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Study Of Treated Water in The Sensitive Environment For  
The Adrar Refinery

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بالتدريس والتعليم في التدرج

A. Hanouzi

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## **ABBREVIATIONS**

<b>ERU :</b>	Urban wastewater
<b>ERU :</b>	Eaux Résiduaires Urbaines
<b>RA1D:</b>	Adrar Refinery
<b>RFCC:</b>	Atmospheric Residue Catalytic Cracking
<b>CDU:</b>	Atmospheric Distillation Unit
<b>CRU:</b>	Catalytic Reforming Unit
<b>MES:</b>	Manufacturing Execution System
<b>BOD:</b>	Biochemical Oxygen Demand
<b>COD :</b>	Chemical Oxygen Demand
<b>PH :</b>	Potentiel d'Hydrogène
<b>SS:</b>	Suspended solid
<b>RW:</b>	Raw Water

# **GENERAL INTRODUCTION**

### **Introduction general**

Water is the most essential element for the life and survival of living things on this planet. Although the Earth's surface is 71% water, less than 2.5% of this amount on Earth is freshwater, with much of this amount inaccessible [1].

Water is necessary for human life and it is also necessary in industrial uses, for example in oil factories, where water is used in the process of oil extraction and cleaning because it contains mineral salts and many other unnecessary materials, these salts dissolve in water, which changes its nature and makes it is not usable again as it makes it toxic and cannot be disposed of due to its effect on nature and living organisms. Where two types of water are produced (concentrated water and oily water). This water is treated and made reusable again or disposed of without affecting nature.

Wastewater treatment has become in recent years a major challenge for today's societies; on the one hand to ensure a continuous water distribution service; for the collection and treatment of wastewater. [2].

As well as the supply of drinking water and on the other hand, to safeguard water as a natural heritage. At the same time, an equally important problem has developed: Maintaining the quality of water treated over the years. [2].

The object of our work is to determine the efficiency of the natural elements used in the experiment and to compare it with the industrial method.

This work consists mainly of three chapters, preceded by a general introduction.

In the first chapter, useful information about the Sbaa industrial zone is provided.

The second chapter is divided into two parts;

The first part is about the methods used. Information was presented on the method used in factories to treat wastewater, And the natural method used to treat wastewater in our experience, in which we used natural elements (activated charcoal, sand, sawdust, and filter paper) due to their high adsorption in comparison between it and other natural elements.

The second part is about the equipment used in our experience and how to use it

The third chapter presents the interpretation of the results obtained at the end of the experiments.

Finally, a general conclusion summarizing the main results obtained within the framework of this work and highlights the perspectives opened by our results.

# *CHAPTER I*

**PRESENTATION OF THE REFINERY**

## I.1 Presentation of the ADRAR refinery R1AD

The refinery is located on a total area of 84 hectares of which 37 hectares are occupied by the process (a rectangle with a length of 740m and a width of 500m). It was commissioned in May 2007 with a treatment capacity of (12,500) barrels per day, in another meaning. (600,000) Tons per year of crude oil from fields located in the Sbaa basin, Hassi Latou,

it is managed by China National Petroleum Corporation, a Chinese country-owned public company [3] [4]



Figure I.1 THE ADRAR REFINERY

### *I.1.1 Situation Géographique de la Raffinerie d'Adrar (RA1D)*

The Adrar refinery (RA1D) occupies an area of 75 hectares and is built on a site located 02 km east of the town of SBAA and 44 km north of Adrar near the gas decarbonization unit. [5]

### *I.1.2 Geographical coordinates*

- ❖ Longitude 00 ° 11 West;
- ❖ Latitude 27 ° 49 North;
- ❖ Average altitude is 275 meters.

The refinery site is located in the middle of the Guebi desert, some 900 m from an area Agricultural attached to the village of SBAA, located to the west of the site.

It is Limited:

- ❖ In the North: by the Daïra of Tsabit;
- ❖ In the South: by the commune of Gourara;
- ❖ To the West: by the municipality of Sbaa;
- ❖ To the east: by non-urbanized land;

Apart from this agricultural area, the landscape is a (no man's land), devoid of any vegetation, dwellings or activities [5]



Figure I.2 Adrar RA1D refinery satellite photo



Figure I.11 ground plan of the ADRAR refinery





Figure I.20 Image by google earth of the Refinery

## I.2 Production capacity of the Adrar refinery

### I.2.1 Annual production capacity

The annual processing capacity of the Saba refinery is approximately 600,000 tons of crude oil for a period of 330 days continuously per twenty-four (24) hour a day. [6] [7]

- ❖ 75 t / h
- ❖ •1800 tons / day
- ❖ 12,500 barrels per day

Tableau I-1 The annual processing capacity of deferent products from the R1AD refinery

Products	Quantities (Tons / Year)
Propane	20 500
Butane	32 500
Super gasoline	10 000
Normal gasoline	208 300
Kerosene (Jet A1)	30 000
Diesel fuel	238 400
Oil fuel	13 000

## ***1.2.2 Refinery structure and equipment***

Structure and equipment of the refinery:

The refinery is mainly made up of:

- ✓ A crude atmospheric distillation unit,
  - ✓ A catalytic reforming unit for naphtha,
  - ✓ An atmospheric residue catalytic cracking unit (RFCC),
  - ✓ An LPG separation unit.
  - ✓ A main control rooms.
  - ✓ Storage tanks for petroleum products.
  - ✓ Utility units with its auxiliaries
  - ✓ Compressed air station;
  - ✓ A technical building and laboratory,
  - ✓ A main electrical station,
  - ✓ Electrical subtraction,
  - ✓ A Mechanics and Maintenance workshop,
  - ✓ Eight supply blocks,
  - ✓ An administrative building,
  - ✓ Fire brigade station, and loading ramps,
- [4] [5]

## ***1.2.3 Processing capacity***

The nominal processing capacity of the SBAA refinery is twelve thousand five hundred (12,500) barrels \ day; crude oil from deposits located in the Sbaa basin, Hassi Ilatou.

The refinery is made up of several zones namely: the production unit, a storage, a shipping area; a propane and butane fuel center, an area of technical and administrative building, and a base of life.

The annual treatment capacity of the SABA refinery is approximately 600,000 tons of crude oil for a period of 330 continuous days per day [6] [7]

## **I.3 Main facilities**

These units are distributed as follows:

- 1) Utilities units with their auxiliaries
- 2) 2 A CDU201 atmospheric distillation unit
- 3) A CRU202 naphtha catalytic reforming unit
- 4) An Atmospheric Residue Catalytic Cracking Unit (RFCC203)
- 5) Storage tanks for petroleum products U300

### ***1.3.1The Atmospheric Distillation Unit (CDU)***

This unit treats 600,000 tons / year of crude by atmospheric distillation or products

heavy extracts from the bottom of the column (its capacity limit is 700,000 tons per year while

its minimum capacity is 420,000 tons / year

Tableau I-2 Main specifications of the treated crude

Density	Na Cl content	sulfur content	acidity
0.834 g/l	8 mg/l	‰0.14	0.04 mg KOH/g

Based on these specifications, the processed crude is classified as a low sulfur aromatic crude.

### 1.3.2 The catalytic reforming unit (CRU)

The catalytic reforming unit treats the naphtha feed produced by the atmospheric distillation unit.

The unit's products are essentially high-octane unleaded fuel: NO = 96 commonly referred to as: super unleaded, L.P.G, light naphtha (lightnaphtha), refined naphtha (refinedoil), Main operations of the trial

- Pre-fractionation of the load



Figure I.21 View of the atmospheric distillation unit

- Purification of the charge. [8]



Figure I.28 Catalytic reforming unit

### ***1.3.3 The catalytic cracking unit (RFCC)***

Catalytic cracking is cracking in which large molecules break down using a catalyst.

The catalytic cracking unit is the 3rd production unit of the refinery. She treats the atmospheric residue, its capacity is: 300,000 tonnes per year, it produces gasoline, light diesel, G.P.L (L.P.G), slurry and non-condensable gases [8]



Figure I.33 Catalytic cracking unit

### ***1.3.4 Utilities***

The utilities are made up of the following units

- water treatment unit.



Figure I.40 water supply tray



Figure I.43 Wastewater treatment tank

- steam production unit.



Figure I.48 Economizer



Figure I.55 boiler

- electrical energy production unit.



Figure I.58 Turbo diesel generator



Figure I.61 Turbo generator

- electrical energy distribution unit.



Figure I.64 Electrical Network Control Room

- compressed air production unit.



Figure I.66 View of the atmospheric distillation unit



Figure I.69 Compressed air unit

- cooling water and wastewater operating unit. [6]



Figure I.72 Water cooling tower





Figure I.18 Reverse osmosis filtration

### I.4 Industrial method

#### I.4.1 Pretreatment

The purpose of pretreatment is to separate the coarser materials and the elements likely to interfere with the later stages of treatment. It includes screening to retain bulky waste, grit removal to obtain a better settling, degreasing and de-oiling to avoid the crushing of the station by fatty substances. [9].

