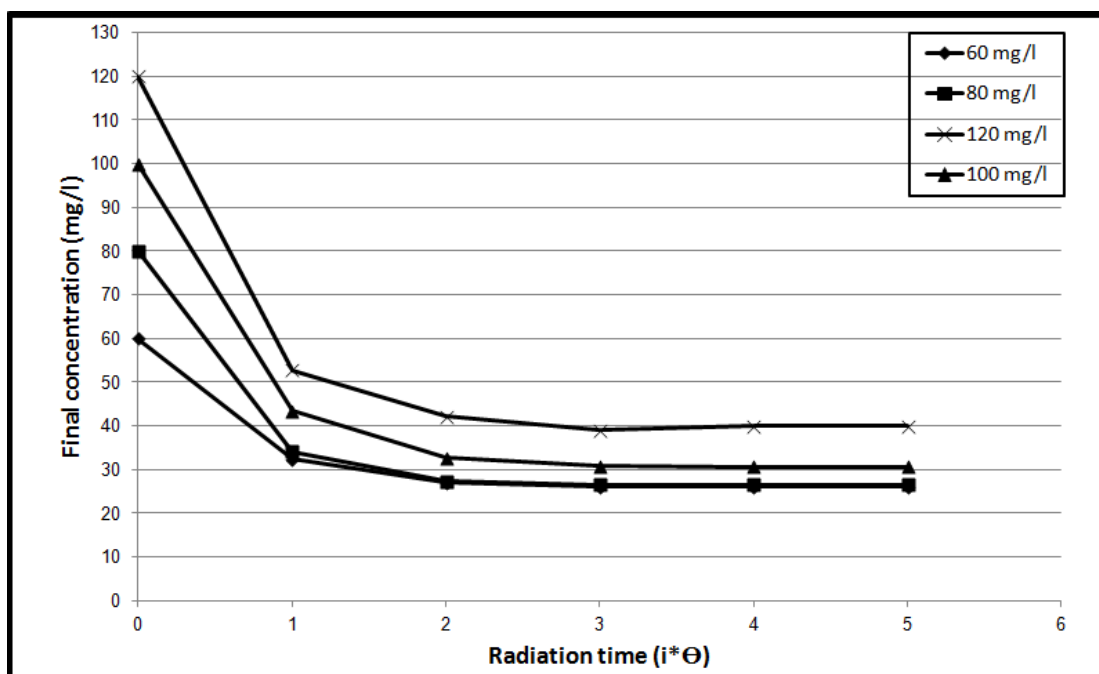


Fig. (4.6) Ammonia nitrogen removal under different initial ammonia concentrations
($\Theta=5.26$ min)

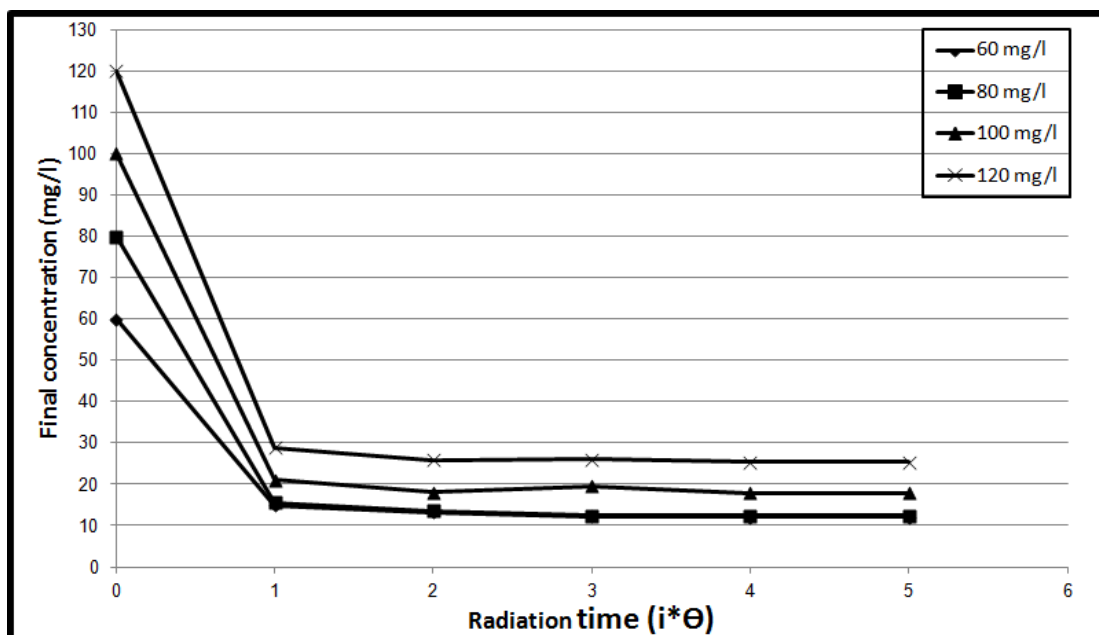


Fig. (4.7) Ammonia nitrogen removal under different initial ammonia concentrations
($\Theta=9$ min)

