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Theme

Regenerated active carbon (RAC) modified NiO-ZnO photo-catalyst for efficient photocatalytic water splitting and degradation of Methylene Blue Dye

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"آخر دعواهم أن الحمد لله رب العالمين"

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List of abbreviations and symbols

AC	Activated carbon
FAC	Fresh activated Carbon
VOCs	Volatile compounds
SAC	Spent activated Carbon
GAC	Granular Activated Carbon
$\mathbf{A_C}$	Ash content (w %).
IN	Iodine Number
PAC	Powdered activated carbons
EAC	Extruded activated carbons
TEG	Tri-Ethylene glycol
$\mathbf{R_c}$	Regeneration capacity
RAC	Regenerated activated carbon
$\mathbf{C_0}$	Initial concentration of the solution.
$\mathbf{C_t}$	Concentration of the solution attime .
$\mathbf{H_2}$	Hydrogen .
MB	Methylene bleu .
NaOH	Hydroxide sodium.
NiO	Nickel oxide .
ZnO	Zinc oxide .
$\mathbf{O_2}$	Oxygen .
PC	Photo-catalyst .
PD	Photo-degradation.
DR	Dye removal.
WS	Water splitting .
λ_{max}	Wavelength max.
wNA	Weight of Nickel acetate .
wZA	Weight of Zinc acetate.
wRAC	Weight of regerated activated carbon

List of figures

List of tables

Table of contents

General inroduction

Introduction:

Activated Carbon (AC) is a material characterized by its high porosity and great absorbency, which makes it an essential component in many environmental and industrial applications. Among these applications, activated carbon plays a pivotal role in the purification of water and air, in addition to being used in the processing of chemicals. However, the constant use of activated charcoal leads to a deterioration in its performance, which entails its periodic replenishment or replacement [1] .

In recent years, new technologies have emerged to regenerate activated charcoal, aimed at effectively restoring its absorption capacity and improving its economic efficiency by exploiting it in other processes such as photocatalysis, photocatalysis is one of the most innovative and effective technologies. Photocatalysis is based on the use of light rays to stimulate chemical reactions in the breakdown of water and the production of green hydrogen, the most important element in sustainable development

The concept of the hydrogen economy presents a transformative vision for a sustainable energy future. Hydrogen, as a means of energy storage and carrier, offers a potential solution to various critical energy challenges, including energy security, environmental sustainability, and economic development. [2]

Photocatalysis is one of the methods for producing hydrogen from water.

While several methods exist for hydrogen production, photocatalysis offers a clean and sustainable alternative. It utilizes sunlight, a readily available and renewable energy source, to split water molecules (H_2O) into hydrogen (H_2) and oxygen (O_2) [3].

Solar energy is one of the most abundant sources of environmentally friendly energy, with a spectral component that consists of 4% ultraviolet, 46% visible, and 50% near-infrared light. Solar energy can be used as a source to induce chemical reactions through photocatalytic processes. Many materials can be used as a photocatalyst, including TiO, CdS, ZnO, and BiWO. Among the various materials, TiO and ZnO are the most widely used as photocatalysts. TiO has a bandgap similar to that of ZnO (3.20 eV), but the absorption efficiency of ZnO is larger than that of TiO in most solar spectra. In addition, ZnO is considered to be a non-toxic metal oxide and its production cost is relatively low. However, ZnO's photo corrosion issues and wide bandgap can limit its

performance because it only absorbs light in the ultraviolet region and accelerates electron-hole pair recombination. Metallic or non-metallic doping of ZnO can significantly overcome the disadvantages of ZnO by lowering the bandgap, thereby increasing the ZnO response to the visible light region. Several reports related to doping using alkali, transition, and nonmetal can reduce the bandgap, but doping with alkaline metals did not show a significant difference in the bandgap change [4]

Doping using transition metals, such as nickel metal, can improve the performance of ZnO. Nickel ions (0.69 Å) have an ionic radius close to zinc ions(0.74 Å), so nickel ions can easily replace zinc ions in the ZnO crystal lattice causing distortion of the ZnO crystal lattice and shifting to the visible region. In addition, nickel has good corrosion resistance properties, which make it suitable for doping ZnO in photocatalyst applications [5].

The purpose of this study is to regenerate spent activated carbon (SAC) obtained from a unit of dehydration of natural gas (Roudh El Baguel), using different regeneration methods. The regenerated activated carbon (RAC) was used as a support for the synthesized photocatalyst. The synthesis of photocatalyst of Nickel oxide – Zinc oxide doped with RAC was investigated at different weight ratios of ZnO-NiO/RAC. In addition to testing the performance of the synthesized photocatalyst in pure water splitting process, it was tested in the wastewater splitting process using Methylene blue (MB) dye as a pollutant model.

Construction of this thesis

This thesis composes of 3 chapters with a brief description about each chapter as given below:

Chapter I presents generalities and definitions related to the methods of regeneration of spent activated carbon, photocatalysts, and the production of hydrogen .

Chapter II: This chapter describes the experimental equipment and procedures, preparation of solutions, methods of regeneration and protocols used to synthesize the NiO with ZnO/RAC photo-catalyst.

Chapter III: This chapter exposes and discuses all results figure out from adequate several experiments, these results expressed in plots and tables of regeneration methods data.

This discussion ended by a general conclusion.

Chapter I: Literature Survey

I.1. General information on activated carbon :

Activated carbon (AC) is a porous form of coal that can be manufactured from various carbonaceous raw materials such as coconut shells, almond fruit shells, peach pits, grape seeds, apricot kernels, cherry pits, olive pits, peanut shells, rice husks, palm oil cans, and bagasse. These are generally the most commonly used adsorbents for the removal of organic compounds and microorganisms in air and water. Anyone with high carbon content can be used as raw material for producing activated carbon. AC is commonly used to treat and decontaminate waste and wastewater, mainly due to its very porous structure and its large specific surface area. The physical and chemical properties of activated carbon depend essentially on the preparation conditions, which can be based on the physical or chemical activation processes favor the formation of new pores. It is the expansion of the pore size of AC during the activation process, that effectively adsorbs microorganisms and other dyes from aqueous solutions [7].

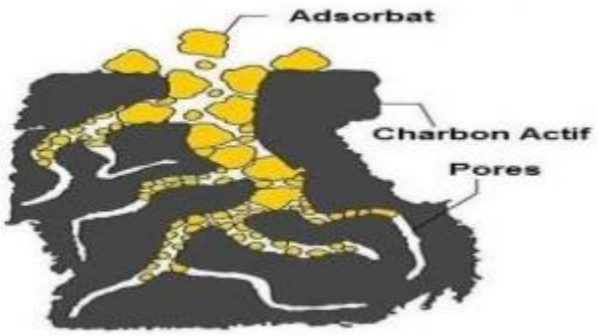


Figure I.1. Structure of an activated carbon **[7]**

I.1.1.The types of activated carbon:

The activated carbons are available either in powder (PAC) or in grains (GAC) and extruded (EAC) as summarized in **Table I.1.**

Table I.1. The different types of AC [8]

Powdered activated carbons	**Granular activated carbons**	**Extruded activated carbons.**
Particles with a size of less than 0.18 mm - Mainly used in liquid phase applications and for the manipulation of gaseous flows. - 2 to 3 times cheaper than (GAC). 	-Irregularly shaped particles, varying in size from 0.2 mm to 5 mm - For liquid phase (water treatment) and gas phase applications. This compound has low Molecular weight contributes to longevity, and Has a high adsorption capacity 	- Cylindrical, whose diameter varies from 0.8 mm to 5 mm - Mainly used in gas phase applications due to its low pressure drop and low dust content 

I.1.2.Porous structure of activated carbon:

Also known as activated charcoal is a form of carbon processed to have small, low-volume pores that increase the surface area available for adsorption or chemical reactions [6].

Carbon surface has a unique character. It has a porous structure which determines its adsorption and desorption capacity, it has a chemical structure which influences its interaction with polar and nonpolar adsorbates, and it has active sites in the form of edges, dislocations and discontinuities which determine its chemical reactions with other atoms. Thus, the adsorption behavior of AC cannot be interpreted on the basis of surface area and pore size distribution alone [6].

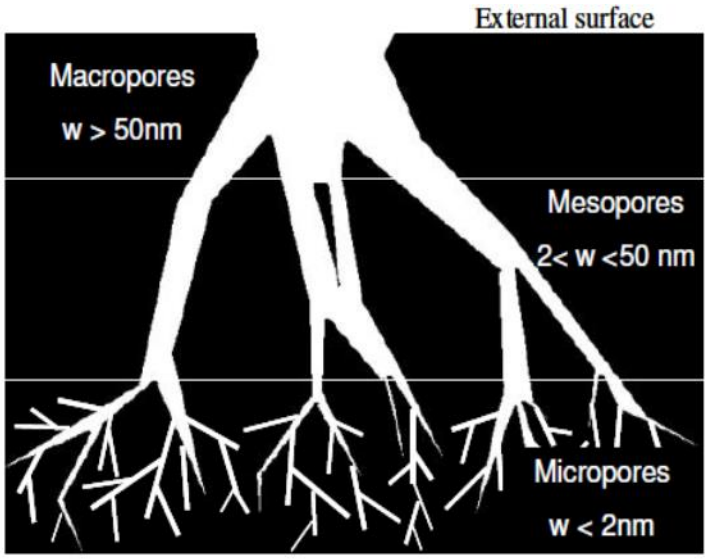


Figure I.2. The pore structure of activated carbon [9]

I.1.3. Definition of adsorption :

Adsorption is a phenomenon of fixation of molecules of a fluid on the surface of a solid, which increases the concentration of the molecules of this fluid on the surface of the solid without modifying the volume of this porous medium[6].

It is a physical and or chemical process in which a substance accumulates at the interface between the phases, the latter can be solid-liquid, liquid-liquid, gas-liquid, or gas-solid. The adsorbate is the substance removed from the liquid phase and the adsorbent: is the solid phase in which the accumulation takes place [6].

A solid material in general appears a heterogeneous distribution of surface energy; fluids molecules may become bound to the surface if they move toward sufficiently close to interact [10].

The number of molecules attracted by the surface of adsorbent depends on more than a few conditions and surface features including temperature, pressure, surface energy distribution, and the surface area of the adsorbent. A graph that represents the variation in the amount of adsorbate **(x)** adsorbed on the surface of the adsorbent with the change in pressure at a constant temperature is called the adsorption isotherm [10].

The main adsorptions isotherms have been proposed by different scientists are:

- Langmuir isotherm
- Freundlich isotherm
- BET theory

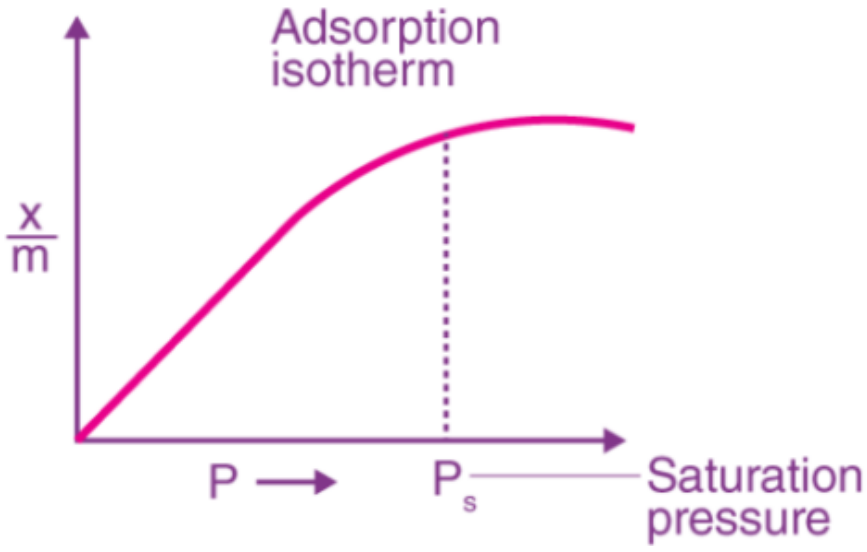


Figure I.3. Isotherm proposed by freundlich [10]

Where:

x = adsorbate mass;

m = adsorbent mass;

p = equilibrium pressure [10]

I.1.4. Adsorption mechanism:

The adsorption of molecules of an initial solute existing in solution on porous solid adsorbents as a function of time is described in four steps.