##### *I.4.1.1 Screening*

At the entrance to the treatment plant, the raw effluents must undergo a screening, thus, the bulky materials, are retained through the grids, this operation is always carried out in order to protect the pumps, and not to interfere with the operation. subsequent stages of treatment. There are different types of grids: [10] [49].



Figure I.19 Screening

##### *I.4.1.1.1 Manual grids*

They are composed of straight bars of circular or rectangular sections, generally inclined to the horizontal ( $60^\circ$  to  $80^\circ$ ). sometimes movable or pivoting to facilitate the cleaning of the canal. these screens are generally reserved for small purification plants, cleaning is done manually using a rake. [11].

### ***1.4.1.1.2 Mechanical grids***

They are essential from a certain size of wastewater treatment plant, even on small installations in order to reduce manual cleaning interventions.

These grids are operated automatically by an electric clock. [11].

### ***1.4.1.2 sifting***

Sifting is an operation recommended before treatment or discharge into the network on industrial effluents loaded with suspended matter.

Three main functions of sifting:

- recovering usable waste.
- pipe protection or pumps.
- limiting the risk of deposits and fermentation. [11] [17].

### ***1.4.1.3 sand removal***

The elimination of sands present in the raw effluents is an essential operation for:

- avoid deposits in the pipe causing them to clog.
- Protect pumps and other mechanical components against abrasion;
- Avoid disturbing the other stages of treatment, in particular the reactor organic;
- Reduce the production of sludge. [12].

### ***1.4.1.4 degreasing - oil removal***

The degreasing and de-oiling operations consist of a separation of the raw effluent, the oils and the fats being products of density slightly lower than water, the fats, and the oils come not only from homes, but also from restaurants, factories, slaughterhouses ... [10].

#### ***The oils***

Are liquid products; vegetable oils, mineral oils, light hydrocarbons, their separation takes place by absorption or filtration.

Oil removal is a liquid separation operation, usually reserved for the removal of oils present in industrial wastewater (petroleum refineries). [10].

#### ***Fats***

These are solid products of animal or plant origin, they are present in urban wastewater and in certain industrial wastewater, mainly agro-food. [13].

### ***II.4.1.4.1 Oil removal***

The oil removal operation mainly concerns two types of industrial effluents:

- regularly oily effluents (petroleum refining factories, mechanical industries, food oil mills, etc.).
- low oily effluent, but with accidentally too high peak flow rates (thermal power stations, refinery rainwater, etc.). [13].

### ***I.1.1.4.2 Degreasing***

Degreasing can be obtained either in a chamber where the water is tranquilized in a tank with siphoid partitions, or in a circular primary settling tank fitted with air blowing to promote the rise of fat particles and their agglomeration on the surface. [13].

### **I.4.2 Primary treatment:**

#### ***1.4.2.1 Chemical settling processes:***

The physical-chemical settling process consists of making the particles in suspension heavier.

They use coagulation and flocculation techniques to facilitate the removal of suspended solids (MES) and colloids, which is carried out in a subsequent solid-liquid separation step.

The coagulation flocculation of industrial discharges is a technique practiced in a fairly systematic way for the treatment of water containing non-biodegradable products indecantable, it is not a question of the coagulation of the colloids, but of creating an artificial floc which will absorb or encompass non-settling products, such as very fine fibers (stationery) or oil droplets (petroleum refinery).

it can also be considered in very specific cases such as latex suspensions or emulsions of oil, fat, etc.

In this case, the addition of reagents has a dual purpose:

- bring the pH to a value such that the emulsion is broken
- absorb the fine droplets thus formed, in a settling floc (washing wool, oil mills, soap factories, etc.). [14].

#### ***1.4.2.2 Coagulation***

Coagulation takes place in a structure equipped with a central stirrer at high-speed allowing vigorous mixing of the effluents prior to this coagulation phase, pre-neutralization of the effluents (pH correction) was carried out in the homogenization tank by injection of sulfuric acid. [27]

#### ***1.4.2.3 flocculation***

The flocculation is carried out in a second structure equipped with a central agitator at slow speed ensuring a homogeneous mixing of the effluent.

This flocculation was obtained by adding an anionic polymer to the flocculator, which causes the magnification of the micro floc formed by coagulation.

These discharge concentrations are obtained only by simple decantation following this coagulation step. [15]

The following tables give the various coagulation and flocculation agents;

## ***PRESENTATION OF THE REFINERY***

Tableau I-3 Coagulation and flocculation agents

Coagulants		
Product	formula	Commercial form
<b>Alumina sulfate</b>	$Al_2(SO_4)_3 \cdot 14H_2O$	Powder and 48% solution
<b>Aluminum chloride</b>	$AlCl_3 \cdot 6H_2O$	Liquid
<b>Basic Poly Alchloride</b>	$A_N(OH)_m Cl_{3N}$	
<b>Sodium aluminate</b>	$NaAlO_2$	Powder and 50% solution $Al_2O_3$
<b>Ferrous sulphate</b>	$FeSO_4 \cdot 7H_2O$	Crystallized
<b>Ferric chloride</b>	$FeCl_3 \cdot 6H_2O$	40% solution
<b>Lime</b>	$Ca(OH)_2$	powder

Lime is often used in combination with metallic salts, the coagulant doses are determined by laboratory tests on the water to be treated, these tests are called jar-tests. [15]

Tableau I-4 (continued) Coagulation and Flocculation Agents

Flocculant		
Product	Mode of action	use
<b>Cationic polyelectrolyte</b>	<ul style="list-style-type: none"> <li>• charge neutralization</li> <li>• absorption</li> <li>• crosslinking</li> </ul>	Adjuvant coagulant
<b>Anionic polyelectrolyte</b>	<ul style="list-style-type: none"> <li>• absorption</li> <li>• crosslinking</li> </ul>	essentially adjuvant
<b>Non-anionic polyelectrolyte</b>	<ul style="list-style-type: none"> <li>• crosslinking</li> <li>• absorption</li> </ul>	

### ***1.4.2.3.1 Physical settling process:***

Settling is the most common method of separating suspended matter and colloids (collected in the form of a floc after a coagulation –flocculation step).

The basis of its solid-liquid separation processes is gravity, we use the term settling when we want to obtain the clarification of raw water, if we want to obtain a concentrated sludge, we speak of sedimentation.

The purpose of sedimentation is to remove suspended matter from the liquid fraction, effective sedimentation is likely to retain a large fraction of organic sedimentable materials. [14] [15].

### ***I.4.2.3.2 flotation processes:***

As opposed to settling, flotation is a solid-liquid separation process. That applies to particles whose density is less than that of the liquid that contains them.

- if this difference in density is naturally sufficient for a separation, then this flotation is natural.
- flotation is said to be assisted, if it implements external means to improve the separation of naturally buoyant particles.
- flotation is said to be caused when the density of the particle, originally greater than that of the liquid, is artificially it takes advantage of the ability that certain solid particles (or liquids) to unite with bubbles of gas (air in general) to form “gas particle” couplings less dense than the liquid of which they constitute the dispersed phase, the phenomenon implemented is therefore a sort-plastic phenomenon (liquid gas - solid in general) which will depend on three phase physicochemical characteristics and in particular their interfaces[16] [17].

### ***I.4.2.3.3 Precipitation of metals:***

It concerns the removal of metals found mainly in industrial effluents from the treatment of metal surfaces, hydrometallurgical leachate and washing water from combustion gases from household and industrial waste.

The most common process, simply by neutralizing these acidic effluents, precipitates metals in the form of hydroxides.

The maximum precipitation pH of all metals not coinciding;

We are forced to seek an optimum zone of the reaction pH which can vary from 7 to 10.5 depending on the minimum values sought for the elimination of the most harmful metals.

Sometimes, several successive precipitations are carried out at staggered pH. For the selective recovery of different metals.

### **I.4.3 Absorption treatment:**

Absorption is one of the means of treatment available to the water processor to remove non-degradable organic matter dissolved biologically.

The basic phenomenon involved is a mass transfer from the liquid or gas phase to the surface of the absorbent material, to which the organic compound tends to bind.

The absorption capacity depends on:

- of the developed surface or specific surface of the material, industrial absorbents (mainly activated carbon).
- Development of specific surfaces from 1000 to 1500 m<sup>2</sup> / g characteristic of a high microporosity.
- of the concentration of the organic substance in solution, at the limit, a balance is established between the concentration of the solution and the mass of pollutant absorbed per unit mass of the absorbent.
- the hydrodynamic characteristics of the exchange and in particular the contact time of the solid-liquid phases;

- the binding energy, i.e., the affinity of the surface of the absorbent for the organic substance, therefore the nature of the products to be absorbed. This affinity is often even a function of the Ph. [18] [5].