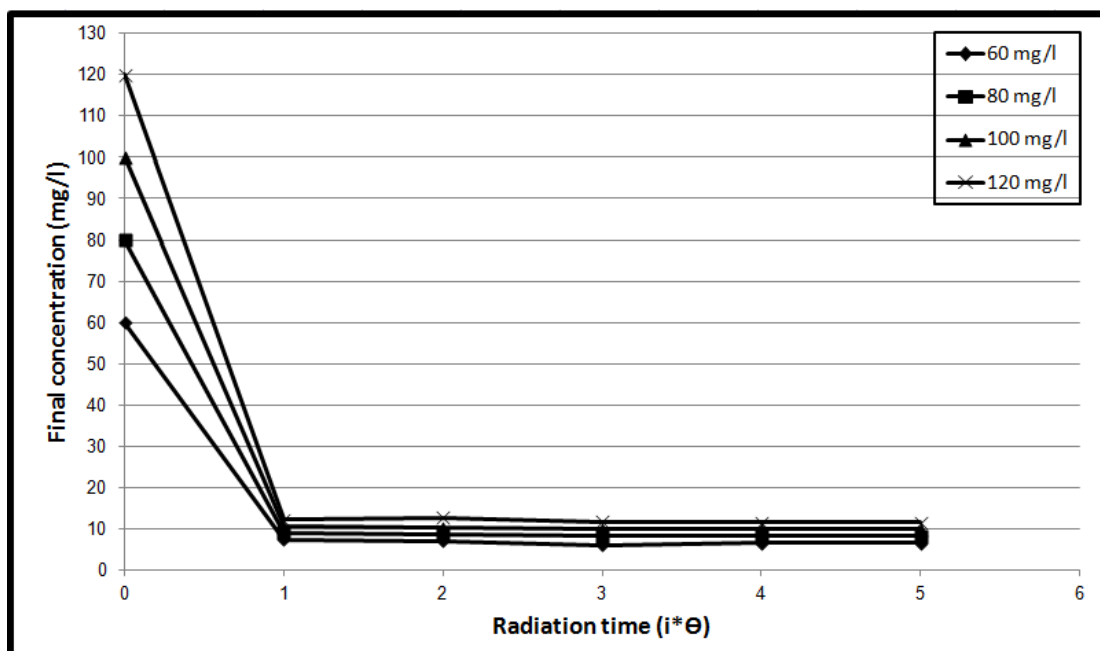


Fig. (4.8) Ammonia nitrogen removal under different initial ammonia concentrations
($\Theta=15$ min)

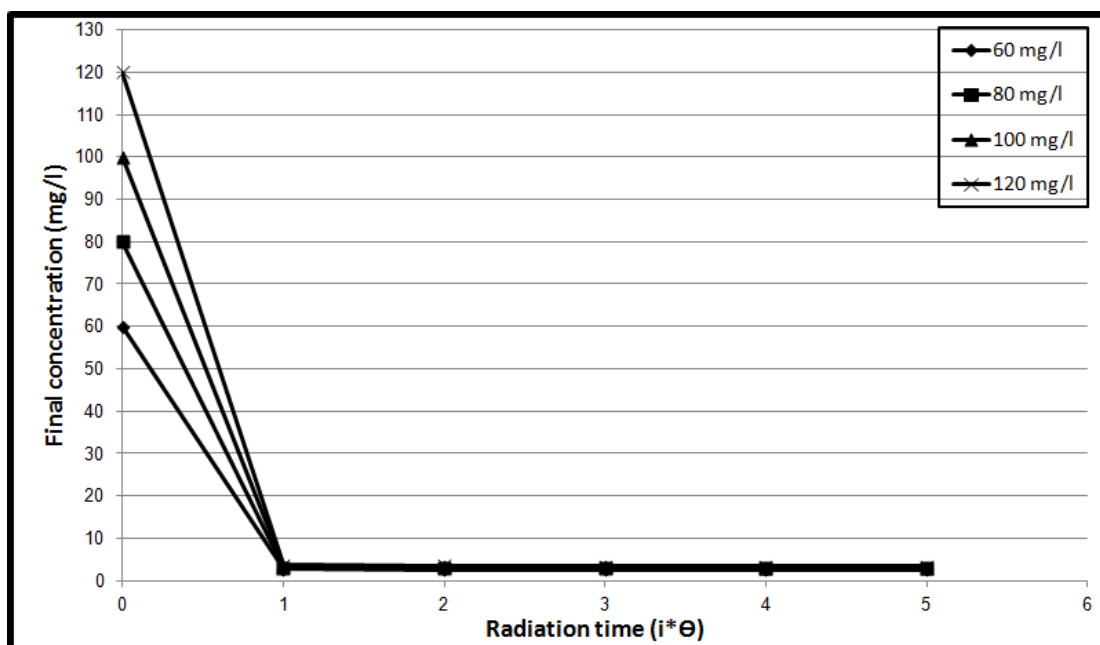


Fig. (4.9) Ammonia nitrogen removal under different initial ammonia concentrations
($\Theta=22$ min)

It could be seen that ammonia removal increased sharply with increasing MW radiation time at the beginning of the process. Figure (4.6) indicates that for flow rate case 1 ($q = 57$ ml/min), the system could reach steady-state after 3 radiation time units (i.e. $3 \times 5.25 = 15.75$ min), and figure (4.7) shows that for flow rate case 2 ($q = 33.33$ ml/min), the system could reach steady-state after about 2 radiation time units (i.e.