1. Transfer of matter by diffusion of solute from the external liquid phase to the liquid phase near the absorbed surface.
2. Transfer of solute to the surface by a liquid layer attached to the solid particle. Grains from the outside.
3. Transfer of material into the porous structure of the outer surface of the grain.

4. Adsorption at the active sites; the molecule is considered to be immobile once adsorbed [11].

I.2. Desorption (Regeneration):

Industrial companies day by day look for high capacity adsorbents which have high surface area and also are specific to the target products. This need Stimulates and enhances huge researches in the adsorption field, through developing the specification and performance of adsorbents. However, these absorbents are very expensive for companies, which have led researchers to be interested in the possibility of regenerating adsorbent in general using many methods and techniques.

Regeneration have been also referred as reactivation, literature survey suggests that regeneration is better term for reuse of adsorbents as it includes both desorption and activation and also carves difference between desorption and regeneration [10].

I.2.1. Thermal regeneration:

Thermal regeneration of SAC is a simple regeneration method but is an energy and time consuming method and causes a considerable amount of carbon loss, it involves three steps: drying for 30 min, pyrolysis of adsorbates about 5 min, and reactivation (oxidation of residue from the adsorbate) for 10 min. It is a method of thermally processing the activated carbon to destroy the adsorbed components contained on its surface [10].

As the temperature is raised to 200 °C, the SAC dries and volatile adsorbates are released as gases. As the temperature is raised to 400-600 °C, reversibly adsorbed substances are driven off while irreversibly adsorbed substances decompose and leave behind a char residue

Then the SAC exposed to heat between 870-1000 °C in a presence of inert gas (CO_2), intention to oxidize the char residue, the detention time and amount of oxidant required is dependent on the particular adsorbate [12]

I.2.2. Chemical regeneration:

An alternative approach to the thermal regeneration method is the chemical regeneration method in which chemical reagents are applied to the spent carbon . Traditionally, acid (HCl, H_2SO_4), and

alkali solutions (with oxidizing capacity) as NaOH or KOH, and organic solvents (solubilizing capacity) have been used to dissolve the adsorbates, so that the ability of adsorption of the activated carbon will be recovered. This regeneration method becomes valuable, particularly when adsorbates have strong bonds with the adsorbent's surface [13]

The main advantages of the chemical regeneration method include high regeneration efficiency, high speed regeneration and low carbon loss [13]

I.2.3. Steam regeneration:

Steam regeneration is extensively used process, cheap, readily available in industry; steam generation could be by skid mounted boiler units which are available at relatively low cost. Steam works especially well with hydrophobic organics, such as chlorinated solvents. Hydrophobic adsorbates have an added advantage in that they can be separated from the condensed water by gravity [14]

Steam regeneration performed at a range of temperature from 140 to 280 °C, this regeneration temperature and period set depending on the adsorbate properties especially the boiling point of the VOCs and attractions forces to the adsorbent. The regeneration must perform at a temperature superior to the boiling point of adsorbate. According to Shah et al, it is typical to regenerate with steam at 30 – 50 °C above the VOC boiling point [10].

I.3. Hydrogen:

The cleanliness of hydrogen and the efficiency of fuel cells taken together offer an appealing alternative to fossil fuels. Implementing hydrogen-powered fuel cells on a significant scale, however, requires major advances in hydrogen production, storage, and use. Splitting water renewable offers the most plentiful and climate-friendly source of hydrogen and can be achieved through electrolytic, photochemical, or biological means. Whereas presently available hydride compounds cannot easily satisfy the competing requirements for on-board storage of hydrogen for transportation, nanoscience offers promising new approaches to this challenge. Fuel cells offer potentially efficient production of electricity for transportation and grid distribution, if cost and performance challenges of components can be overcome. Hydrogen offers a variety of routes for achieving a transition to a mix of renewable fuels [15]

I.3.1. Hydrogen production method:

To produce hydrogen, it must be separated from the other elements in the molecules where it occurs. Hydrogen can be produced from many different sources in different ways to use as a fuel. The two most common methods for producing hydrogen are steam-methane reforming and electrolysis (splitting water with electricity) [16].

In Table 2 we will present the most used sources in the production of hydrogen [16]

Table I.2. Origine of hydrogen product (coproduction incluse)

Origin of the hydrogen	Pourcentage
Percentage Natural gas	49%
Liquid hydrocarbons	29%
Coal	18%
Electrolysis	4%

I.3.1.a. Reforming fossil fuels with water vapor (or vapor reforming):

Reference process, the most economical (but its cost price remains much higher than that of natural gas). Steam reforming of natural gas is the most common method. It breaks the methane molecule (CH4), the main component of natural gas, which has 4 hydrogen atoms, with water vapor at 900 ° C. Two successive reactions produce hydrogen (H_2) and carbon dioxide (CO_2):

$$\mathbf{CH_4 + 2\ H_2O \longrightarrow 4\ H_2 + CO_2} \qquad \mathbf{(I.1)}$$

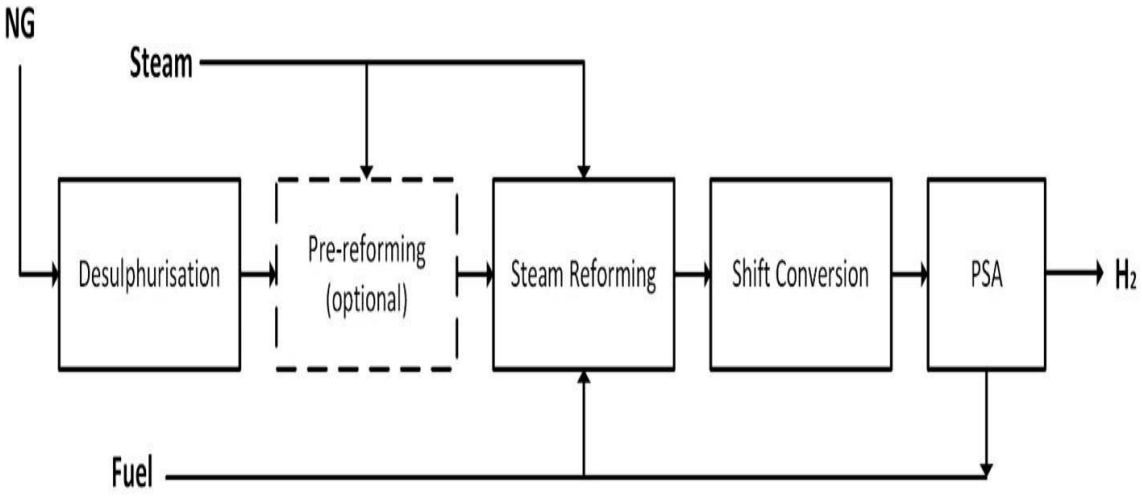


Figure I.4. Common process flow for a steam reforming plant [17]

I.3.1.b. Electrolysis of water:

A process that requires electricity (profitable if the production of electricity itself has a low cost). The electric current decomposes the water molecule into hydrogen and oxygen (O_2) [18]

$$H_2O + \text{electricity} \longrightarrow H_2 + \tfrac{1}{2} O_2 + \text{heat} \qquad \textbf{(I.2)}$$

This process corresponds to the reverse reaction of that occurring in a fuel cell. It makes it possible to produce a very "clean" hydrogen (if the electricity is produced using renewable sources) but is not yet economically viable (2 to 3 times more expensive than the steam reforming process). The efficiency of this technique is 40% over the entire chain but can reach 80% by recovering the heat [18].

I.3.1.c. Gasification and pyrolysis of biomass (in particular charcoal):

A process under research and development which makes it possible, for example, to obtain hydrogen by chemical transformation of wood at very high temperatures (between 1,200 °C and 1,500 ° C). A mixture of gases containing hydrogen (H_2) and carbon monoxide (CO) is obtained. After purification of this mixture, hydrogen is obtained

I.3.1.d. Photolysis:

is part of the light-dependent reaction or light phase or photochemical phase or Hill reaction of photosynthesis. The general reaction of photosynthetic photolysis can be given in terms of photons as [19]:

$$H_2O\ (I) + H_2O \longrightarrow H_2\ (g) + 1/2\ O_2\ (g) \qquad \textbf{(I.3)}$$

The chemical nature depends on the type of organism. Purple sulfur bacteria oxidize hydrogen sulfide (H_2S) to sulfur (S). In oxygenic photosynthesis, water (H_2O) serves as a substrate for photolysis resulting in the generation of diatomic oxygen (O_2). This is the process which returns oxygen to Earth's atmosphere. Photolysis of water occurs in the thylakoids of cyanobacteria and the chloroplasts of green algae and plants [19].

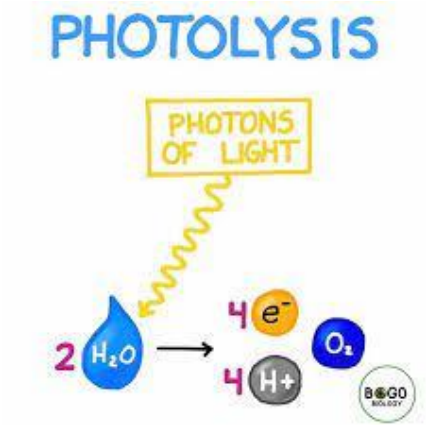


Figure I.5. Photolysis

I.3.2. The advantages and disadvantages of hydrogen:

The advantages of hydrogen are numerous: This is a real solution to replace fossil fuels (especially for transport because autonomy is great).

We consume energy without directly emitting greenhouse gases. But this technology must also improve to :

- can only be produced from renewable sources, which is not the case at all today. can last longer and be cheaper.
- to improve efficiency because all these conversions, it leads to losses!
- recycle all used materials [7].

I.4. Photocatalysis :

Is an advanced oxidation technology, which is based on the activation of a semiconductor by light. The materials likely to cause these reactions are often based on titanium dioxide. After having explained the principle of photocatalysis, a detailed presentation of the environmental applications is conducted. Those for water treatment cover inorganic and organic pollutants. Disinfection by photocatalysis is a very explored field, even if the understanding of the mechanisms of action against microorganisms needs to be deepened. The applications for air treatment concern the removal of nitrogen oxides and the treatment of volatile organic compounds for indoor air. Finally, the self-cleaning surface properties recovered from titanium dioxide are recalled [20].