### ***1.4.3.1 Main absorbents:***

Absorbents developed on an industrial scale are diverse in nature, including aluminas, synthetic absorbent resins that are neutral or contain exchangeable radicals, such as ions of the same polarity, and especially the most widely used activated charcoals by far.

These are carbonaceous materials of mineral (coke, anthracite, coal) or organic (peat, wood, coconut, etc.) nature prepared by carbonization and chemical activation to increase their microporosity and thus develop their specific surface area which can reach 1500 to 2000 m<sup>2</sup>/g.

Activated charcoal is available in two forms: powdered and granulated charcoals. [16][3].

#### ***1.4.3.1.1 Powdered coal:***

It is in the form of particles (particle size 10 to 50 µm) and is introduced continuously into the water with the conditioning reagents (coagulation-flocculation), it is inserted into the "flocs" and is extracted with them at the level of the clarification generally carried out by decantation.

Less expensive than grain coal, it has the advantage of allowing an overdose in the event of peak pollution, its major drawback is that it is practically impossible to regenerate ... we therefore work with wasted coal, which essentially reduces its use for discontinuous or small dosages (less than 20 g /m<sup>3</sup>). [18] [5].

#### ***1.4.3.1.2 Coal grains:***

Its particle size is between 0.5 to 2 mm, it is used in the form of a filter bed (notch height 2 to 3 m) crossed by the water to be treated, the pollutants of which are thus subjected to a methodical extraction by the mass of carbon which will gradually become saturated depending on the displacement of the adsorption front in the filtration column. [18][5].

### ***1.4.3.2 Ion exchange:***

Ion exchangers consist of resins having active groups capable of exchanging mobile ions with other ions of the same sign, by passing through mobile resins, the polluting cations and anions are fixed and replaced respectively by ions H<sup>+</sup> and OH<sup>-</sup>.

Depending on the nature of the pollutants, four groups of ion exchangers are used:

- weakly acidic cation exchanger.
- weakly acidic cation exchanger.
- weak / moderately basic anion exchanger.
- strongly basic anion exchanger.

The acidic and basic waters are channeled in different circuits.

The cyanide and alkaline water are recycled after passing through:

- on a strongly acidic cation exchanger which fixes all the free cations (Na<sup>+</sup>, Ca<sup>+2</sup>) but not the heavy metals involved in stable cyanide complexes.

## ***PRESENTATION OF THE REFINERY***

---

- then on a moderately basic anion exchanger which retains the cyanide complexes ( $\text{Cu}(\text{CN})^{3-4}$ ,  $\text{Zn}(\text{CN})^{2-4}$ ..).
- at the end on a strongly basic anion exchanger which retains all the other anions ( $\text{CN}^-$ ,  $\text{C}(\text{O}_3)^{2-}$  ...)

The polluting anions are trapped on the exchangers, and the purified water is reused (as rinsing water for example).

The resins end up being saturated.

They can be shipped for treatment or regenerated on site by elution, that is, stripped of fixed elements and returned to their original form.

The cation exchangers are treated with a strong acid, the acid eluates contain the metals in solution.

The anion exchangers are treated with soda, the alkaline eluates contain anions including chromates or cyanides as appropriate. [21] [50] [51]

### **I.4.4 Filtration:**

Filtration is a separation process that uses the passage of a solid / liquid mixture through a porous medium (filter) which retains solid particles and allows liquid to pass (filtra).

There are two main categories of filtration process depending on the mode of implementation:

- filtration on support,
- granular bed filtration, [19].

#### ***1.4.4.1 filtration on support***

We distinguish:

- ✚ Screening, i.e. surface filtration up to relatively large opening thresholds (above around 30 microns)
- ✚ the accurate filtration, in which the filtration on thin support of the largest particles coexists with the thickness filtration of the finest.

One aspect of surface filtration should be emphasized: it does not ensure the elimination of its initial state by efficient and economical washing, the mode of which is linked to the type of filter and the nature of the elements retained. [19] [20].

Depending on the application, different materials can be used:

# PRESENTATION OF THE REFINERY

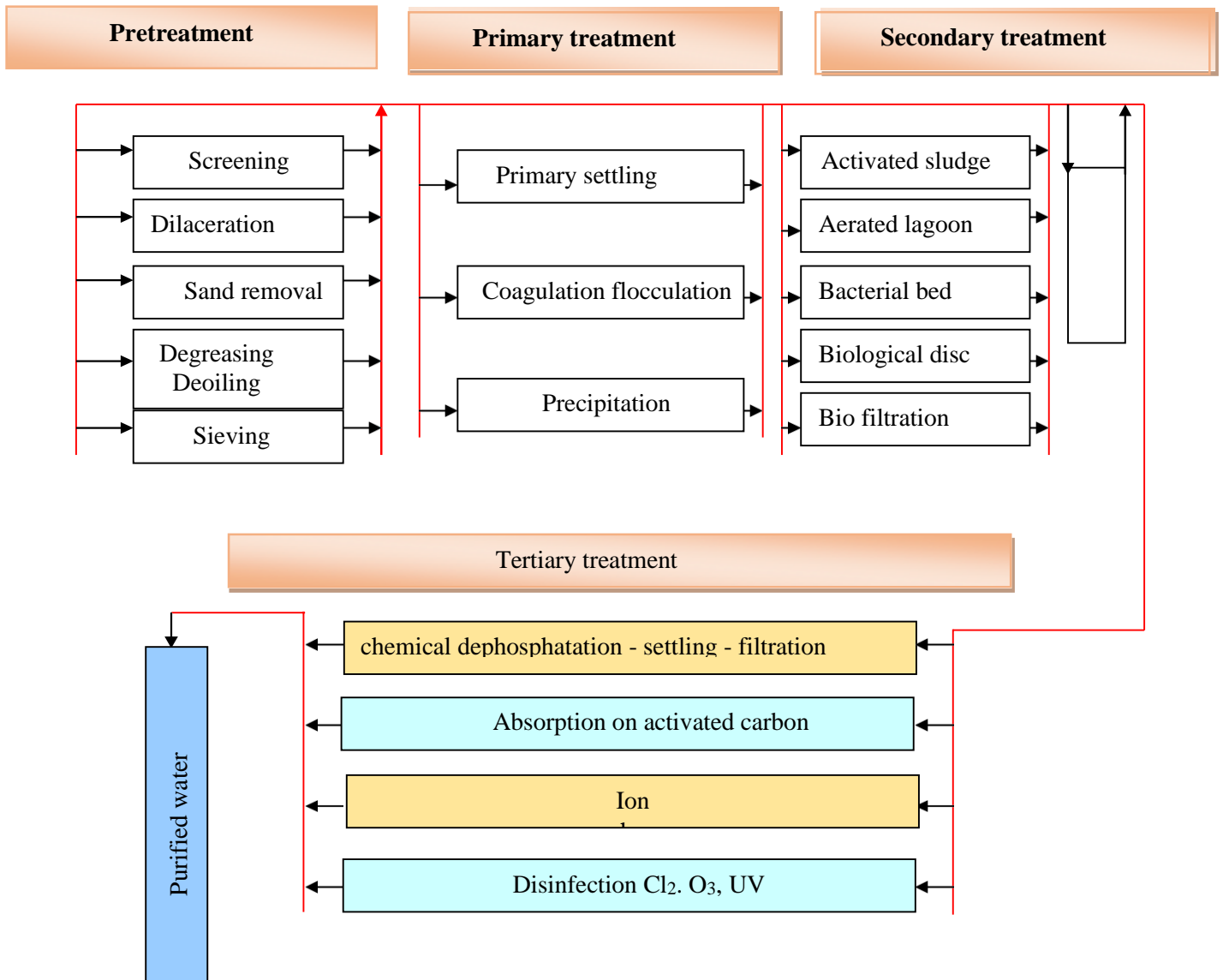


Figure I.20 Industrial water treatment chain or channels.



# **CHAPTER II**

## **Methods and Materials**

### **II.1 The Method Used in This Study**

The method used in this study is the treatment of wastewater naturally, so it uses activated charcoal, filter paper, sand, and sawdust, according to its characteristics in a high absorption rate, and then compares between the incoming and outgoing water for natural treatment, then it is compared with the water treated by the industrial method and determines Efficiency ratio of natural treatment compared to industrial.

#### ***II.1.1 Definition of Adsorption***

The adsorption process is one of the most widely used methods for removing pollutants. In addition, adsorption is a separation process by which certain components of a phase (liquid, gas) are attached to the surface of a solid adsorbent by chemical or physical bonds. It is based on specific interactions (thermodynamics and kinetics) [28].