2×9 = 18 min). Whereas figures (4.8) & (4.9) illustrate that, for flow rates case 3 ($q = 20$ ml/min) and case 4 ($q = 13.5$ ml/min) respectively, steady-state could be reached after about 1 radiation time unit or less (i.e. 15 and 22 min respectively). The curves in both figure (4.6) and (4.7) are clearly separated after steady state, which indicates clear effect of initial ammonia concentration, since the flow rates were relatively high. On the other hand, the curves in both figure (4.8) and (4.9) are close together after steady state, which indicates that the initial ammonia concentration had not any clear effect on ammonia removal efficiency when the flow rate was lower than 20 ml/min.

Thus, the previous figures perceive that initial concentration had a clear reflect on ammonia removal efficiency at a flow rate higher than 33 ml/min, while the effect was negligible at lower flow rates.

Moreover, for each initial ammonia concentration, it could be noticed that 15 and 22 min detention times (flow rates = 20 and 13.5 ml/min respectively) resulted in the highest ammonia removal (90.2% and 94.2% respectively), since the wastewater was exposed to longer radiation time.

Finally, it could be noticed that treating initial ammonia concentration of 60 mg/l resulted in the best ammonia removal efficiency in all flow rate cases, which could be considered as the optimal initial concentration. This means that ammonia nitrogen requires quite long radiation time periods to be removed significantly.

4.3.3. Effect of initial temperature

In order to study the effect of initial temperature of wastewater, heat exchanger was used. Heat exchanger is a device built for efficient heat transfer from one fluid to another, and is widely used in the engineering processes. Many types of heat exchangers are used for various applications, as refrigeration, air conditioning, power plants, natural gas processing and sewage treatment.

In the present research, the aim of using heat exchanger was to recover effluent heat energy, so as to increase influent temperature. The applied heat exchanger was hand-made, consisted of an insulated pipe (inner diameter = 0.75in \approx 1.90cm) which the hot effluent stream passes through it, making a hot water jacket around the cold

influent stream pipe (inner diameter = 0.2in \approx 0.5cm) that is inserted into the insulated pipe, as shown in figure (4.10).

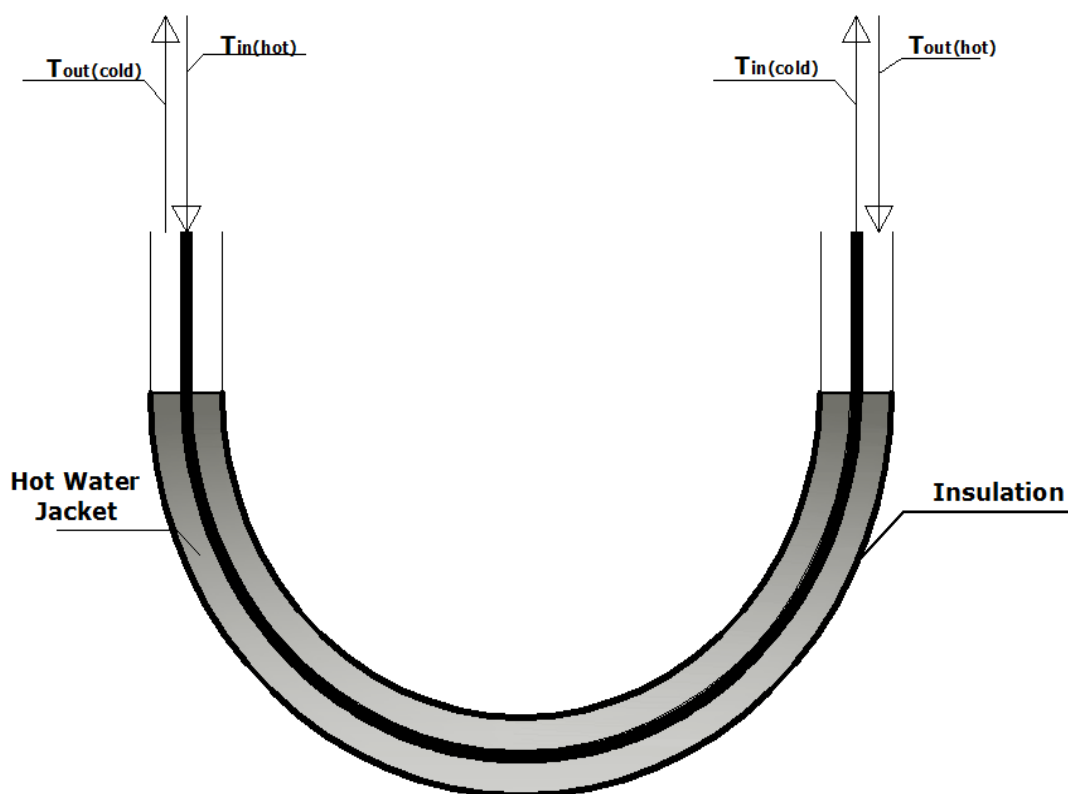


Fig. (4.10) Hand-made heat exchanger

The hot effluent ($T_{out(hot)}$) entered the heat exchanger with a temperature of 97 °C, not 100 °C, due to the lost heat in the pipe connecting between the MW oven outlet and the heat exchanger.

The case of 20 ml/min flow rate ($\Theta = 15$ min) was selected to apply the heat exchanger, in order to investigate the influence on heat recovery and ammonia removal efficiency. Continuously, after collecting 5 samples, the flow rate was increased to 33.33 ml/min ($\Theta = 9$ min), and another 5 samples were taken. Then, the final ammonia nitrogen concentration was measured for each sample. In addition, the temperatures of the heated influent ($T_{in(hot)}$) and the cooled effluent ($T_{out(cold)}$) were measured for both flow rate cases, using a multi-meter portable device (BoeKel, Conductivity/TDS/°C Meter, Model 530, Germany), and the records are presented in table (4.3).