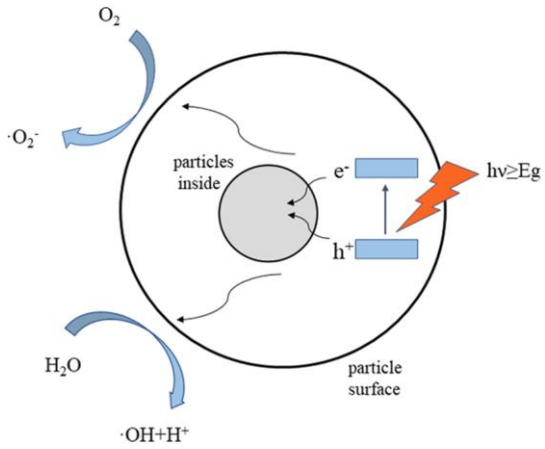


Figure I.6. Mechanism-of-the-photocatalytic-water-splitting

I.4.1.Photocatalytic water splitting :

I s a process that uses photocatalysis for the dissociation of water (H_2O) into hydrogen (H_2) and oxygen (O_2). The inputs are light energy (photons), water, and a catalyst(s). The process is inspired by Photosynthesis, which converts water and carbon dioxide into oxygen and

carbohydrates. Water splitting using solar radiation has not been commercialized [1]. Photocatalytic water splitting is done by dispersing photocatalyst particles in water or depositing them on a substrate, unlike Photoelectrochemical cell, which are assembled into a cell with a photoelectrode[2].

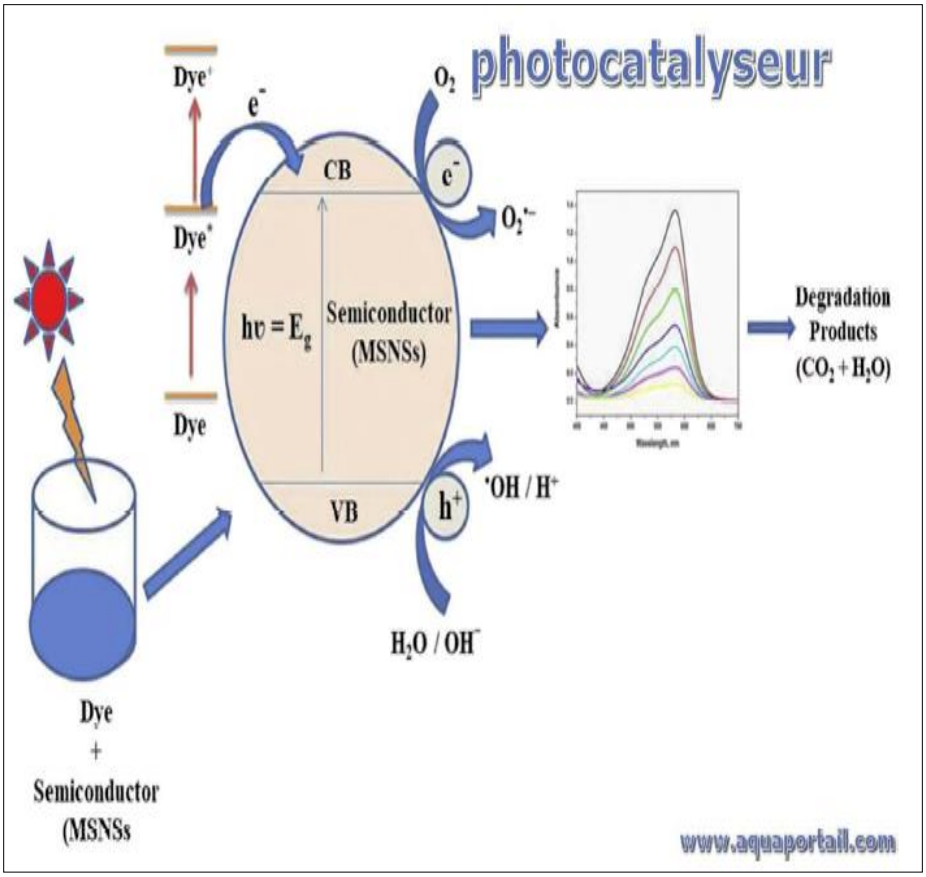


Figure I.7. Principle of photo-catalyst

Chapter II: Experimental setup and Conditions

II.1. Introduction

This chapter demonstrates the experimental procedures, methods, apparatus, and chemicals included in the experiment of regeneration of the spent activated carbon and the experimental for the synthesis NiO-ZnO/AC photocatalyst, in addition to the experimental setup to test the ability of the synthesized photocatalyst in pure water splitting to hydrogen and oxygen.

And finally, the experimental setup for testing the ability of the synthesized NiO-ZnO/AC photo-catalyst in wastewater splitting to hydrogen and oxygen.

II.2. Material

In this study, spent activated carbon (SAC) grains were used as a feedstock to prepare regenerated activated carbon (RAC), which was used as a support to the synthesized photo-catalyst. All chemicals used in the investigation such as KOH, Nickel acetate, Zinc acetate, Methylene Blue, NaOH and HCl were of analytical grade.

II.2.1. Spent granular activated (SAC):

The SAC used for the experiment were obtained from activated carbon filters, used in TEG dehydration unit, located in Rourd El Baguel TCF (Turbo compressors facilities) gas reinjection plant in Algeria. Table 3 shows the physical-chemical proprieties which extracted from the Certificate of Analysis 2021 According with GB/7702.7-1987 standard.

Table II.1. Fresh activated carbon properties[21].

Sample	Particle size (mm)	IN (mg/g)	Specific Area (m^2/g)	Hardness (%)	Bulk Density (g/cm^3)	Ash (%)	Moisture (%)
GAC 1122-C	1.5 – 1.7	**> 900**	**> 850**	**98**	**0.45**	**6**	**4**

II.3.Preparation of photo-catalysts

A two-step process was used to prepare the photo-catalyst, firstly regeneration of SAC, and secondly, doping of activated carbon with photo-catalysts.

II.3.1. Regeneration of spent activated carbon (SAC):

Various methods of regeneration were used in this study to regenerate the SAC, including thermal, chemical, steam, and chemical followed by steam regeneration.

II.3.1.1. Thermal regeneration:

Samples of SAC were regenerated thermally, by exposing 8 g of SAC to heat in a muffle furnace at a temperature of 900 °C for 1hour, as illustrated in Figure 8.

FigureII.1. Thermal regeneration in muffle furnace

II.3.1.2. Chemical regeneration:

The SAC was regenerated chemically using two different solutions hydrochloric acid (HCl) solution and sodium hydroxide (NaOH) solution. A sample of 1 g of SAC was mixed with 20 ml of solution in a shaker at room temperature (around 40 °C) for 30 minutes, the mixture was filtered and washed with hot water and the sample is then dried in an oven at 105 °C. [11]

II.3.1.3. Steam regeneration:

Experimental steam regeneration was conducted with a small glass fixed bed reactor (din = 15 mm, L = 40 cm); which was placed vertically in a tubular furnace. A sample of 5 g of EAC was placed in the reactor furnace at 104 °C. The regeneration time of each sample was fixed at 2 hours. Pumping water into the reactor at different flow rates was achieved using a flow meter, as illustrated in Figure 9.

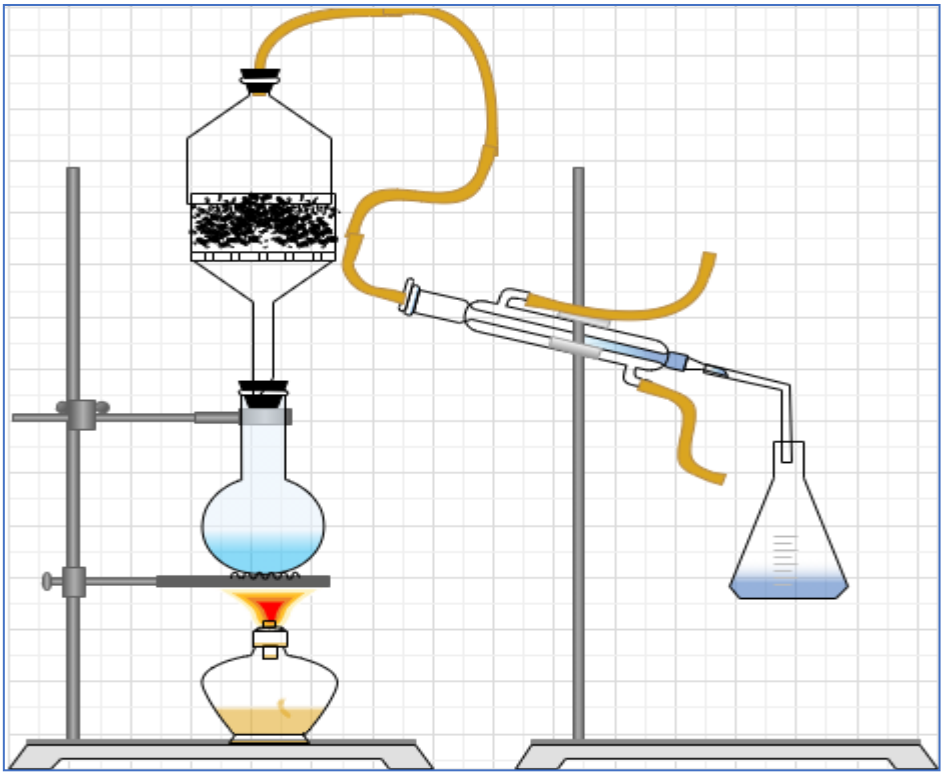


Figure II.2. Steam regeneration apparatus

II.3.1.4. Chemical followed by the steam regeneration:

As a combined method, SAC was pretreated for 30 minutes with dilute acid HCl of 5% of concertation in case of acid wash and with 4 M of NaOH in case of alkali wash and then placed in a reactor of steam regeneration for 2 hours at 104 °C with a steam flow rate of 1,5 kg/h [11].

II.3.1.5. Chemical combined with thermal regeneration:

A regeneration method combining chemical and thermal processes was used to regenerate the SAC, by mixing KOH with SAC, the ratio is 4:1 (four grams of KOH for every one gram of carbon), the mixture was then introduced to a tubular furnace at a temperature of 700 °C for an hour, with the presence of nitrogen gas at a flow rate of 0.8 ml/h, as shown in the **Figure 10.** The obtained AC was washed, and its pH was monitored until obtaining a neutral value with a portable pH meter.

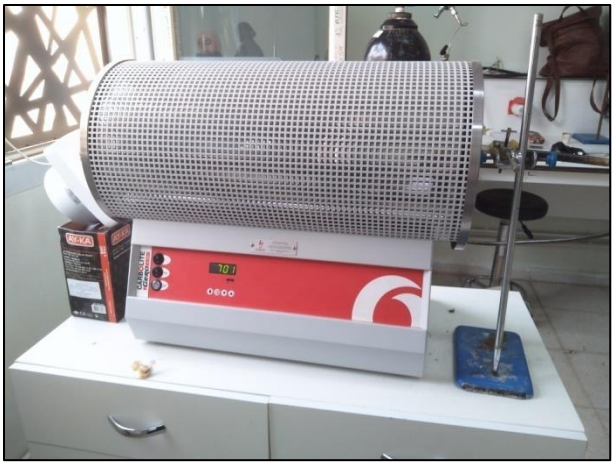


Figure 8. Tubular furnace used in the SAC regeneration process

II.3.2. Synthesis of NiO-ZnO supported regenerated activated carbon (RAC) composite

The method of preparing nickel oxide NiO-ZnO/RAC photo-catalyst supported by regenerated activated carbon (RAC) is based on the precipitation method. In the first step, solution A was prepared in a beaker by dissolving a mass (w_{NA}) of Nickel acetate and a mass (w_{ZA}) of Zinc acétate in 50 ml of distilled water under continuous stirring for 30 minutes, and another solution B was prepared by dissolving a mass of RAC (w_{RAC}) of AC in 50 ml. of distilled water with continuous stirring for 30 minutes, then pouring solution A into B with continuous stirring for an hour at a temperature not exceeding 180 °C until all the waterevaporates, then dry the mixture in an oven at a temperature of 120 °C for a period of 1 to 5 hours to produce the NiO-ZnO/RAC PC.