#### ***II.1.2 Types of Adsorption***

According to the binding energies involved, the adsorption is divided into two types:

##### ***II.1.2.1 Chemical Adsorption***

is an irreversible phenomenon, due to a strong chemical bond of the covalent type between the surface atoms of the solid and the adsorbed molecules, this type of adsorption involves high attraction energies, which lead to heat of adsorption high, often approaching the energies of covalent chemical bonds of the order of 20 to 80 Kcal / mol. [31]

##### ***II.1.2.2 Physical Adsorption***

Unlike chemorption (Chemical Adsorption), physical adsorption occurs at low temperatures and is characterized by low adsorption energy (the adsorbed molecule is therefore not strongly bound); it results from the forces of Van Der Waals. The adsorbed molecule is attached to a specific site and can move freely at the interface. Physisorption is rapid, reversible and does not cause any modification of the adsorbed molecules. It is of the order of 2 to 10 Kcal / mol. [28]

#### ***II.1.3 The Adsorption Mechanism***

The adsorption process is one of the transfers of matter that takes place between the fluid called adsorbate and the solid surface called the adsorbent. It mainly occurs in several stages. During the adsorption of a species to a solid, the transfer of material takes place from the fluid phase to the active sites of the adsorbent through the following steps:

Diffusion of the adsorbate from the external liquid phase to that located near the surface of the adsorbent.

- Extra-granular diffusion of the material (transfer of the solute through the liquid film towards the surface of the grains).
- Intra-granular transfer of matter (transfer of matter in the porous structure of the outer surface of the grains to the active sites).

Dye adsorption interaction on contact with active sites. Once adsorbed, the molecule is considered immobile. [33]

#### ***II.1.4 The Most Important Elements Affecting Adsorption***

There are many parameters and properties that can affect adsorption, the main ones being:

### ***II.1.4.1 Mass Effect***

The influence of the mass of the adsorbent has been reported for many systems. The amount of adsorbate attached to the surface of the adsorbent is closely related to the mass and particle size of the adsorbent. The finer the particle size distribution, the more the specific surface area increases and the more the adsorbed quantity increases. [34]

### ***II.1.4.2 PH Effect***

The pH is an important factor in any chemical study such as the absorption kinetics study because it can affect both the structure of the adsorbents and on the adsorption mechanism. [31]

### ***II.1.4.3 Temperature Effect***

The adsorption processes are not complicated by chemical reactions always accompanied by the release of heat (exothermic), so that with an increase in temperature, the phenomenon of desorption becomes dominant. On the other hand, for activated adsorption (sorption chemistry), the adsorption equilibrium is not reached quickly, and the increase in temperature promotes adsorption. [36]

### ***II.1.4.4 Effect of Concentration***

The difference in adsorbate concentrations definitely affects the adsorption process in addition to several other conditions where there is a concentration compatible with certain conditions. [32]

### ***II.1.5 Types of Adsorbent***

There are many types of adsorbents because the adsorption processes are used for many other applications than the treatment of volatile organic compounds, whether in the gas phase or in the liquid phase: separation of oxygen and water, Nitrogen from the air, purification of hydrogen, desulphurization of natural gas, decolorating of sweet juices, purification of effluents, separation of molecules for the pharmacy, water treatment, through our experience, we study the properties of the elements used. [37]

#### ***II.2.1.1 Activated charcoal:***



Figure II.2 Coal grains

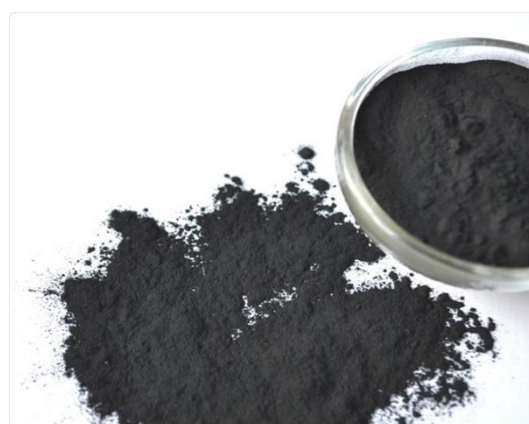


Figure II.1 Powdered coal

Activated carbons are by far the most widely manufactured and used adsorbents in industry. They have various applications, particularly in the fields of water and air purification. The adsorption power of activated carbons is attributed to porosity, specific surface area, and surface functional groups. They are carbon products with a porous structure with a very large

internal contact surface. The characteristics of activated carbons vary depending on the precursors (starting material) and the method of activation. They can be presented in three different forms: grains, powder, or fiber. [27]

### II.1.5.2 Sand



Figure II.4 coarse sand



Figure II.3 fine sand

Sand filtration is one of the most popular physical processes in water treatment. This purification method consists in passing the water to be treated through a sand which, in view of its filtering properties, is able to retain a large number of solid particles. A fine biological layer (bio-film) forms on the surface of the sand, which is responsible for removing most of the microorganisms that exist in the water. A sand filter requires continuous cleaning to protect satisfactory and acceptable processing capabilities. [38]

There are two categories of sand filtration:

#### ***Slow filtration (slow sand filters):***

Slow filtration is a biological purification method which involves passing the water to be treated through a bed of filter material (sand) at a relatively low speed of 2 to 6 m / day. Its main goal is the elimination of pathogenic organisms contained in raw water, so we will produce bacteriologically pure water [48] [18].

#### **❖ The advantages of the slow filter:**

- No mechanical element (simple process they can be found on site);
- Can be used in developing countries;
- Does not require the addition of chemicals;
- Produces high quality water (elimination of suspended solids; degradation of OM and good elimination of bacteria) [43].

#### **❖ The disadvantages of the slow filter:**

- Requires large areas and large amounts of filter media (sand);
- Highly charged water can quickly clog these filters;
- Limitation in cold countries [43].

#### ***Fast filtration (fast sand filters):***

Rapid filtration is a physical (use of pump) and sometimes chemical (use of chemicals) process which involves filtering water through different layers of sand in order to remove the

solids suspended in the water at a very high speed. high from 4 to 25 m / h; These sand filters can be gravity or pressurized [44].

❖ **The advantages of the rapid filter:**

- It has the advantage of not promoting the appearance of algae;
- Does not require large surfaces;
- It has the advantage of being able to treat water laden with colloids [45].

❖ **The disadvantages of the quick filter:**

- The biological action is weak (low elimination of bacteria);
- It is difficult to clean and replace filter media (sand, gravel) due to the shape of the filter;
- Consumes a lot of energy and water [45].

### II.1.5.3 Sawdust



Figure II.6 coarse Sawdust



Figure II.5 fine Sawdust

It powdery particles of wood produced by sawing.

A researcher from the National Research Center in Egypt has found that waste from furniture making workshops, known as "sawdust", is used to purify water in a way that turns it into harmful waste for the environment. environment as a means of combating pollution.

This discovery is part of a study conducted by Amal Abdel Aziz, a researcher in the Preparations and equipment for cellulosic fibers division of the textile industries division, on "the conversion of certain waste into materials used to eliminate certain industrial dyes which pollute the environment", because industrial wastewater contains large quantities of them.

In special statements to the correspondent of the Anadolu Agency, the researcher said that the idea of using "sawdust" in water purification is due to its high content of "cellulose", which has a role in the absorption of chemical dyes.

The researcher explained that the mechanism of use is based on the transformation of sawdust into small granules by crushing them, placing them in the drainage path of factories, and absorb industrial dyes.

The study tested the effectiveness of "sawdust" in absorbing three industrial dyes, namely red, green and blue acid. The results showed the ability of "sawdust" to absorb at rates of 86, 70, and 82 mg/gram for the three dyes, respectively [46].

### II.1.5.4 Filter Paper

It's a paper intended to purify liquid and atmospheric substances. It is made from a derivative of cellulose, which is a substance present in the membrane of plant cells, depending on the use that the paper will have, its texture and diameter will be different. Its components make it a sufficiently resistant paper to allow the passage of an insoluble solution and insulation, which in this case would be impurities. Therefore, it is a semi-permeable paper, in addition to being able to withstand any kind of material, it can also withstand high and low temperature, this kind of paper is commonly used in analysis labs for the purpose of distilling liquid materials and obtaining a clean substance separated from the contaminated residue, however, this is not the only filter paper present. The design of this paper differs depending on what needs to be filtered, in that you can find filter papers for professional use such as those used in the chemistry lab, and for personal use [47].



Figure II.7 filter paper

#### *Types of filter paper*

As mentioned earlier, the different proportions, sizes, and roughness of filter paper differ, and this difference is created by the need. There is a type of filter for every use. In some cases, the choice of which filter paper to use must be made based on the components that will be analyzed and the components that make up the filter paper. In other cases, it will depend on the device or mechanism that needs filter paper to function properly.