Table (4.3) Temperature recovery recordings

Sample No.	Θ (min)	Average temperature after heat exchange ($^{\circ}\text{C}$)	
		$T_{\text{in(hot)}}$	$T_{\text{out(cold)}}$
1	15	43	40
2		48	43
3		57.5	44
4		58	45
5		59	44
6	9	42	47
7		46	51
8		53	52
9		54	51
10		54	51

It was observed that influent temperature increased from 22.5°C to a range between $54\text{--}59^{\circ}\text{C}$. The maximum heat recovery efficiency of the applied heat exchanger can be calculated according to equation (4.2):

$$\text{Efficiency} = \frac{T_{\text{in(hot)}} - T_{\text{in(cold)}}}{T_{\text{out(hot)}} - T_{\text{in(cold)}}} \times 100\% \quad \dots \quad (4.2)$$

Table (4.4) shows the efficiency of the heat exchanger for each flow rate case.

Table (4.4) Max. efficiency of the heat exchanger

Flow rate (ml/min)	Max. efficiency (%)
20	49.00
33.33	42.25

It could be seen that the overall efficiency of the applied heat exchanger was low, because heat energy was lost in the heat exchanger pipe, since the pipe was not insulated well, as it was hand-made.

Final ammonia nitrogen concentration was measured for both flow rate cases, and the results are illustrated in figure (4.11).

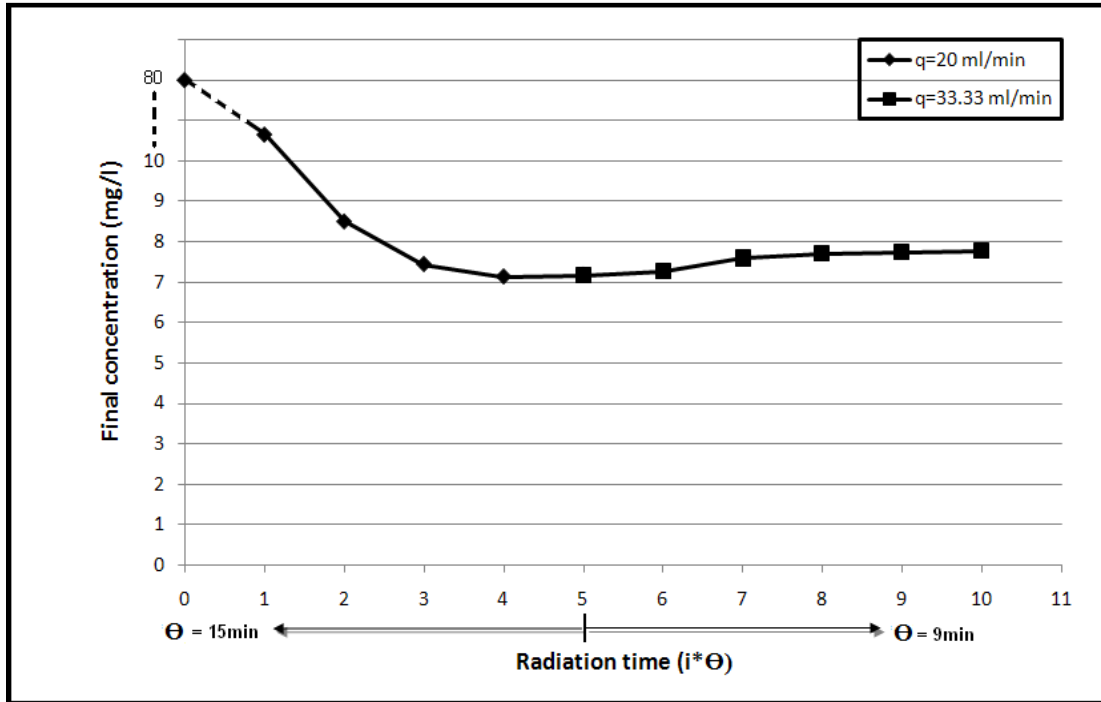


Fig. (4.11) Final ammonia nitrogen concentration after using heat exchanger (initial concentration = 80 mg/l)

After collecting the first 5 samples, with $\Theta = 15$ min, steady state was reached, and the final ammonia concentration was about 7.17 mg/l, i.e. removal efficiency of 91.04%. Then, after collecting 3 samples, with $\Theta = 9$ min, the steady state was achieved for the second flow rate case with a final ammonia concentration of about 7.77 mg/l.

It could be seen that ammonia removal was approximately the same for both examined detention times, which indicates that heat recovery had a significant effect on ammonia removal, and increasing initial influent temperature could notably enhance the overall system's efficiency.

Without using heat exchanger, the maximum removal efficiency achieved for the flow rate of 33.33 ml/min was about 84.50%, with initial concentration of 80 mg/l. However, using heat exchanger improved the removal efficiency to 90.28%.

Besides, similar ammonia removal efficiencies were achieved, though 33.33 ml/min is greater than 20 ml/min by 66.7%, which denotes that energy consumption for the flow rate of 20 ml/min could be considerably decreased, since larger volume of wastewater might be treated, with the same efficiency, even though the flow rate was increased by about two thirds.