RAC is one of the most important candidates for the manufacture of high-performance NiO-ZnO/RAC nanospheres due to its large surface area, high conductivity, and high durability [23-24] .Moreover, due to its environmental friendship, abundance, rapid renewal, and low cost, the biomass-derived AC has attracted wide attention from the next-generation energy technology community [23-24].

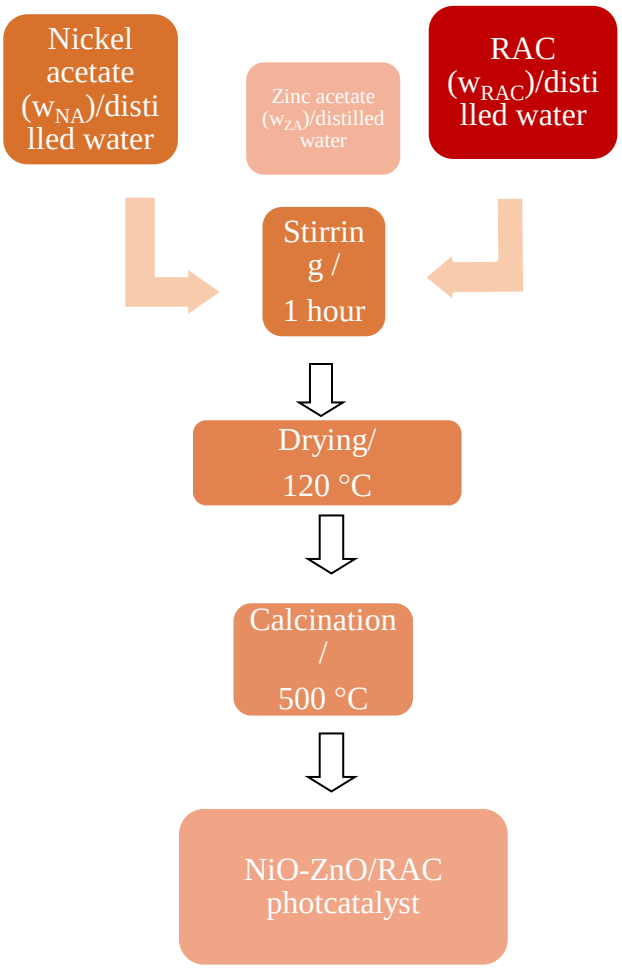
In this study, four different photo-catalysts were prepared with different weight ratios of nickel oxide andzinc oxide (NiO-ZnO/RAC) regenerated activated carbon, in order to study the weight ratio on the performance of the photo-catalyst, as summarized in **Table II.2**.

Table II.2. Four different photo catalysts were synthesized in this study.

Representation of photo-catalyst	Weight of NiO (w_{NA}) (g)	Weight of ZnO (w_{ZA}) (g)	Weight of RAC (w_{RAC}) (g)
3/7 NiO-ZnO/RAC	1,5	1,5	7
4/6 NiO-ZnO/RAC	2	2	6
5/5 NiO-ZnO/RAC	2,5	2,5	5

II.3.2.1. Calcination

Calcinations is the last stage in the preparation of the photo-catalyst, the NiO-ZnO/RAC powder prepared from the synthesis is poured into a ceramic crucible and placed in the muffle furnace at a temperature of 500 °C for 3 hours to prepare the nano composite The general protocol followed in this study is described in **Figure II.4**.



FigureII.4. Method for preparing the NiO-ZnO/RACphotocatalyst.

II.4.Photo-catalytic activity experiments

An experiment was conducted with 1g of NiO-ZnO/RAC added to 1200 ml of water (messed water) to examine how the photo-catalyst enhanced the water-splitting process in the electrolyse as shown in the **Figure II.5**, an electric generator is used to generate an electric current of 24 volts. The experimental set-up for the electrolysis test is exposed to the visible light of 1000 W. The flow rates of produced gases including oxygen and hydrogen produced through the water splitting process are measured during the test.

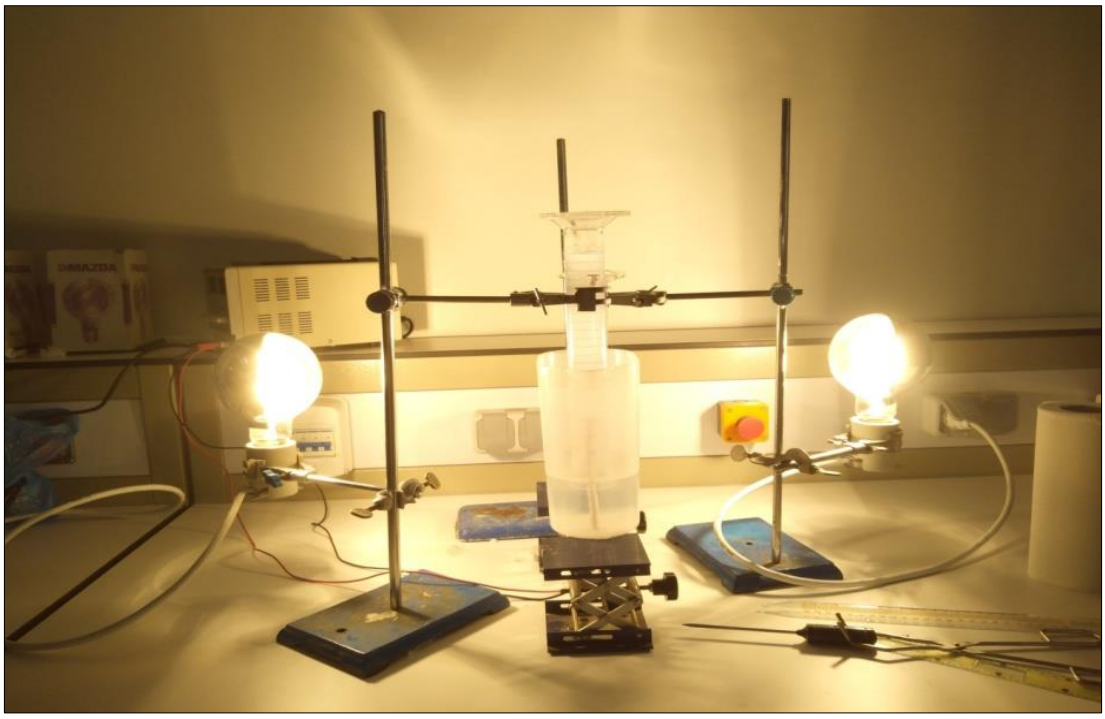


Figure II.5. Experimental set-up for electrolysis test

II.4.1. Measurement of the flow rate of produced gases

The flow rates of hydrogen and oxygen or the volume of the produced gases are measured every 10 minutes during the water-splitting process, two graduated cylinders filled with the same solution used in the electrolyse, and placed inversely above the anode and the cathode to measure the volume of produced oxygen and hydrogen respectively. Temperature was also measured by using a thermometer **Figure 13**.

Figure II.6. The measurements of the produced gases during the water-splitting process

II.4.2. Determination of(λ_{max}) and calibration curve

In addition, the performance of the NiO-ZnO/RAC photo-catalyst was also tested in the splitting of colored water process and the dye photo-degradation simultaneously. Methylene Blue (MB) dye was chosen in this study due to its known strong adsorption on materials solids, and their recognized usefulness in describing adsorptive materials, often serve as a model for the removal of organic pollutants and colored particles from aqueous solutions [25].

A 1200 ml solution of 20 ppm of MB dye was prepared, 1 g of NiO-ZnO/RAC photo-catalyst, and 2 g of NaOH were added, and then the mixture was poured into the electrolyse. Every 10 minutes, 10 ml of the solution was drawn to measure its absorbance using a UV-Vis spectrophotometer (SHIMADZU 1280) As the **Figure 14**. shows

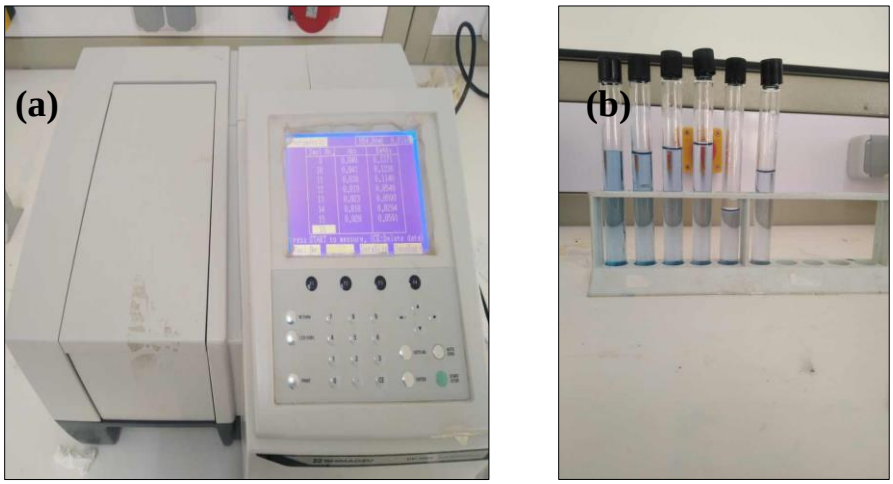


Figure II.7. (a)UV-Vis spectrophotometer (SHIMADZU UV 1280),(b) Degradation of the MB dye of solution

II.4.2.1. Calibration curve for Methylene Blue (MB)

The calibration curve is created by preparing several diluted solutions with different concentrations of MB dye.Absorption is measured at the maximum wavelength(λ_{max}) .

The amount of BM photo-degraded over NiO-ZnO/AC photo-catalyst is calculated by the following relation:

$$R=(C_0-C_t)/C_0*100 \quad \text{.....(II.1)}$$

Where:

C_0: Initial concentration of the solution (mg/L)

C_t: Concentration of the solution at time t (mg/L)

R:Pd rates (%)

II.5. Parameters affecting photo-catalytic activity

To deeply understand the performance of the water-splitting process over NiO-ZnO/RAC photo-catalyst, the effect of several parameters was investigated in this study, such as time, light source, NaOH mass, weight ratio of NiO-ZnO/AC.

In addition, the performance of the NiO-ZnO/RAC photo-catalyst was also tested in the presence of 20 ppm of Methylene Blue (MB) dye in the water. **Table5**. Summarize the parameters investigated in this study.

Table 3. Parameters affecting photo-catalytic activity.

	Mass of NaOH (g)	Catalyst type	Time (min)	Light source (W)	Volume of water (ml)	Temperature (^{0}C)	Initial concentration of MB dye (ppm)	Mass of catalyst (g)
Serie (1)	1 2 3	/ / /	40 180 40	1000	1200	23-60	/ / /	/ / /
Serie (2)	2	5/5 NiO-ZnO/RAC 4/6 NiO-ZnO/RAC 3/7 NiO-ZnO/RAC	180 180 180	1000	1200	22-62	/ / / /	1 1 1 1
Serie (3)	2	5/5 NiO-ZnO/RAC 4/6 NiO-ZnO/RAC 3/7 NiO-ZnO/RAC	120 120 120	1000	1200	20-64	20	1 1 1

Chapter III:

Results and discussion

III.1.Comparasion between regeneration methods:

A comparison of different regeneration methods was conducted in this study. The efficiency of each regeneration method of SAC is expressed by the iodine number (IN) of RAC, as it is represented in Figure 15. It is noted that the IN of RAC increased after regeneration compared to its initial value of SAC (266,49 mg/g). In terms of efficiency, the most efficient method was followed by the least efficient method as follows: Thermal combined with chemical method, thermal, chemical with NaOH Followed with steam regeneration, Chemical with HCl followed with steam regeneration, chemical with HCl, Chemical with NaOH, and finally steam regeneration. Suggesting that thermal combined with chemical as the best method for efficient regeneration of SAC, offering an IN of 1332,49 mg/g.