Here are the most popular types of filter paper:

- Qualitative filter paper

This type of filter paper is used for a qualitative analysis, which serves to identify the particles present in the filtered solution. It is manufactured with 100% cellulose and is designed to withstand a PH from 0 to 12. It is also able to withstand temperatures up to 120 ° C. There are 13 different grades of qualitative filter paper. The largest is Grade 4 and the smallest is Grade 602 hours. Also, there are different diameters [47].

- Quantitative filter paper

This type of filter paper is made of 100% cellulose cotton and contains 0.01% ash. It is used to perform gravity analysis and monitor the environment. The goal is that by separating the present materials, a quantitative analysis can be done. Therefore, the characteristic that this filter paper stands out with is not that its components possess the materials to be analyzed. Like qualitative filter paper, quantitative filter paper also has different grades and diameters [47].

- Chromatographic filter paper

This filter paper is used by chemists to separate the components, a technique known as electrophoresis. It is also functional to conduct qualitative and quantitative analysis. They are characterized by the presence of a certain rate of water flow and fast absorption, which provides accurate analysis [47].

### II.2 Method of Analysis

This method of work is taken from the experimental protocol of the Adrar refinery [53].

#### II.2.1 filtration

Filtration is a process used to separate solids from liquids or gases using a filter medium that allows the fluid to pass through but not the solid. The term "filtration" applies whether the filter is mechanical, biological, or physical. The fluid that passes through the filter is called the filtrate. The filter medium may be a surface filter, which is a solid that traps solid particles, or a depth filter, which is a bed of material that traps the solid.

Filtration is typically an imperfect process. Some fluid remains on the feed side of the filter or embedded in the filter media and some small solid particulates find their way through the filter. As a chemistry and engineering technique, there is always some lost product, whether it's the liquid or solid being collected [52].



Figure II.8 filtration method

### *filtration methods*

There are different types of filtrations. Which method is used depends largely on whether the solid is a particulate (suspended) or dissolved in the fluid. [52]

#### *General Filtration:*

The most basic form of filtration is using gravity to filter a mixture. The mixture is poured from above onto a filter medium (e.g., filter paper) and gravity pulls the liquid down. The solid is left on the filter, while the liquid flows below it [52].

### *II.3.2 parameter analysis*

This method of work is taken from the experimental protocol of the Adrar refinery

#### *II.2.2.1 PH*

One of the fundamental characteristics of water is pH (hydrogen potential). The pH gives an indication of the acidity of a substance. It is determined from the amount of hydrogen hydronium ( $H^+$ ) or hydroxide ( $OH^-$ ) ions in the substance. When the amounts of these two ions are equal, the water (or the substance) is considered neutral, and the pH has a value around 7. The pH of a substance varies between 1 and 14. Above 7, the substance is considered basic and the amount of  $OH^-$  ions is greater than that of  $H^+$  ions. Below 7, the substance is acidic; the  $H^+$  ions are in higher quantities. The pH value is to be taken into consideration during the majority of water treatment operations, especially when these involve a chemical reaction and because some processes need to be carried out with a specific pH to be effective.

One of the important chemical parameters when determining the quality of water is PH. It is used to control the quality of the water entering the treatment plant (significant variations in pH are almost always the result of industrial discharges).

#### *Principle*

The method is based on the use of a pH meter. The pH meter is a somewhat peculiar voltmeter that is characterized by a very high input impedance due to the high resistance presented by the measuring electrode.

#### *Equipment*

The pH measuring equipment consists of:

- A WTW 521 pH meter equipped with a combination electrode;
- An integrated thermometer ;
- A magnetic stirrer.

#### *Operating mode*

##### **Preparation of instrumentation**

- Check the various connections: mains, electrodes, etc.;
- Release the electrode from its support;
- Remove the protective cap from the double electrode, put it in a safe place;
- If necessary, top up the electrolyte level, rinse the end of the electrode thoroughly with distilled water;
- Wipe the tip of the electrode with JOSEPH paper;



- Replace the electrode on its support.

### *Calibration*

#### ❖ 1<sup>st</sup> step

Approximately measure the pH of the sample using indicator paper.

#### ❖ 2<sup>nd</sup> step

- Place a small magnetic bar in the perfectly clean measuring vessel;
- Rinse the vessel and the bar with very clean distilled water and then with a first buffer whose pH is lower than that of the solution to be studied;
- Place the buffer in the vase;
- Immerse the electrode making sure:
  - that the frit of the reference electrode is immersed,
  - that the magnetic bar can turn freely.
- Switch on the device, wait a few minutes (2 - 5 minutes);
- Measure the temperature of the buffer and display it on the temperature corrector, a certain pH value is displayed;
- Bring this value to that of the buffer by activating the standardization potentiometer (left button) and wait for the reading to be stable;
- Remove the electrode, rinse it and wipe it dry;
- Return the tampon to its original bottle;
- Rinse the measuring vessel and the electrode with distilled water and then with a second buffer whose pH is higher than that of the sample.

#### ❖ 3<sup>rd</sup> step

- Place the second buffer in the vase;
- Act as if above for the temperature, immerse the electrode, a new pH value is displayed;
- Bring this value to that corresponding to the pH of the second buffer using the slope adjustment potentiometer;
- Wait until the reading is stable;
- Return the second buffer to its vial and rinse the electrode.
- The device is then calibrated and ready for use.

### *PH measurement*

- Rinse the vessel, the magnetic bar, the electrode, with distilled water and then with the sample;
- Fill the measuring vessel with the sample;
- Correct the temperature;
- Immerse the electrode with the usual precautions and shake;
- Read the pH directly when the value has stabilized.

### *work to be done*

For each of the samples offered:

- Approximately evaluate the pH using pH paper,
- Select the buffers best suited to the calibration of the device,
- Standardize and adjust the slope,
- Measure the pH of the sample.

### II.2.2.2(MES) (SS)

Suspended solids (SS) constitute all of the minerals and/or organic particles present in natural or polluted water. They can be composed of particles of sand, earth, and sediment torn up by erosion, various debris brought in by wastewater, or rainwater very rich in SS, living planktonic beings (especially algae). They correspond to the concentration of undissolved elements in a sample.

The abundance of suspended matter in the water promotes reduction in light and lowers biological production, in particular, due to a drop in dissolved oxygen following a reduction in photosynthesis. We used filter syringe to calculate suspended matter



Figure II.9 filter syringe

#### *Principle*

SS is obtained either by filtration of low-load effluents or by centrifugation of the solutions, drying until a dry residue is obtained. For the monitoring of the CREPA station, the determination of SS will be done by filtration through a glass fiber filter, taking into account the domestic origin of the effluents. The measurement of suspended solids by filtration is based on the principle of double weighing: a volume of water is filtered through a 1.5-micron membrane (previously weighed under vacuum) and the residues on the latter are weighed. The ratio of the difference in mass to the volume of filtered water gives the concentration of suspended solids in milligrams / liter.

#### *Apparatus and Glassware*

- Vacuum filtration equipment ;
- Whatman GF / C glass microfiber filters ( $\varnothing$  47mm);
- Volumetric flasks or graduated cylinders;

#### *Reagents*

This determination protocol does not use reagents.

#### *Operating mode*

- Take a GFC membrane and mark it carefully so as not to damage it;
- Weigh the membrane and note its empty mass  $M_0$ ;

- Place the membrane on the filtration manifold;
- Shake the sample well;
- Take a volume of the sample and transfer it to the membrane;
- Proceed with filtration: the filtered volume must not exceed 1 liter and filtration must not last more than half an hour.
- Recover the membrane after filtration, then place it in an oven at 105 ° C for 1 hour 30 minutes to remove excess water;
- Weigh the membrane again, after drying, then note its mass  $M_1$ .

### ***Expression of results***

The ratio of the difference in masses to the filtered volume gives the concentration of suspended solids in the sample. We apply the following formula:

$$C_{MES} = \frac{M_1 - M_0}{V} \quad (\text{EQ 1})$$

$C_{MES}$ : concentration of suspended solids in mg / l;

$M_0$ : mass of the membrane before filtration;

$M_1$ : mass of the membrane after filtration;

$V$ : volume of filtered sample.

### ***II.2.2.3 COD***

The degradation of organic matter (carbohydrates, proteins, amino acids, lipids, and other reserve substances) discharged into rivers leads to the consumption of oxygen dissolved in the water. This is done to the detriment of living organisms and can thus lead to the suffocation of the environment. Pollution by organic matter is caused by industrial discharges (chemical, petroleum, agro-food industries, etc.) and discharges from urban populations. The extent of this pollution in the effluent can be assessed by the chemical oxygen demand (COD).

The COD makes it possible to assess the concentration of organic or mineral matter, dissolved or suspended in water, through the quantity of oxygen necessary for their total chemical oxidation. Thus, by measuring the COD, we can assess the polluting load of wastewater in organic matter before and after a physical, chemical, or biological treatment in order to control the functioning of a WWTP and the activity of microorganisms.