CHAPTER 5: Economic aspects study

5.1. MW energy calculation

The basic point in the economic analysis of the MW system is to calculate energy consumption. Equation (5.1) was used to calculate the power of the MW reactor based on energy balance and temperature difference between inlet and outlet of the MW heating cavity.

$$Q = Cm\Delta T \quad \dots \quad (5.1)$$

where Q is the absorbed energy in W, C is the specific heat capacity of wastewater in J/(kg.°C) (assumed 4.2×10^3 J/(kg.°C), the same as H₂O), m is the mass flow rate in kg/s, and ΔT is the change of temperature in °C. In the present experiment, the volume of reactor was 300 ml, the inlet temperature was about 22.5 °C, and at the outlet the wastewater was boiling. For each flow rate, equation (5.1) was applied, and Q was calculated. Table (5.1) shows the value of the absorbed energy for each flow rate case.

Table (5.1) Effective absorbed energy

Flow rate (ml/min)	Absorbed energy (W/300 ml)
57	309.23
33.33	180.82
20	108.50
13.5	73.24

It should be mentioned that reactor volume was, for experiment requirement, only 300 ml. However, the energy conversion efficiency from electric energy to MW energy is 50-75% (Lin, *et al.*, 2009), with the conversion efficiency of the present MW reactor being 75%. Since the input electric power value of the MW reactor was 700 W, the actual output MW power value should be approximately 525 W. Applying equation (5.1) by substituting the value of absorbed energy (Q) by 525W, it should give different value of mass flow rate (m) as follows:

$$m = \frac{Q}{C\Delta T} = \frac{525}{4.2 \times 10^3 \times (100 - 22.5)} = 1.613 \times 10^{-3} \text{kg/s} = 96.77 \text{ml/min}$$

Considering the same detention time (22min), the reactor volume should be:

$$V = \theta \times q = 22 \times 96.77 = 2129.03 \text{ml} \approx 2.13 \text{L}$$

$$\text{The daily capacity of the reactor} = 96.77 \times \frac{60 \times 24}{10^6} = 0.14 \text{m}^3/\text{day}$$

$$\text{Electricity consumption} = \frac{700 \times 24}{0.14 \times 1000} = 120 \text{kW/m}^3$$

This high power consumption can be reduced by using "heat exchanger".

5.1.1. Reduction of power consumption using heat exchanger

A heat exchanger could be used to transfer heat from the boiling effluent (100°C) to the cold influent (22.5°C). As mentioned in Chapter 4, the max efficiency of the applied heat exchanger was about 49%, and this is a low efficiency. Assuming a heat exchanger with heat transfer coefficient of 90%, and the mass flow of both influent and effluent are equaled, electricity consumption could be reduced as follows:

$$t_{\text{in}} = \frac{100 + 22.5}{2} \times 0.90 = 55.125^\circ\text{C} \approx 55^\circ\text{C}$$

$$t_{\text{out}} = \frac{100 + 22.5}{2 \times 0.9} = 69.44 \approx 70^\circ\text{C}$$

$$m = \frac{Q}{C\Delta T} = \frac{525}{4.2 \times 10^3 \times (100 - 55)} = 2.77 \times 10^{-3} \text{kg/s} \approx 167 \text{ml/min}$$

$$\text{The daily capacity of the reactor} = 167 \times \frac{60 \times 24}{10^6} = 0.241 \text{m}^3/\text{day}$$

$$V = \theta \times q = 22 \times 167 = 3674 \text{ml} \approx 3.7 \text{L}$$

$$\text{Electricity consumption} = \frac{700 \times 24}{0.241 \times 1000} \approx 70 \text{kW/m}^3$$

It is still high consumption rate, and much higher than power consumption in other wastewater treatment systems. But, energy consumption is not the only item in running costs, and not the only thing that comparison between treatment systems is based on. In some special cases, a more expensive system can be selected for special

purposes, like high toxicity of wastewater and very cold climates, as will be discussed later.

5.2. Chemicals

In the present research, the only used chemical substance was sodium hydroxide (NaOH), to raise pH of wastewater, which plays a significant role in ammonia nitrogen removal process. Specifically, NaOH solution (2 mol/L) was used for the experiment purpose only as a base solution. About 0.50 ml of this solution could raise pH to 11 for a 100 ml wastewater sample. It should be mentioned that no side effects of raising wastewater pH using NaOH were investigated, since it needs further wastewater tests, which are out of the research's scope.

Other types of bases can be used in wastewater treatment processes, such as Lime $[\text{Ca}(\text{OH})_2]$ and Soda Ash $[\text{Na}_2\text{CO}_3]$ in hardness removal, but none of them was used in the present research.

5.3. Economic benefits

From the previous experimental results, the MW energy might be an effective alternative to traditional technologies for eliminating ammonia nitrogen from wastewater. However, power consumption in a MW-based ammonia removal system is very high. When MW energy is used to treat wastewater, a large amount of energy is needed because the specific heat capacity of water is very high.

Although MW-based wastewater treatment system consumes much energy in the treatment process, it has unique characteristics that are lacking in other systems, such as:

- 1) MW system is not affected by weather conditions. It can operate effectively in both cold and hot weathers. Nitrification process in biological treatment systems is sensitive to weather conditions. Concerns about cold temperature nitrification usually arise when water temperature in the biological treatment system drop to 5°C or below. At this temperature, the nitrifying bacteria responsible for oxidizing ammonia tend to go dormant. This case is always present in cold countries, like Sweden, Norway, Russia and UK. MW system can overcome this obstacle, due to the rapid heating caused by radiation energy.