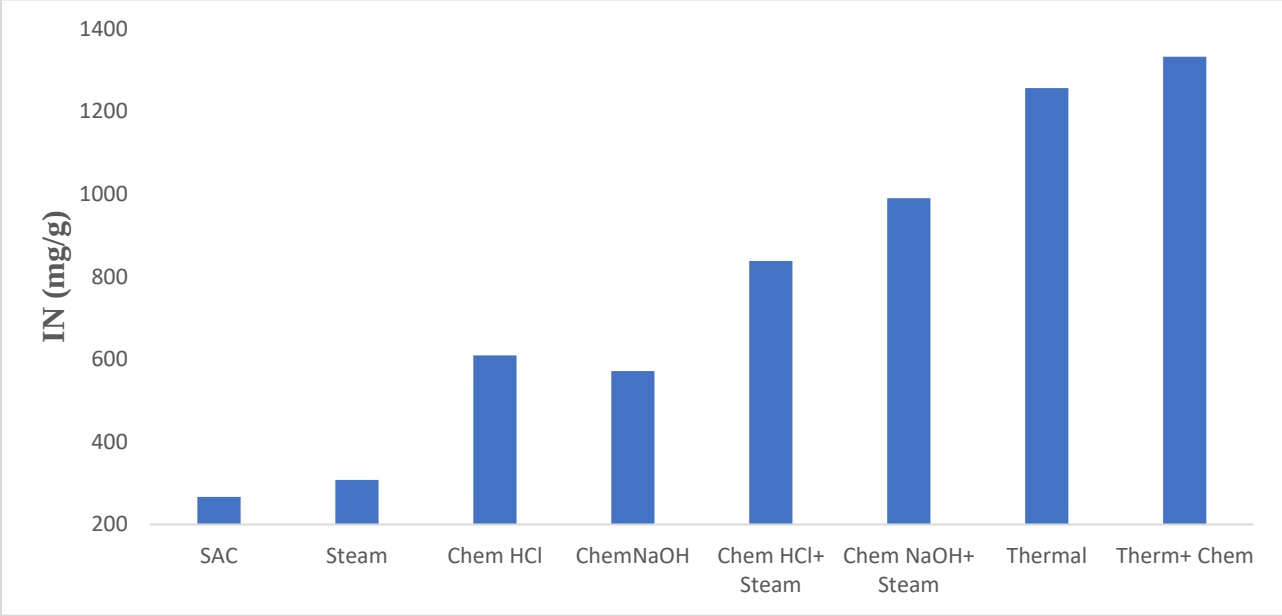


Figure III.1. Effiecincy of differents methods of regeneration

III.2.Electrolytic activity

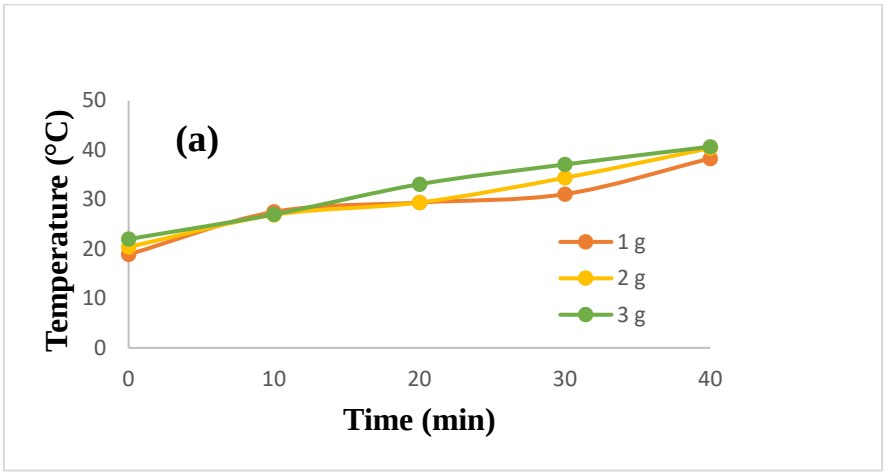
In second step has studied the production of hydrogen and oxygen by the process of splitting water, the performance of sodium hydroxide mass (1, 2, 3 g) H2 and O2 productivity under the influence of light rays (1000 W), and electric current.

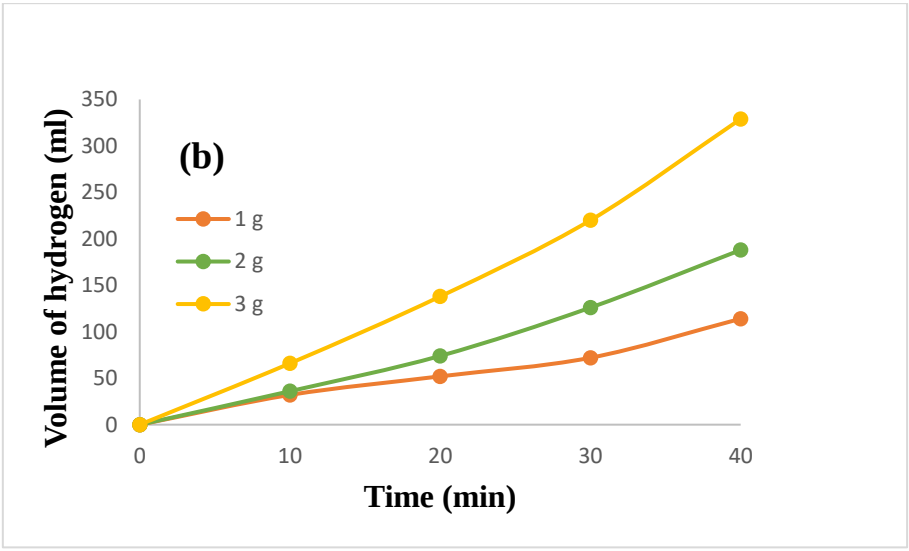
III.2.1. Effect of NaOH mass

Figure 16 shows the effect of the sodium hydroxide mass on the efficiency of the water splitting process and the total productivity of hydrogen and oxygen.The NaOH mass strongly affects the efficiency of the water-splitting process, as the NaOH mass increases the water-splitting's efficiency increases, leading to higher hydrogen and oxygen productivity.

The electrolysis of water with the addition of 1 g of NaOH produced (115 ml) of hydrogen and (0.09ml) of oxygen. The water-splitting process with 3 g of NaOH reached the highest production of hydrogen(329 ml) and oxygen(30 ml).However, all three experiments were conducted at the same temperature interval, as shown in Figure 16(a).

It is noted that the hydrogen gas produced has increment in the addition of the mass of sodium hydroxide solution. This condition shows that the higher the mass of the NaOH electrolyte solution, the formed electrons are denser and more numerous, making it easier to transfer electrons from the solution to the electrode [21]





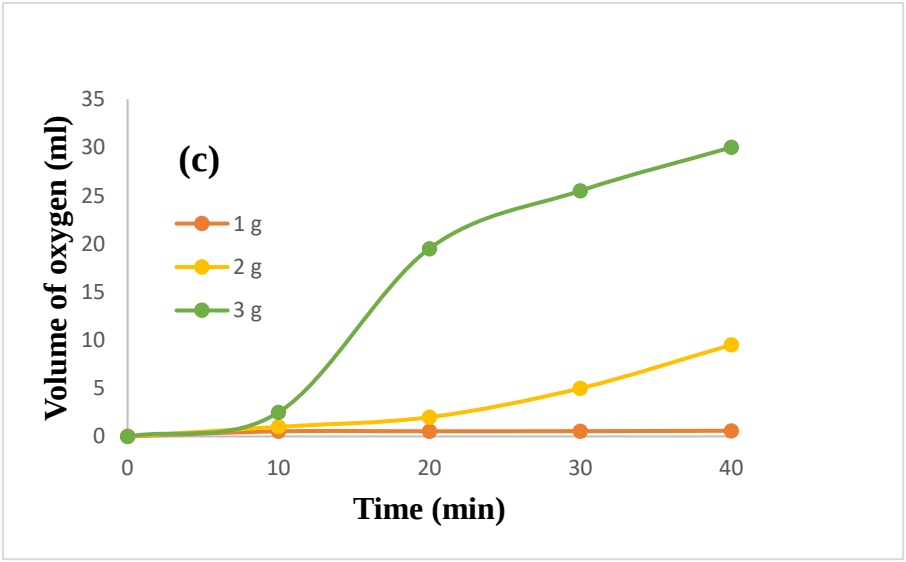


Figure III.2. Effect of NaOH mass on electrolytic activity, variation of (a- Temperature, b- Volume of hydrogen produced, and c- Volume of oxygen)

III.3. Photo-catalytic activity

The performance of photo-catalytic water splitting was studied and compared with electrolyte in terms of hydrogen and oxygen production

III.3.1. Photo-catalytic water splitting

Figure 17 shows the performance of 1 g of the 5/5 NiO-ZnO/RAC photo-catalyst in the water splitting process, it is observed that the effect of the photo-catalyst on the water splitting is divided into two phases, in the first phase (0-100 min) the productivity of hydrogen and oxygen was identical, this indicates that the process of splitting water through an electrolyte is identical to the process of splitting water through a photo-catalyst, which means that the effect of the 5/5 NiO-ZnO/RAC photo-catalyst is neglected. In the second phases(150-180 min) increase in productivity of hydrogen and oxygen,which means that the effect of 5/5 NiO-ZnO/RAC photo-catalyst is a noticed on the efficiency of the WS process.

Previous studies have proven that the NiO-ZnO/RAC photo-catalysis a good candidate in WS process[27-29].