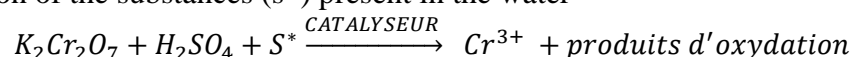
### ***Principle***

Under well-defined operating conditions, certain materials contained in the water are oxidized by potassium dichromate in an acidic medium and in the presence of catalysts. A masking agent helps prevent possible interference from chlorides.

The excess dichromate introduced is determined by a reducing agent, ferrous sulfate, so we can go back to the amount of dichromate consumed by the oxidizable materials. An appropriate indicator can be used to detect the end of the dosage.

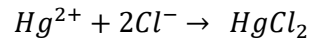
The reactions can be schematized as follows:

- ❖ Oxidation of the substances ( $s^*$ ) present in the water

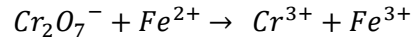


- ❖ Intervention of a masking agent

To prevent the oxidation of chloride ions to chlorine, mercury (II) sulfate is used which complexes the Cl<sup>-</sup> ions.:



Dosing reaction



### *Products and materials*

#### ❖ Products

- Concentrated sulfuric acid d = 1,83 g/l (dangerous);
- Dilute sulfuric acid 4 M;
- Solution of silver sulphate in concentrated sulfuric acid, (dangerous);
- Ammonium iron sulfate solution approximately 0,12 M;
- Mercury sulphate crystals;
- Potassium dichromate solution 0,04 M;
- Ferroin solution.

#### ❖ materials

- Reflux device;
- 250 ml flasks;
- 5 ml volumetric pipette;
- 25 ml burette;
- 10 ml and 20 ml test tubes;
- Heating ramp;
- Magnetic agitator.

### *Operating mode*

It is necessary, before carrying out the determination of the COD itself, to check the titer of the ferrous solution and to carry out a blank test. Checking the titer of the ferrous solution Given the extreme oxidability of Fe<sup>2+</sup>, this check must be done every day.

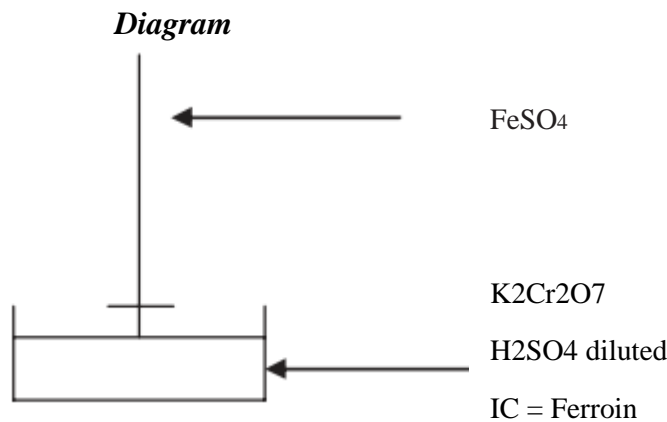


Figure II.10 Ferrous solution dosing device

❖ **Protocol**

- Place 10 ml of dichromate solution in a beaker using a pipette;
- Make up to about 100 ml with the dilute sulfuric acid solution;
- Add with the iron solution until the mixture turns a purplish red color. Let  $V_1$  be the volume of ferrous solution used.

❖ **Expression of results**

During the redox reaction, the following changes in the degrees of oxidation are observed:



So: A molar solution of  $Cr_2O_7^-$  corresponds to 6 N and a molar solution of  $Fe^{++}$  corresponds to 1N.

Soit :  $N_1 =$  normality of  $Fe^{++}$

$V_1 =$  volume of iron solution used

$N_2 =$  normality of the dichromate solution, i.e.  $0.04 \times 6 = 0.24 \text{ N}$

$V_2 =$  dichromate volume

On a:  $N_1 * V_1 = N_2 * V_2$  (Eq.2)

$$N_1 = \frac{N_2 * V_2}{V_1} \quad \text{(Eq.3)}$$

$$N_1 = \frac{0.24 * 10}{V_1} = \frac{2.4}{V_1} \quad \text{(Eq.4)}$$

**Blank test**

Its purpose is to assess the consumption of dichromate by reducing agents which may be in the mixture and which originate from a lack of purity of the reagents and the use of questionable glassware.

Carry out this test in parallel with the COD determination, but replacing the test portion with 10 ml of distilled water.

The volume of ferrous solution used to obtain the change in color will be called  $V_B$ .

**Determination of COD**

**Preparation of the sample**

Homogenize the sample if necessary and introduce in order, in a 250 ml flask:

- 10 ml of sample using the cylinder; rinse the test tube with a jet of distilled water, transfer the washing water to the flask;
- A few glass beads or equivalent;
- A pinch of mercuric sulfate, about 0.4 g;
- 5 ml of dichromate with a pipette;
- 15 ml of concentrated sulfuric acid (dangerous), using a test tube; proceed with caution and gently shake the vase in a circular motion.
- It is desirable to place the balloon on a bed of ice during the entire operation in order to prevent the release of heat from causing the disappearance of volatiles. (Optionally, the flask can be cooled under running water).
- Connect the refrigerant to the balloon and supply it with tap water;

- Bring to the boil under reflux for 2 h; boiling should be regular, without jerks or excess;
- Let the ball cool;
- Drag into the bottom of the balloon, by a jet of a squeeze bottle, the deposits which have formed on the internal wall;
- Remove the flask from the heater and cooler;
- Make up to about 75 ml with distilled water and allow to cool to room temperature.

**Dosage**

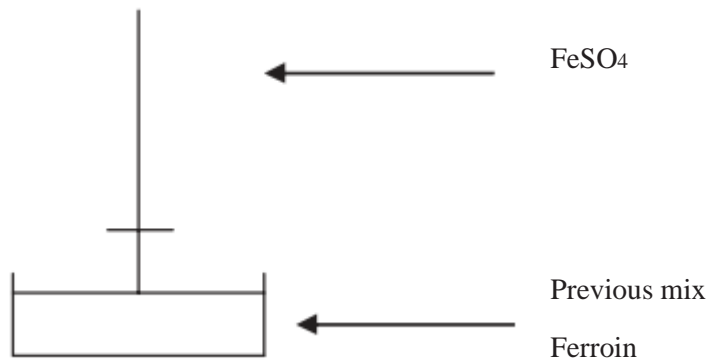


Figure II.11 COD dosing device

**Protocol**

- Transfer the contents of the flask into a 250 Erlenmeyer flask;
- Rinse the flask with the minimum amount of distilled water and add the washings to the mixture;
- Place a few drops of ferrouin in the Erlenmeyer flask;
- Titrate with the ferrous solution until the blue-green color changes to red-brown.
- Let  $V_e$  be the volume of ferrous solution used.

**Expression of results**

The COD expressed in mg / l is given by the formula:

$$DCO \text{ mg/l} = \frac{800N_1(V_B - V_e)}{V_0} \quad (\text{Eq.5})$$

With:

$V_B$  = volume of ferrous solution used for the blank test

$V_e$  = volume of ferrous solution used for the sample

$V_0$  = volume of the test sample

$N_1$  = normality of the ferrous solution

**Application domain**

The method is applicable to waters with a COD between 30 and 700 mg / l. The chloride concentration should not exceed 2000 mg / l. Dilution of the sample is required if these maximum limits are reached.

**Tips for handling**

Upon arrival at the laboratory, the heating ramp will be energized at 2/3 of its power. It is recommended to prepare in duplicate:

- Each blank test;
- Each pure sample;
- Each sample diluted if dilution seems necessary (see monitor).

- The ferrous solution will be checked during boiling. Under no circumstances should the heating ramp be left unattended!

### **II.2.2.4 BOD**

#### ***Object***

Biochemical oxygen demand (BOD) is an expression to indicate the amount of oxygen that is used for the destruction of decomposable organic material by biochemical processes. The determination of the BOD is used to evaluate the concentration of organic pollutants in the inputs and outputs of biological treatment plants, that is to say, to measure the yield.

The measurement of BOD5 is made according to the manometric method based on the principle of the WARBURG respirometer in which the respiration of the biomass is directly measured by a device. A volume of sample is placed in ground-top vials.

#### ***Principle of the manometric method***

A quantity of water is poured into a 300 ml incubation bottle, connected to a mercury manometer or closed with a cap fitted with a pressure sensor (Oxytop). The chosen volume depends on the desired range of measurements. The measuring device, type IS 602, is placed in a refrigerator maintained at 20 ° C. We then monitor, as a function of time, ie every day for 5 days for BOD5, the oxygen consumption, which results in a decrease in air pressure. Finally, the measurement is corrected by a corrective factor which depends on the quantity of sample taken and the desired measurement range.