- 2) Ammonia gas collection and recovery can be easily achieved using MW-based system. As mentioned in the literature review (Chapter 2), ammonia can be utilized in many applications. On the other hand, other ammonia removal systems can't recover ammonia, because it is converted to gaseous nitrogen that emit to the atmosphere.
- 3) During heating wastewater by MW energy, significant amounts of water is evaporated. During the present experiment, the amounts of evaporated water were recorded. Table (5.2) shows the amount of water evaporated in each detention time unit, for the four flow rate cases.

Table (5.2) Evaporated water amounts

Detention time (min)	Evaporated water (ml/300 ml)	Evaporated water (L/m³)
5.25	30	100
9	40.5	135
15	52.2	174
22	78	260

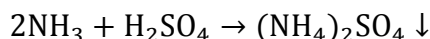
It can be seen that huge amounts of evaporated water is produced, which is a noticeable thing that should be taken into account. After condensation, evaporated water can be utilized for several purposes, like using it to produce distilled or deionized water that is widely used in many chemical and industrial applications. Also, wastewater effluent can be diluted with water condensate, to decrease its pollution.

- 4) While wastewater was heated, thus evaporated, the vapor generates a hot steam, that could be pressurized, and utilized in electricity generation, soil sterilization or piped into buildings through a district heating system.
- 5) Biological treatment systems require large infrastructures (like aerated lagoons, oxidation ditches and activated sludge systems), which cost much money for implementation. Also, air stripping system's infrastructure is massive, because it needs large towers.

The Infrastructure of the MW system is not so large. Lin, *et al.*, (2009) developed a pilot-scale MW system, that could treat 5m³ of industrial wastewater per day.

The main components of the experiment's infrastructure were a cylindrical shaped glass tube (inner diameter = 22 cm, length = 205 cm), a lye mixing chamber and the magnetrons. According to the authors, these components didn't cost much, except the glass tube, since it was made of boron glass, which could stand high temperatures. The glass tube needs to be replaced regularly, as its temperature will be very high during the treatment process, which leads to a short life of the glass tube. In fact, there isn't any MW system applied on a full-scale, so a comparison couldn't be obviously conducted.

- 6) Heating wastewater by MW radiation leads to boiling. When wastewater temperature reaches 100°C, it is sterilized due to boiling. Therefore, all harmful bacteria, organisms and maybe viruses will be killed, which will save capital and running costs required for biological wastewater treatment processes.
- 7) The contaminant vapor generated from heating wastewater could be passed through vessels containing sulfuric acid (H₂SO₄), in order to precipitate ammonium sulfate, to produce fertilizers. The following equation illustrates this reaction:



- 8) Lime (Ca(OH)₂) could be used as alkali instead of NaOH. Furthermore lime is cheaper, it can also be reclaimed and reused through a process called "recalcination". For large wastewater treatment plants, a lime recovery system might be installed in which the calcium carbonate in the sludge is reconverted into lime.

More than 60% of original lime could be reclaimed, that would save much of running costs carried out in chemical additives (Parker, *et al.*, 1975).

From an engineering point of view, renewable energy technologies should be considered as an energy resource for MW systems, in order to save electrical energy consumption. Furthermore, high-efficiency heat recovery system should be developed, so as to increase influent temperature, that will lower down the required energy for ammonia removal.

Actually, this experiment was carried out through a bench-scale. The results are not completely sufficient to estimate real practical capital, operation and maintenance costs.

CHAPTER 6: Conclusion and recommendations

6.1. Conclusion

The main objective of this study was to investigate the characteristics of ammonia nitrogen removal from municipal wastewater using MW radiation. The study was based on two experiments: batch reactor and continuous reactor. The batch reactor experiment explored the effect of initial ammonia concentration, initial pH and radiation time on ammonia nitrogen removal. In this experiment, the removal of ammonia nitrogen reached about 95%, which was achieved in around 4min. pH and MW radiation time showed heavy influence on ammonia removal, while initial ammonia concentration presented minute effect. Higher pH and longer MW radiation time resulted in larger removal efficiencies.

The continuous reactor experiment studied the effect of detention time and initial ammonia concentration. According to experimental results, using MW energy in ammonia nitrogen removal from municipal wastewater is possible. About 94% ammonia nitrogen removal was achieved at detention time of 22min, which was the optimum operation condition. Even though power consumption of the system was relatively high, the system has many economic benefits, like using ammonia in several industries and utilizing the hot steam resulted from wastewater heating in many beneficial applications.

6.2. Recommendations

- 1- The MW technique could be economically feasible if it is used at cold or snowy countries, where biological methods can't achieve full nitrification, that threatens the environment.
- 2- Because of the high power consumption, the optimal application field of the MW technique is the toxic industrial wastewater which contains high concentrations of ammonia nitrogen and is hard to be treated by conventional methods, such as wastewater from tannery, textile and landfill leachates.
- 3- When using MW energy system, ammonia should be gathered, either as a gas or as a precipitate, to be exploited for industrial applications and warming buildings.