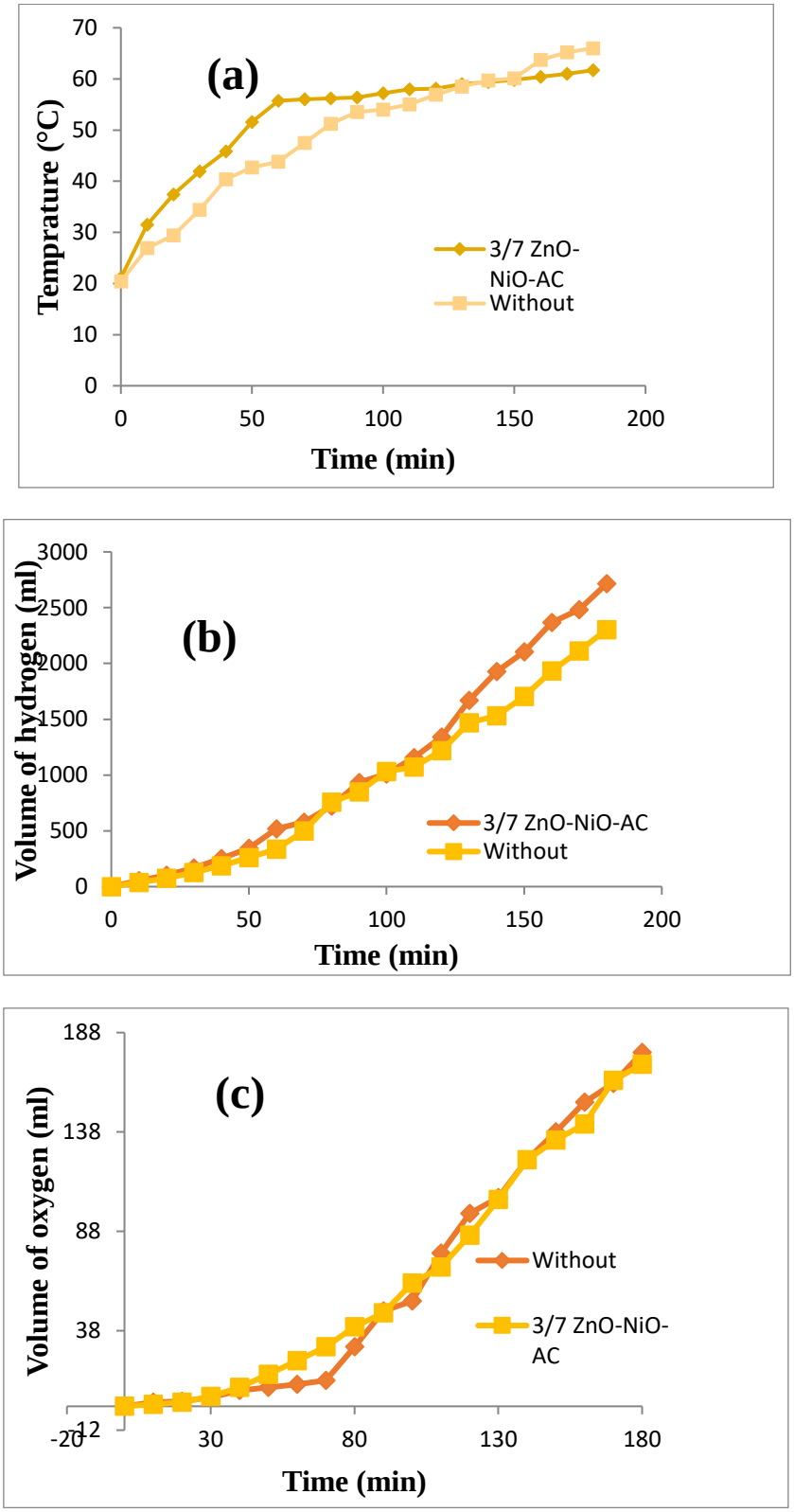


Figure III.3. Photocatalytic activity of 3/7 NiO-ZnO/RAC, variation of (a- Temperature, b- Volume of hydrogen produced, and c- Volume of oxygen)

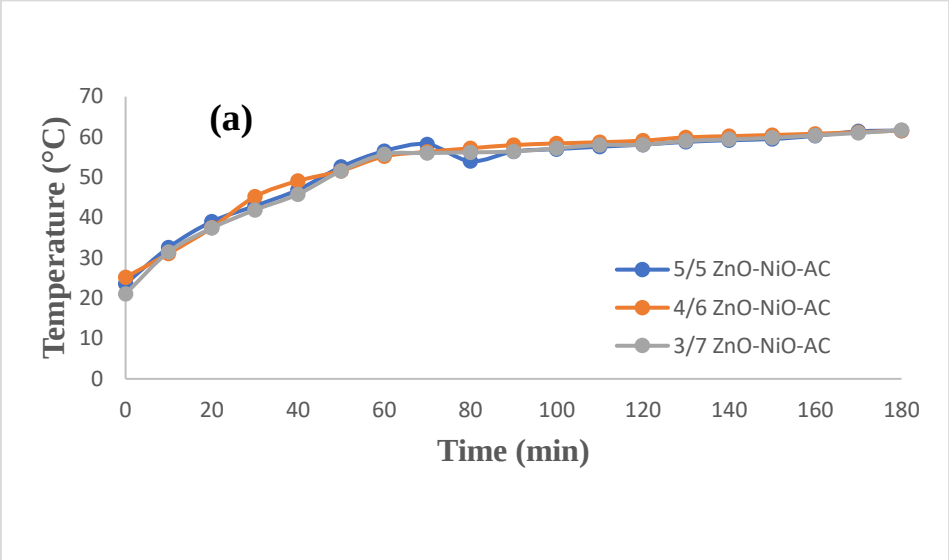
III.2.1.1.Effect of the weight ratio of NiO-ZnO/Regenerate activated carbon(RAC)

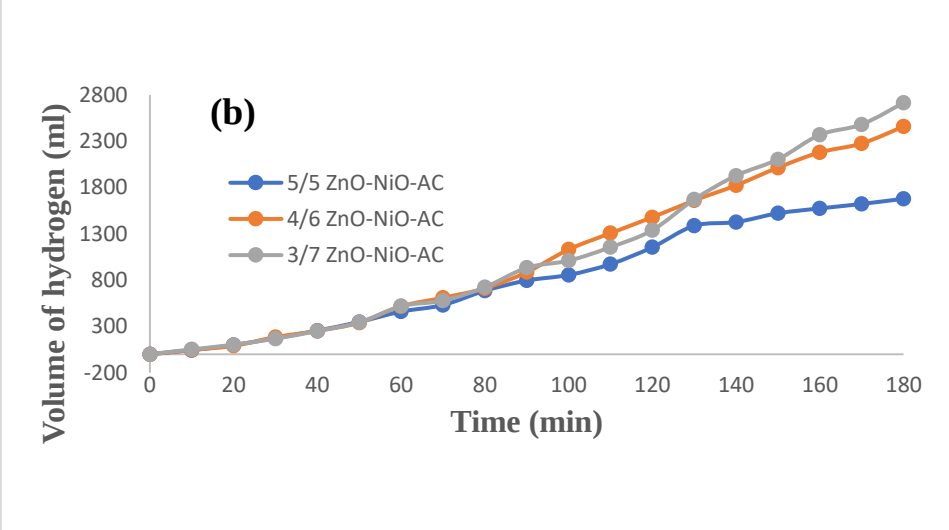
Figure 18 represents the effect of the NiO-ZnO/RAC weight ratio on the efficiency of the water-splitting process. Viewed from the **figure 18 (b),** the volume of hydrogen produced by the water splitting process increases with a decrease in the weight ratio of NiO-ZnO/RAC from 3/7 to 5/5 this means that an increase in RAC has a positive effect on hydrogen production.

In addition, after this, a decrease from 4/6 to 3/7 has the opposite effect, since it is noticed that the volume of hydrogen produced decreases.

The optimal ratio is considered to be 3/7 zinc oxide-RAC for the best performance of the water-splitting process.The previous study proved that the addition of significant amounts of activated carbon to NiO-ZnO/RAC gives good decomposition results, this may be explain by the efficiency of the semiconductor system is improved by increase its surface area and make appropriate adjustments to it surface sites[30-31].

The aggregated structure of the NiO-ZnO/RAC composites could improve its performances because of the increases in both the electrochemically active area and the electrical conductivity due to the incorporation of RAC [32].


(a)
Temperature (°C)
70
60
50
40
30
20
10
0
0
20
40
60
80
100
120
140
160
180
Time (min)
5/5 ZnO-NiO-AC
4/6 ZnO-NiO-AC
3/7 ZnO-NiO-AC


(b)
Volume of hydrogen (ml)
2800
2300
1800
1300
800
300
-200
0
20
40
60
80
100
120
140
160
180
Time (min)
5/5 ZnO-NiO-AC
4/6 ZnO-NiO-AC
3/7 ZnO-NiO-AC

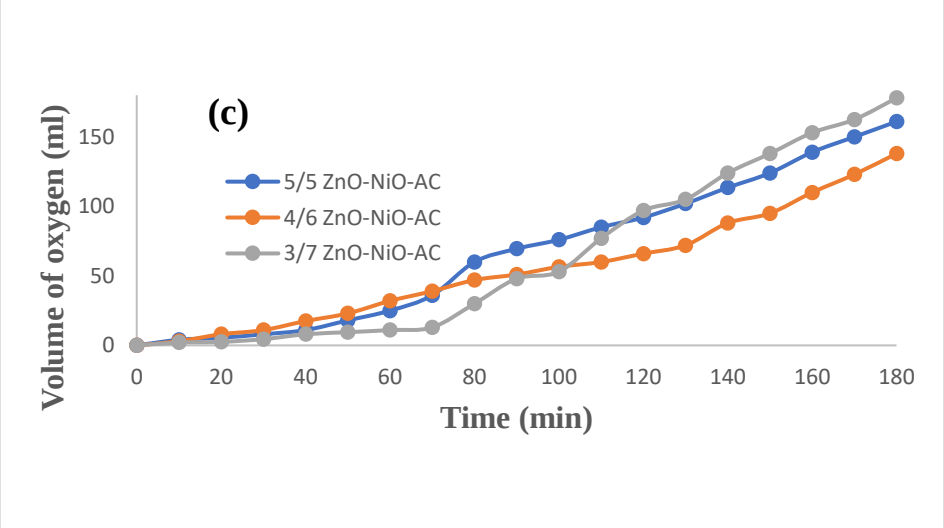

(c)
Volume of oxygen (ml)
150
100
50
0
0
20
40
60
80
100
120
140
160
180
Time (min)
5/5 ZnO-NiO-AC
4/6 ZnO-NiO-AC
3/7 ZnO-NiO-AC

Figure III.4. Effect of the weight ratio of NiO-ZnO and regenerate activated carbon (RAC) on photocatalytic activity of NiO-ZnO/RAC variation of (a- Temperature, b- Volume o hydrogen produced, and c- Volume of oxygen)

III.2.2. Photo-catalytic coupled-dye degradation, and water splitting

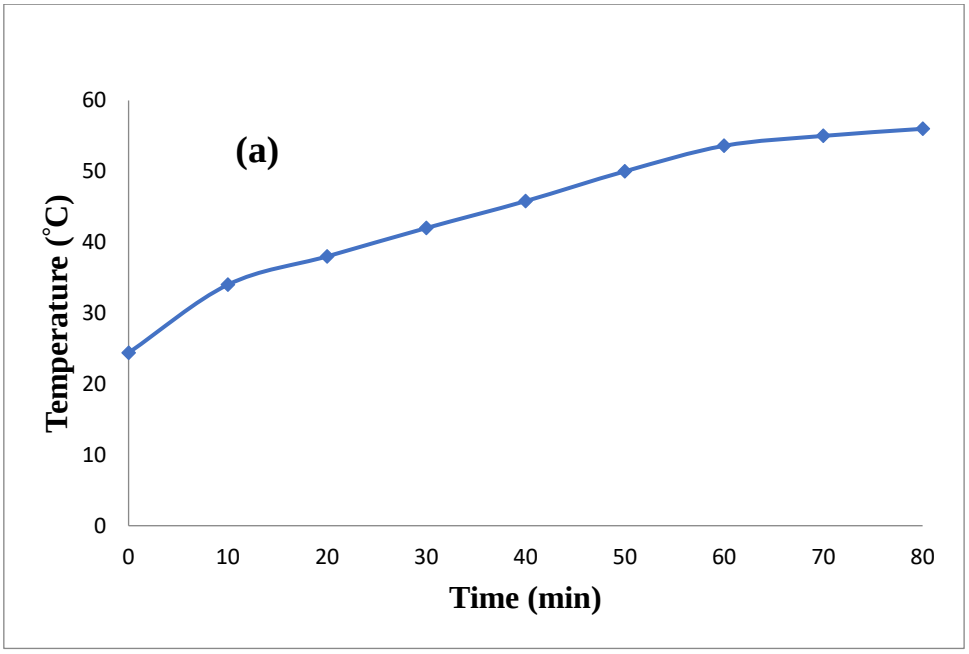
To investigate the performance of the photocatalyst in water splitting and methylene blue (MB) dye degradation simultaneously, methylene blue (MB) dye was added to water.

III.2.2.1. Photo-degradation of Methylene Blue (MB) dye on 3/7 NiO-ZnO/RAC

The obtained results are represented in **Figure 19**, it is noted that as time passes the photo-degradation rate of MB dye rate over 3/7 NiO-ZnO/RAC increases until reaching a complete photo-degradation rate of MB dye (100 %), the photo-degradation is a very fast to degrade 1200 ml solution of 20 ppm of MB dye in 20 min.Results reveal that the NiO-ZnO/RAC act as an excellent in photo-catalyst degradation of MB dye.