Oxidation of organic matter causes the formation of CO<sub>2</sub> which will be trapped by a KOH solution. Thus, a depression develops in the bottle.

The addition of 1 allyl 2 thiourea: C<sub>4</sub>H<sub>8</sub>N<sub>2</sub>S inhibits nitrification because the oxidation of ammoniacal derivatives and nitrites to nitrates also absorbs oxygen. This amine acts as an inhibitor. To be introduced for the measurement of outlet water.

#### ***Operating mode***

- Measure the desired quantity (see table below) with the calibrated overflow flask and pour into the clean bottle;
- Introduce the magnetic stirrer into each bottle;
- Add a pinch of allyl thiourea;
- Put 2 potassium hydroxide tablets in each inner cap (black) with two tweezers;
- Screw on without hermetically closing the cap;
- Put on the stirring system at 20 ° C;
- Allow to establish equilibrium for 30 minutes and close the cap tightly;
- Read the values after 5 days (Oxytop system);
- Use measurements from other groups and determine the accuracy of the measurements.

It is recommended to perform the double of each dosage (depending on the availability of measuring equipment).

#### ***Parameters influencing the measurement***

##### ***Quantity to analyze***

The biochemical oxygen demand for analysis depends on a load of organic substances. The BOD5 measurement can be estimated at around 80% of the COD

Tableau II-1 BOD5 conversion factor as a function of the intake volume

Measuring range	Amount	Postman
0 – 40	432 ml	1
0 – 80	365 ml	2
0 – 200	250 ml	5
0 – 400	164 ml	10
0 – 800	97 ml	20
0 – 2000	43.5 ml	50
0 – 4000	22.7 ml	100

The actual value is calculated as follows:

$$\text{DBO}_5 \text{ (mgO}_2\text{/l)} = \text{Read value} * \text{factor}$$

Measure the quantities to do the analysis with the overflow balloon and to take the exact quantities.

***Quantity to measure***

- Raw waters 164 ml;
- Settled water 250 ml;
- Purified water 432 ml.

***pH value***

The most favorable pH values for biological processes are between 6.5 and 7.5

***Temperature***

The sample must be introduced into the chamber at exactly 20 ° C.

***II.3.2.5 Dilution method***

Appropriate samples of the wastewater to be examined are prepared with mains water (water stabilized at 20 ° C for 48 hours). The change in dissolved oxygen is measured at time 0 and time 5 days. The best results are obtained for a variation of 35 to 60%.

***Operating mode***

- Make the appropriate solutions with pure water;
- Put a pinch of allyl thiourea in the bottle to avoid nitrification (water out);
- Fill the vial to the brim with the dilution;
- Put the volume expander on the bottle;
- Introduce the probe with the stirring system;
- Place the flask with the probe on the magnetic stirrer;
- Stir until the pO2 value stabilizes;
- Note this value;

After 5 days in the dark and at 20 ° C, measure the concentration of dissolved oxygen.



### *Sample volume*

The volume of the test sample is a function of the COD value. For urban water, the following formula is used: Maximum test portion = 4000 / COD (mgO<sub>2</sub> / l) Two different test portions are carried out: one max and one mini (half of the max intake)

### *Calculation of BOD*

The following relationship is used to calculate the BOD value in mgO<sub>2</sub> / l:

$$DBO_5 = ((P_0 - P_5) - (K_0 - K_5)) * V / E \quad (\text{Eq. 6})$$

With: P<sub>0</sub>: concentration of O<sub>2</sub> in the dilution at the start of the test;

P<sub>5</sub>: O<sub>2</sub> concentration in the dilution at the end of the test (after 5 days);

K<sub>0</sub>: O<sub>2</sub> concentration in the dilution water at the start of the test

K<sub>5</sub>: O<sub>2</sub> concentration in the dilution water at the end of the test (after 5 days);

V : volume du flacon

E : test sample

Note: Both tests can be done with the same maximum dilution. The test with water is not necessary if the O<sub>2</sub> consumption is less than 0.2 mg / l

# ***CHAPTER III***

**Results and discussion**

This chapter presents the results obtained through the studied methods (filtration, suspended matter, chemical oxygen demand, and biological oxygen demand) and provides explanations with discussion to compare industrially treated water and naturally treated water and to know the quality of water before and after use.

### III.1 Results of Analysis

#### III.1.1 Filtration

In this method, used natural elements (activated charcoal, sand, sawdust, and filter paper) the following results were obtained;



Figure III.1 before and after filtering concentrated water and oily water

Tableau III-1 Filtration results

	Before (ml)	filtering	After filtering (ml)	Filtered (ml)
<b>Oily water</b>	600		350	250
<b>concentrated water</b>	400		297.5	102.5

Notice that many changes occur after filtering,

which are:

- Color Change;
- The change in volume indicates the filtering of many substances in the water;
- Column consumption;
- Mass loss.

**III.1.2 pH**

In this parameter the following results were obtained;

Tableau III-2 pH results

	Before filtering	After filtering
<b>Oily water</b>	9.54	7.05
<b>concentrated water</b>	10.54	7.94

Note from **Tableau III-2** the change in the value of pH in both concentrated and oily water, and this is due to the effectiveness of the column in absorbing salts.

Note from the obtained results that the pH values of wastewater before filtration differ in an interval between 9.54 and 10.54, while these values differ after filtration of wastewater within a very narrow time period, between 7.05 and 7.94. These results satisfy the required pH value for the vacuum criteria. The latter must be between: 5.5 and 8.

**III.1.3 SS**

At this parameter, was measured the weight of the empty filter syringe and was measured the weight before and after filtering both the concentrated and oily water, and then calculated the percentage of suspended substances.

Tableau III-3 The weight of the empty filter syringe

<b>filter syringe (g)</b>	<b>2.6185</b>	<b>2.6104</b>	<b>2.6187</b>	<b>2.6104</b>
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Tableau III-4 Weight of the before and after filtering the concentrated and oily water

	Before filtering (g)	After filtering (g)
<b>Oily water</b>	2.6457	2.6350
<b>concentrated water</b>	2.6326	2.6191

Tableau III-5 Weight of the ss

	Before filtering (g)	After filtering (g)
<b>Oily water</b>	0.0272	0.0246
<b>concentrated water</b>	0.0139	0.0087

Tableau III-6 SS

	Before filtering (mg/l)	After filtering (mg/l)
<b>Oily water</b>	1.030	0.734
<b>concentrated water</b>	0.227	0.087

**Tableau III-3** shows the different weights of the filter syringe ,**Tableau III-4** shows the different weights of the filter syringe with the suspensions.

**Tableau III-5** show us the difference between the weight of the empty and full filtration syringe is shown, which is the weight of the suspended materials.

**Tableau III-6** shows the values of the suspended materials in the concentrated and oily water before and by the number of filtrations.

Note from the obtained results that the values of suspended matter for samples taken before and after filtration of both oily and concentrated water ranged between 2.6457 and 2.6191.

Thus, Note that the natural elements have great effectiveness in reducing suspended materials.

### **III.1.4 COD**

At this parameter, measured the amount of chemical contamination in both concentrated and oily water before and after filtration.

Tableau III-7 Results of cod

	Before filtering (mg/l)	After filtering (mg/l)
<b>Oily water</b>	3456	2752
<b>concentrated water</b>	1248	896

Note from the **Tableau III-7** that there is a lot of chemical pollution in the oily water, and this is due to the absence of organic substances in the water its decreases after that in filtered water.

Also note the presence of chemical pollution in concentrated water and its decrease after that in filtered water, and this indicates the absorption of elements for chemical pollution and its effectiveness in reducing the degree of pollution.

### **III.2 The industry results**

This part will put some results from the Adrar refinery, and then compare with the natural treatment

**Tableau III-8 The industry result of january**

	pH	SS (MES) (mg/l)	COD (mg/l)	BOD (mg/l)
<b>Oily water (Before treatment)</b>	8.08	152.7143	538.17	90.66667
<b>Oily water (after treatment)</b>	7.32	86.16667	200.97	73.71429
<b>concentrated water (Before treatment)</b>	8.38	46.66	86.52	53
<b>concentrated water (after treatment)</b>	7.74	0	65.7	46.55

Tableau III-9 The industry result of february

	pH	SS (MES) (mg/l)	COD (mg/l)	BOD (mg/l)
<b>Oily water (Before treatment)</b>	8.46	150	0	108
<b>Oily water (after treatment)</b>	7.39	86.16	0	0
<b>concentrated water (Before treatment)</b>	8.52	85	97.6	46.55
<b>concentrated water (after treatment)</b>	7.39	72	57.6	0

Tableau III-10 The industry result of march

	pH	SS (MES) (mg/l)	COD (mg/l)	BOD (mg/l)
<b>Oily water (Before treatment)</b>	8.46	195	5729	0
<b>Oily water (after treatment)</b>	7.39	90	70.0	0
<b>concentrated water (Before treatment)</b>	7.60	70	188	0
<b>concentrated water (after treatment)</b>	7.80	65	40.3	0

Tableau III-11 The industry result of april

	pH	SS (MES) (mg/l)	COD (mg/l)	BOD (mg/l)
<b>Oily water (Before treatment)</b>	7.53	1421	148	282
<b>Oily water (after treatment)</b>	8.43	53	50.9	19
<b>concentrated water (Before treatment)</b>	7.76	189	4198	0
<b>concentrated water (after treatment)</b>	7.70	49	89.9	0

Note from **Tableau III-8, III-9, III-10** and **III-11** the results obtained in the Adrar refinery that the pH values of the samples taken at the entrance and after treatment ranged between 7.32 and 8.52.