- 4- Future studies should focus on optimizing the system for maximum power utilization and energy efficiency by recovering part of the process heat through heat exchangers. Using effective heat exchanger system would reduce the energy costs
- 5- A pilot scale study should be carried out in a further study to estimate real capital and operational costs of using MW energy in municipal wastewater treatment.
- 6- Environmental impacts of using MW radiation in wastewater treatment should be assessed in future research studies.

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Appendices

Appendix (A)

• Batch experiment – Synthetic wastewater:-

Table (A.1) pH = 11, Initial concentration = 100 mg/l

Radiation Time (min)	T_{in} (°C)	T_{out} (°C)	C_{in} (mg/l)	C_{out} (mg/l)	Removal (%)
0.0	28	28	100.00	100.00	0.00
1.0	28	65	100.00	61.30	38.70
1.5	28	84	100.00	56.24	43.76
2.0	28	100	100.00	49.62	50.38
2.5	28	100	100.00	29.77	70.23
3.0	28	100	100.00	19.85	80.15
3.5	28	100	100.00	13.86	86.14
4.0	28	100	100.00	12.57	87.43
4.5	28	100	100.00	12.30	87.70
5.0	28	100	100.00	12.10	87.90

Table (A.2) pH = 11, Initial concentration = 85 mg/l

Radiation Time (min)	T_{in} (°C)	T_{out} (°C)	C_{in} (mg/l)	C_{out} (mg/l)	Removal (%)
0.0	25	25	85.00	85.00	0.00
1.0	25	63	85.00	56.24	33.84
1.5	25	85	85.00	44.66	47.46
2.0	25	100	85.00	32.75	61.47
2.5	25	100	85.00	28.12	66.92
3.0	25	100	85.00	23.16	72.76
3.5	25	100	85.00	13.23	84.43
4.0	25	100	85.00	7.61	91.05
4.5	25	100	85.00	7.50	91.18
5.0	25	100	85.00	7.45	91.24

Appendices

Table (A.3) pH = 11, Initial concentration = 70 mg/l

Radiation Time (min)	T_{in} (°C)	T_{out} (°C)	C_{in} (mg/l)	C_{out} (mg/l)	Removal (%)
0.0	25	25	70.00	70.00	0.00
1.0	25	63	70.00	47.97	31.48
1.5	25	85	70.00	43.00	38.57
2.0	25	100	70.00	32.42	53.69
2.5	25	100	70.00	24.81	64.56
3.0	25	100	70.00	13.23	81.10
3.5	25	100	70.00	11.35	83.79
4.0	25	100	70.00	6.62	90.55
4.5	25	100	70.00	6.30	91.00
5.0	25	100	70.00	6.10	91.29

Table (A.4) pH = 11, Initial concentration = 55 mg/l

Radiation Time (min)	T_{in} (°C)	T_{out} (°C)	C_{in} (mg/l)	C_{out} (mg/l)	Removal (%)
0.0	22	22	55.00	55.00	0.00
1.0	22	62	55.00	44.92	18.32
1.5	22	83	55.00	39.70	27.83
2.0	22	100	55.00	33.44	39.19
2.5	22	100	55.00	21.50	60.91
3.0	22	100	55.00	10.82	80.33
3.5	22	100	55.00	6.52	88.15
4.0	22	100	55.00	4.96	90.98
4.5	22	100	55.00	4.50	91.82
5.0	22	100	55.00	4.35	92.09

Appendices

Table (A.5) pH = 11, Initial concentration = 40 mg/l

Radiation Time (min)	T_{in} (°C)	T_{out} (°C)	C_{in} (mg/l)	C_{out} (mg/l)	Removal (%)
0.0	28	28	40.00	40.00	0.00
1.0	28	65	40.00	37.03	7.44
1.5	28	84	40.00	35.73	10.67
2.0	28	100	40.00	20.98	47.55
2.5	28	100	40.00	18.95	52.63
3.0	28	100	40.00	7.51	81.23
3.5	28	100	40.00	6.95	82.63
4.0	28	100	40.00	3.31	91.73
4.5	28	100	40.00	3.00	92.50
5.0	28	100	40.00	2.91	92.73

Table (A.6) pH = 11, Initial concentration = 25 mg/l

Radiation Time (min)	T_{in} (°C)	T_{out} (°C)	C_{in} (mg/l)	C_{out} (mg/l)	Removal (%)
0.0	25	25	25.00	25.00	0.00
1.0	25	65	25.00	23.40	6.39
1.5	25	84	25.00	19.37	22.53
2.0	25	100	25.00	13.88	44.48
2.5	25	100	25.00	10.17	59.33
3.0	25	100	25.00	7.59	69.66
3.5	25	100	25.00	4.84	80.63
4.0	25	100	25.00	1.94	92.25
4.5	25	100	25.00	1.85	92.60
5.0	25	100	25.00	1.77	92.92

• Batch experiment – Real wastewater:-

Table (A.7) pH = 11, Initial concentration = 86.70 mg/l

Radiation Time (min)	T_{in} (°C)	T_{out} (°C)	C_{in} (mg/l)	C_{out} (mg/l)	Removal (%)
0.0	10	10	100.00	82.71	4.60
1.0	10	72	100.00	77.71	10.37
1.5	10	95	100.00	71.63	17.38
2.0	10	100	100.00	61.43	29.15
2.5	10	100	100.00	51.76	40.30
3.0	10	100	100.00	27.57	68.20
3.5	10	100	100.00	13.49	84.44
4.0	10	100	100.00	10.84	87.50
4.5	10	100	100.00	9.63	88.89
5.0	10	100	100.00	7.80	91.00