NiO-ZnO PC were synthesized by a chemical precipitation method using nickel acetate as precursors and Sodium hydroxide as precipitating agent. The NiO-ZnO PC have been successfully used as a photo-catalyst for degradation of Methylene Blue dye from the aqueous medium. The photo-catalytic degradation study results revealed that the NiO-ZnO PC are potential photo-catalyst in degradation of Methylene Blue[35].

The photocatalyst NiO-ZnO/RAC degrades methylene blue dye through a photocatalytic process that involves the adsorption of the dye molecules onto the surface of the NiO-ZnO/RAC material, followed by the degradation of the dye through a series of redox reactions [43]. The absorption of light energy by the NiO-ZnO/RAC material initiates the process, which excites electrons and creates electron-hole pairs. These electron-hole pairs then participate in redox reactions with the adsorbed dye molecules, leading to their degradation [44].



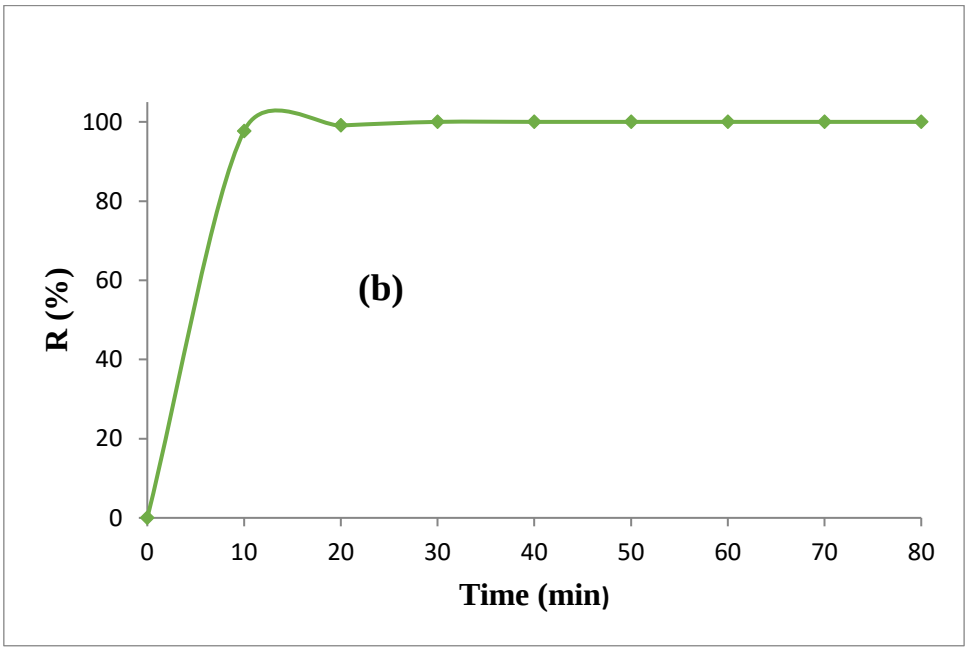


Figure III.5. Photo-degradation of Methylene Blue (MB) dye on 3/7 NiO-ZnO/RAC, variation of (a- Temperature, b-Dye removal)

III.2.2.2. Photo-catalytic water splitting on 3/7 NiO-ZnO/RAC:

A comparison was conducted between the performance of the photocatalyst 3/7 NiO-ZnO/RAC in water-splitting with and without MB dye. The obtained results are represented in **Figure 20** As expected, the addition of MB dye has negatively affected the performance of the 3/7 NiO-ZnO/RAC photocatalyst. It is noted in **Figure 20** (a, b) that there is a decrease in the volume of hydrogen and oxygen production in water splitting with MB dye compared to its productivity without MB dye, which means that the addition of MB dye has a negative effect on the performance of the photo-catalyst of 3/7 NiO-ZnO/RAC, This may be due to the double function of the photo-catalyst in degrading the MB dye and splitting the water simultaneously.

For the decrease in volumes of hydrogen and oxygen produced when Methylene Blue is present, the explanation is that the photo-catalyst has dual functions: degrading Methylene Blue and splitting water. This dual functionality means that some of the photocatalyst's active sites are occupied with degrading the dye, which reduces its availability for the water-splitting reaction. As a result, less hydrogen and oxygen are produced [36].

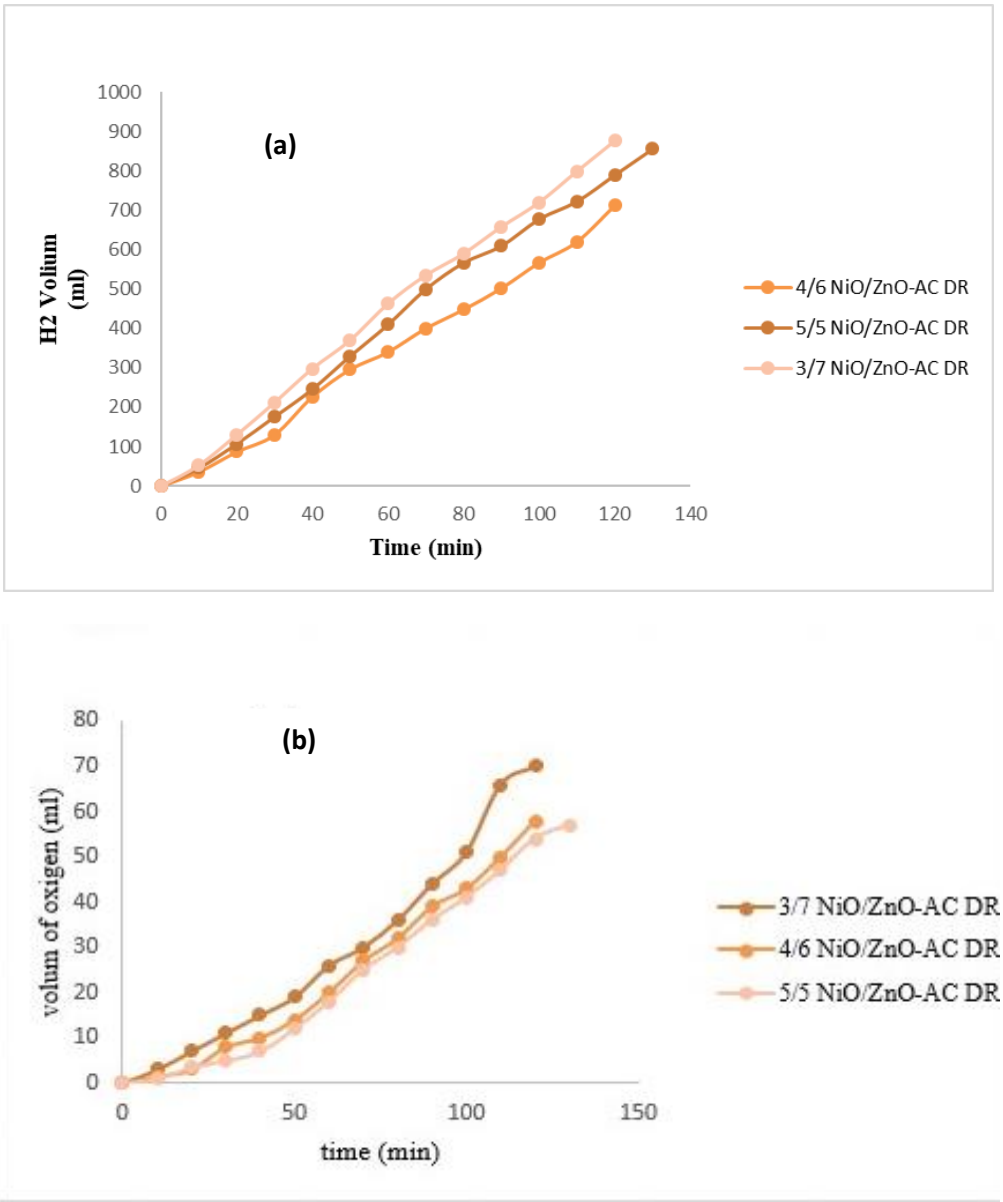


Figure III.6. Photocatalytic activity of 3/7 NiO-ZnO/RAC, variation of (a- Volume of hydrogen produced, and b- Volume of oxygen)

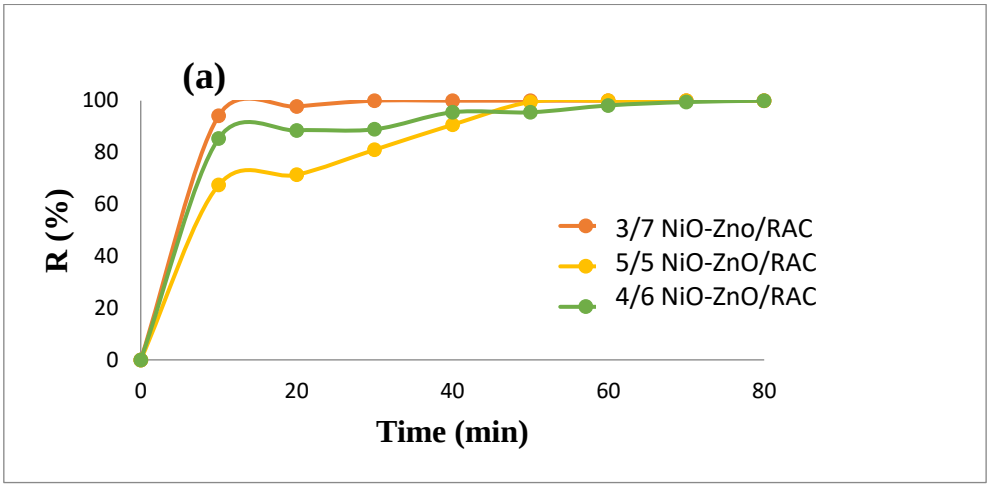
III.3. Comparison of photo-catalytic performance over different weight ratios NiO-ZnO/RAC

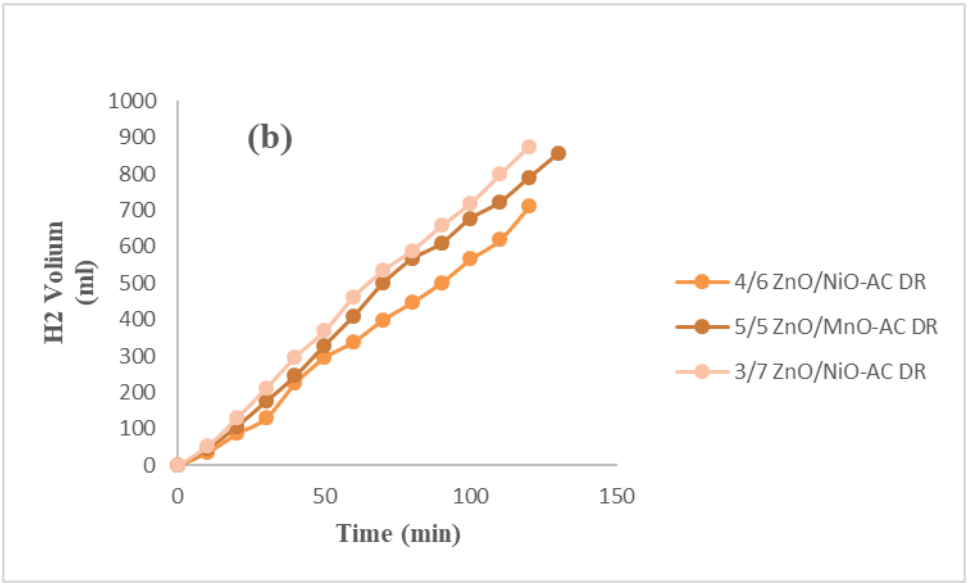
A comparison was conducted between the performance of the photocatalyst 4/6,3/7, and 5/5 NiO-ZnO/RAC in water-splitting with MB dye .The obtained results are represented in **Figure.III.7.**

As seen from **Figure.III.7.(a)** shows the PD rate of MB dye as function of time,it is noted that as time passes in general photo-degradation rate of MB dye increases until reaching a complete photodegradation rate of MB dye (100 %), it is also very important to note that the PC 3/7 NiO-ZnO/RAC shows an excellent performance in degradation of MB dye followed by the PC 4/6 NiO-ZnO/RAC ,at the bottom 3/7 NiO-ZnO/RAC .