Table (A.8) pH = 11, Initial concentration = 67.50 mg/l

Radiation Time (min)	T_{in} (°C)	T_{out} (°C)	C_{in} (mg/l)	C_{out} (mg/l)	Removal (%)
0.0	10	10	100.00	64.80	4.00
1.0	10	72	100.00	60.08	11.00
1.5	10	95	100.00	56.03	17.00
2.0	10	100	100.00	45.90	32.00
2.5	10	100	100.00	41.18	39.00
3.0	10	100	100.00	25.65	62.00
3.5	10	100	100.00	13.50	80.00
4.0	10	100	100.00	6.89	89.80
4.5	10	100	100.00	6.28	90.70
5.0	10	100	100.00	6.08	91.00

Appendices

Table (A.9) pH = 11, Initial concentration = 52.30 mg/l

Radiation Time (min)	T_{in} (°C)	T_{out} (°C)	C_{in} (mg/l)	C_{out} (mg/l)	Removal (%)
0.0	10	10	100.00	49.69	5.00
1.0	10	72	100.00	46.02	12.00
1.5	10	95	100.00	43.93	16.00
2.0	10	100	100.00	36.61	30.00
2.5	10	100	100.00	32.43	38.00
3.0	10	100	100.00	18.31	65.00
3.5	10	100	100.00	8.37	84.00
4.0	10	100	100.00	5.49	89.50
4.5	10	100	100.00	4.71	91.00
5.0	10	100	100.00	4.45	91.50

Appendix (B)

- Continuous experiment:-

Table (A.10) Initial concentration = 60 mg/l

Q (ml/min)	Radiation time unit (<i>i</i>)	Average Final Conc. (mg/l)	Removal (%)
57	0	60	0.00
	1	32.30	46.16
	2	26.97	55.05
	3	26.04	56.60
	4	26.04	56.60
	5	26.04	56.60
33.33	0	60	0.00
	1	14.94	75.10
	2	13.27	77.89
	3	12.21	79.64
	4	12.09	79.85
	5	12.09	79.85
20	0	60.00	0.00
	1	7.44	87.60
	2	7.13	88.12
	3	6.82	88.63
	4	6.70	88.84
	5	6.70	88.84
13.5	0	60.00	0.00
	1	2.73	95.45
	2	2.67	95.56
	3	2.60	95.66
	4	2.60	95.66
	5	2.60	95.66

Table (A.11) Initial concentration = 80 mg/l

Q (ml/min)	Radiation time unit (i)	Average Final Conc. (mg/l)	Removal (%)
57	0	80	0.00
	1	34.10	57.38
	2	27.28	65.90
	3	26.54	66.83
	4	26.54	66.83
	5	26.54	66.83
33.33	0	80.00	0.00
	1	15.50	80.63
	2	13.64	82.95
	3	12.46	84.42
	4	12.40	84.50
	5	12.40	84.50
20	0	80.00	0.00
	1	9.11	88.61
	2	8.56	89.31
	3	8.37	89.54
	4	8.31	89.62
	5	8.31	89.62
13.5	0	80.00	0.00
	1	3.04	94.94
	2	2.98	95.04
	3	2.91	95.14
	4	2.91	95.14
	5	2.91	95.14

Table (A.12) Initial concentration = 100 mg/l

Q (ml/min)	Radiation time unit (i)	Average Final Conc. (mg/l)	Removal (%)
57	0	100	0.00
	1	43.40	56.60
	2	32.74	67.26
	3	30.81	69.19
	4	30.57	69.43
	5	30.57	69.43
33.33	0	100	0.00
	1	21.02	78.98
	2	18.10	81.90
	3	19.53	80.47
	4	17.86	82.14
	5	17.86	82.14
20	0	100.00	0.00
	1	10.66	89.34
	2	10.23	89.77
	3	9.92	90.08
	4	9.92	90.08
	5	9.92	90.08
13.5	0	100.00	0.00
	1	3.41	94.32
	2	3.29	94.52
	3	3.22	94.63
	4	3.16	94.73
	5	3.16	94.73

Table (A.13) Initial concentration = 120 mg/l

Q (ml/min)	Radiation time unit (i)	Average Final Conc. (mg/l)	Removal (%)
57	0	120	0.00
	1	52.70	56.08
	2	42.16	64.87
	3	39.06	67.45
	4	39.99	66.68
	5	39.99	66.68
33.33	0	120	0.00
	1	28.83	75.98
	2	25.85	78.46
	3	26.07	78.27
	4	25.30	78.92
	5	25.30	78.92
20	0	120.00	0.00
	1	12.33	89.73
	2	12.77	89.36
	3	11.75	90.21
	4	11.67	90.28
	5	11.67	90.28
13.5	0	120.00	0.00
	1	3.66	93.90
	2	3.60	94.01
	3	3.47	94.21
	4	3.41	94.32
	5	3.41	94.32

Appendix (C)

Figures



Fig. (A.1) Modified microwave oven



Fig. (A.2) Peristaltic pump



Fig. (A.3) pH meter



Fig. (A.4) Sensitive balance



Fig. (A.5) Spectrophotometer



Fig. (A.6) Samples examination using Nessler Standard Method



Fig. (A.7) Hand-made heat exchanger