The productivity of hydrogen and oxygen through the wastewater splitting over NiO-ZnO/RAC was also presented in **Figure.III.7 (b,c),** it noted that 3/7 NiO-ZnO/RAC offers the highest volume of H2 and O2 it could be considered the optimal ratio.

Increasing the RAC content leads to enhanced photocatalytic activity of hydrogen generation due to the increase of active sites on the surface of the photocatalyst [37].


(a)
R (%)
100
80
60
40
20
0
0
20
40
60
80
Time (min)
3/7 NiO-Zno/RAC
5/5 NiO-ZnO/RAC
4/6 NiO-ZnO/RAC


(b)
H2 Volium (ml)
1000
900
800
700
600
500
400
300
200
100
0
0
50
100
150
Time (min)
4/6 ZnO/NiO-AC DR
5/5 ZnO/MnO-AC DR
3/7 ZnO/NiO-AC DR

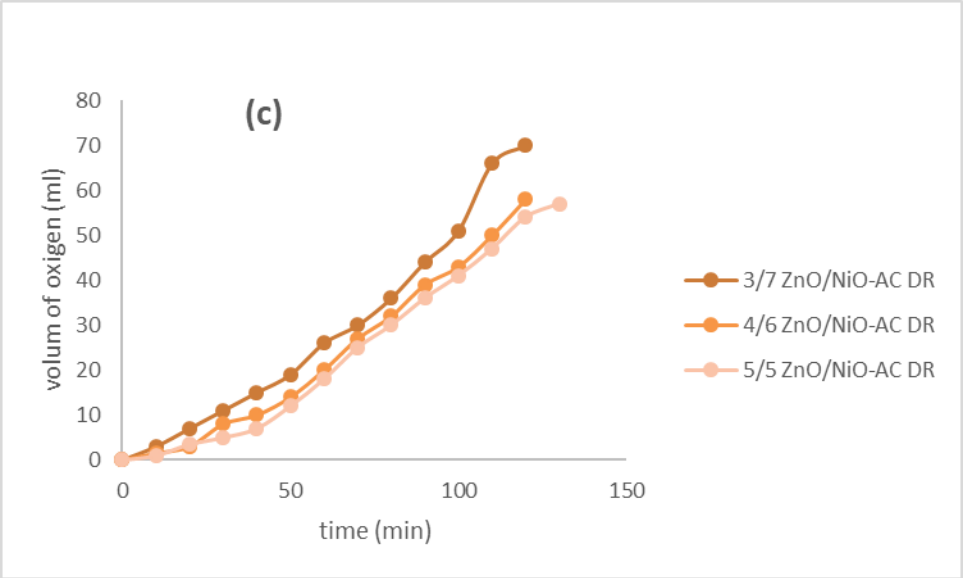


Figure III.7. Photo-catalytic activity of 3/7, 4/6 and 5/5 NiO-ZnO/RAC, variation of (a- dye removal, and b- Volume of hydrogen produced, c-Volume of oxygen produced)

General conclusion

General conclusion

The main objective of this study was achieved through the possibility of regenerating saturated activate carbon and the use of regenerated activated carbon in the photocatalysis process of water decomposition for hydrogen production.

In this thesis, through the experiments that were carried out on granular SAC (mesh 8*3) samples, has been proven that GAC has a high ability and responses to the three main regeneration methods ; thermal, chemical and steam regenerations and the secondes regenerations methods , and can restores its high regeneration capacity without losing the physical and chemical properties.

All results showed that it is very possible to remove the VOCs from carbon and reactivate it more than fresh one, to create a large new areas for adsorption.

In thermal regeneration, according to experimental results which prove that the temperature of 700 °C and residence time of 1 hour is the optimal conditions for regenerate the spent carbon. This degree was as thermal activation condition for the carbon, because after its regeneration the adsorption capacity of iodine rises to the highest value in all experiments from 266.49mg/g (IN of fresh AC) to 1332.49 mg/g with the 8 g sample. As for the process a good results, by other hand has two disadvantages; high consumption of energy for long duration, and high ration of mass lost superior than 82% for the 8 g sample (ash content represents only 1.65%), that mean a 80% of SPC is loosed.

Although thermal regeneration is efficient, but the structure of carbon is destroyed, and with repeated cycles, the carbon will completely disappear.

A photocatalyst based on NiO-ZnO doped with activated carbon regenerated with different ratios has been successfully synthesized in this work, and its performance was evaluated in the process of water splitting into hydrogen and oxygen gas.

The optimal ratio was found to 3/7 NiO-ZnO/RAC for the waste-water splitting, leading to the highest hydrogen production in the water-splitting process, and simultaneously to complete photodegradation of MB dye.

As a general conclusion on this thesis, through experimental work and carbon purification tests, we conclude that the process of chemical purification followed by a temperature of 700 degrees Celsius is a very important process to take into account.Its efficiency was good for its use in photocatalytic water splitting with the two catalysts in the production of hydrogen and can be developed by improving the process conditions by taking into account the recommendations below.

General conclusion

This study demonstrated the successful development of a composite photocatalyst based on regenerated active carbon (RAC) modified with NiO-ZnO .

The NiO-ZnO/RAC composite exhibited enhanced photocatalytic activity for both hydrogen production via water splitting and degradation of the organic pollutant Methylene Blue dye under visible light irradiation.

The RAC provided a large surface area for pollutant adsorption and promoted the separation of photogenerated electron-hole pairs, while the NiO-ZnO enhanced light absorption and facilitated charge transfer processes. The successful of the 3/7 NiO-ZnO/RAC in production of hydrogen ,is composite paves the way for its potential application in sustainable wastewater treatment and solar fuel production.

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عنوان المذكرة: تجديد الكربون النشط (RAC)وتحضيرالمحفز الضوئي (NiO-ZnO)المعدل الفعال لفصل الماء و تحلل صبغة الميثيلين الزرقاء .

اللقب: **غباني** **الإسم: حفيظة** **المؤطر: ج. براهيمي**
اللقب: **حاكمي** **الإسم: بسمة**

ملخص: تم تجديد الكربون النشط المستخدم لامتصاص المركبات العضوية المتطايرة في وحدة التجفيف TEG وإعادة استخدامها في معالجة الغاز اوالبتروكيماويات افضل من رميه. يعد االتجديد الحراري طريقة فعالة في هذه الأطروحة,و الهدف من هذه الدراسة هو تقييم فعالية الطريقة المركبة لتجديد الكربون المنشط باستخدامه لتطعيم أكسيد الزنك (ZnO) و اكسيد النيكل (NiO)بالكربون المنشط المجدد بطريقة الانحلال الحراري لتصنيع المحفز الضوئيNiO-ZnO/RAC. تم تقييم أداء المحفز الضوئي المركب في عملية تقسيم مياه الصرف الصحي. وتم التحقق من كفاءة عملية تقسيم المياه على المحفز الضوئي NiO-ZnO/RACفي ظل معايير تجريبية مختلفة مثل مصدر الضوء، ونسبة الوزن NiO-ZnO/RAC، ووقت التشعيع. تشير النتائج إلى أن النسبة المثلى للمحفز الضوئي هي ZnO-AC 7/3، حيث أن أفضل مصدر للضوء هو 1000 واط. وأظهرت النتائج التي تم الحصول عليها أن المحفز الضوئي NiO-ZnO/RAC 3/7هو مرشح واعد لفصل مياه الصرف الصحي، حيث تكون مياه الصرف الصحي يمكن معالجتها ويتم إنتاج الهيدروجين والأكسجين أيضًا.

الكلمات المفتاحية: الكربون المنشط المستهلك , التجديد ,المحفز الضوئي NiO/ZnO-RAC لفصل الماء، انتاج الهيدروجين

Memory title: Regenerated active carbon (RAC) modified NiO-ZnO photo-catalyst for efficient photocatalytic water splitting and degradation of Methylene Blue Dye.

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Abstract: The activated carbon used to absorb volatile organic compounds in the TEG drying unit has been regenerated and reused in gas processing or petrochemicals better than thrown away. Thermal regeneration is an effective method in this thesis, and the aim of this study is to evaluate the effectiveness of the combined method of activated carbon regeneration using it to inoculate zinc oxide (ZnO) and nickel oxide (NiO)with activated carbon regenerated by pyrolysis method for the manufacture of photocatalyst NiO-ZnO/RAC. The performance of the composite photocatalyst in the process of wastewater splitting was evaluated. The efficiency of the water splitting process on the NiO-ZnO/RAC photocatalyst was verified under various experimental parameters such as the light source, the NiO-ZnO/RAC weight ratio, and the irradiation time. The results indicate that the optimal ratio of the photocatalyst is 3/7NiO-ZnO/AC, since the best light source is 1000 watts. The results obtained showed that the photocatalyst 3/7 NiO-ZnO/racis a promising filter for wastewater separation, in which the wastewater can be treated and hydrogen and oxygen are also produced.

Keywords: Spent Activated Carbon ,Regeneration , water splitting ZnO/NiO-AC photocatalyst,production of hydrogen

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Titre du mémoire : : Photocatalyseur NiO-ZnO modifié au charbon actif régénéré (RAC) pour une division photocatalytique efficace de l'eau et une dégradation du colorant bleu de méthylène.

Nom : GHABANI **Prénom : Hafidha** **Encadreur :** D. Brahimi
Nom :HAKMI **Prénom : Besma**

Résumé : Le charbon actif utilisé pour absorber les composés organiques volatils dans l'unité de séchage TEG a été régénéré et réutilisé dans le traitement du gaz ou la pétrochimie mieux que jeté. La régénération thermique est une méthode efficace dans cette thèse, et le but de cette étude est d'évaluer l'efficacité de la méthode combinée de régénération du charbon actif en l'utilisant pour inoculer de l'oxyde de zinc (ZnO) et de l'oxyde de nickel (NiO)avec du charbon actif régénéré par pyrolyse pour la fabrication de photocatalyst NiO-ZnO/RAC. La performance du photocatalyseur composite dans le processus de fractionnement des eaux usées a été évaluée. L'efficacité du processus de séparation de l'eau sur le photocatalyseur NiO-ZnO/RAC a été vérifiée sous divers paramètres expérimentaux tels que la source lumineuse, le rapport pondéral NIO-ZnO/RAC et le temps d'irradiation. Les résultats indiquent que le rapport optimal du photocatalyseur est de 3/7 NiO-ZnO/RAC, puisque la meilleure source de lumière est de 1000 watts. Les résultats obtenus ont montré que le photocatalyseur 3/7 NiO-ZnO / racest un filtre prometteur pour la séparation des eaux usées, dans lequel les eaux usées peuvent être traitées et de l'hydrogène et de l'oxygène sont également produits.

Mots clés: charbon actif épuisé ;Régénérqtion ,Fractionnement de l'eau, photo-catalyseur NiO-ZnO/RAC ,production d'hydrogen