Note from **Tableau III-8, III-9, III-10** and **III-11** the results obtained in the Adrar refinery that the SS values of the samples taken at the entrance and after treatment ranged between 1421 and 49.

Note from **Tableau III-8, III-9, III-10** and **III-11** the results obtained in the Adrar refinery that the COD values of the samples taken at the entrance and after treatment ranged between 5729 and 40.

However, these values drop significantly at the exit of the plant, and thus industrial treatment is more effective in treating the wastewater.

### **CONCLUSION**

The purpose of this was to study the effectiveness of natural elements (activated charcoal, sand, sawdust, and filter paper) in treating wastewater in petroleum factories due to its high absorption rate in comparison between it and other natural materials and to compare the results obtained with the results of industrial treatment and determine its effectiveness.

Hence, concluded that industrial treatment is more effective, but the natural treatment remains acceptable and applicable because it gave satisfactory results. Compete with the results of the industrial treatment. Only the problem remains in the bacterial contamination that cannot be eliminated through natural treatment.



***GENERAL  
CONCLUSION***

## ***GENERAL CONCLUSION***

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According to some recent projections, by 2025 two-thirds of the world's population will experience a scarcity of freshwater; moderate to severe. While about half will face severe and real supply difficulties.

Almost all renewable freshwater, such as rivers, lakes, or groundwater, known as “conventional resources”, has already been exploited.

Algeria is a country severely affected by decreases in natural water resources, which is why we have thought of reclaiming purified water through its reuse instead of rejecting it in natural environments.

The purpose of this thesis was to study the effectiveness of natural elements (activated charcoal, sand, sawdust, and filter paper) in treating wastewater in petroleum factories due to its high absorption rate in compared between it and other natural materials and to compare the results obtained with the results of industrial treatment and determine its effectiveness.

We can say that this study is applicable in water treatment plants, but it should overlap with the industrial method because it is very slow in treatment and takes a long time to affect the wastewater and its treatment, and because its effectiveness in biological treatment is weak in compared between it and the chemical one, so the industrial method must be introduced with this treatment But in terms of chemical pollution, it is very good in reducing the proportion of chemical pollution, and in terms of suspended matter, it is excellent, in addition to absorbing colors, it is very effective.

In conclusion, we draw attention to the fact that the reuse of purified wastewater from the Adrar refinery is a first experience on the national territory, we will have to maintain and rehabilitate it by adding a natural treatment to the industrial treatment to ensuring the continuity of the good return of pure water, in terms of pollution standards. And in doing so, we help protect the environment and conserve increasingly scarce primary resources.

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# **Annex**





People's Democratic Republic of Algeria  
Ministry of Higher Education and Scientific Research  
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# Study of treated water in the sensitive environment for the Adrar refinery

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**Abstract**— The organization of discharges varies according to the nature, age and size of the refineries, but it is recommended to use a provision (and increasingly observed) of separation of effluents into at least three circuits:

- “contaminated” rainwater;
- Process water;
- deballasting water.

This segregation becomes essential if we seek to recycle as well as possible, after treatment, the non-saline effluents to the refrigeration and fire circuits, to the process, or even to LP or MP - HP boilers.

Our objective in this thesis is to study the case of the Adrar refinery to treat and recycle industrial effluents. We will seek to optimize the treatment of wastewater to promote its reuse.

**Keywords**—Adrar refinery, recycle, treat, water, wastewater

## INTRODUCTION

Water is necessary for human life and it is also necessary in industrial uses, for example in oil factories, where water is used in the process of oil extraction and cleaning because it contains mineral salts and many other unnecessary materials, these salts dissolve in water, which changes its nature and makes it is not usable again as it makes it toxic and cannot be disposed of due to its effect on nature and living organisms. Where two types of water are produced (concentrated water and oily water). Here comes our role in treating and filtering it for reuse or disposal without affecting nature.

## OUR METHOD

Our study is to treat wastewater in a natural way, so we use charcoal, filter paper, sand and sawdust, depending on their characteristics in the high absorption rate, after which we compare the water entering and leaving the natural treatment process, then we compare it with the water treated by the industrial method and determine the efficiency ratio of the natural treatment compared to the industrial one.

## Definition of Adsorption

The adsorption process is one of the most widely used methods for removing pollutants. In addition, adsorption is a separation process by which certain components of a phase (liquid, gas) are attached to the surface of a solid adsorbent by chemical or physical bonds. It is based on specific interactions (thermodynamics and kinetics) [1].

## Types of Adsorption

Be According to the binding energies involved, the adsorption is divided into two types:

### Chemical Adsorption

is an irreversible phenomenon, due to a strong chemical bond of the covalent type between the surface atoms of the solid and the adsorbed molecules, this type of adsorption involves high attraction energies, which lead to heat of adsorption high, often approaching the energies of covalent chemical bonds of the order of 20 to 80 Kcal / mol. [2]

### Physical Adsorption

Unlike chemorption (Chemical Adsorption), physical adsorption occurs at low temperatures and is characterized by low adsorption energy (the adsorbed molecule is therefore not strongly bound); it results from the forces of Van Der Waals. The adsorbed molecule is attached to a specific site and can move freely at the interface. Physisorption is rapid, reversible and does not cause any modification of the adsorbed molecules. It is of the order of 2 to 10 Kcal / mol. [3]

## The Adsorption Mechanism

The adsorption process is one of the transfers of matter that takes place between the fluid called adsorbate and the solid surface called the adsorbent. It mainly occurs in several stages. During the adsorption of a species to a solid, the transfer of material takes place from the fluid phase to the active sites of the adsorbent through the following steps:

- Extra-granular diffusion of the material (transfer of the solute through the liquid film towards the surface of the grains).

- Intra-granular transfer of matter (transfer of matter in the porous structure of the outer surface of the grains to the active sites).

Dye adsorption interaction on contact with active sites. Once adsorbed, the molecule is considered immobile. [4]

#### THE MOST IMPORTANT ELEMENTS AFFECTING ADSORPTION

There are many parameters and properties that can affect adsorption, the main ones being:

##### *Mass Effect*

The influence of the mass of the adsorbent has been reported for many systems. The amount of adsorbate attached to the surface of the adsorbent is closely related to the mass and particle size of the adsorbent. The finer the particle size distribution, the more the specific surface area increases and the more the adsorbed quantity increases. [5]

##### *PH Effect*

The pH is an important factor in any chemical study such as the absorption kinetics study because it can affect both the structure of the adsorbents and on the adsorption mechanism. [6]

##### *Temperature Effect*

The adsorption processes are not complicated by chemical reactions always accompanied by the release of heat (exothermic), so that with an increase in temperature, the phenomenon of desorption becomes dominant. On the other hand, for activated adsorption (sorption chemistry), the adsorption equilibrium is not reached quickly, and the increase in temperature promotes adsorption. [7]

##### *Effect of Concentration*

The difference in adsorbate concentrations definitely affects the adsorption process in addition to several other conditions where there is a concentration compatible with certain conditions. [7]

#### TYPES OF ADSORBANT

There are many types of adsorbents because the adsorption processes are used for many other applications than the treatment of volatile organic compounds, whether in the gas phase or in the liquid phase: separation of oxygen and water, nitrogen from the air, purification of hydrogen, desulphurization of natural gas, decolorating of sweet juices, purification of effluents, separation of molecules for the pharmacy, water treatment, through our experience, we study the properties of the elements used. [7]

#### *Activated charcoal*

Activated carbons are by far the most widely manufactured and used adsorbents in industry. They have various applications, particularly in the fields of water and air purification. The adsorption power of activated carbons is attributed to porosity, specific surface area, and surface functional groups. They are carbon products with a porous structure with a very large internal contact surface. The characteristics of activated carbons vary depending on the precursors (starting material) and the method of activation. They can be presented in three different forms: grains, powder, or fiber. [19]

#### *Sand*

Sand filtration is one of the most popular physical processes in water treatment. This purification method consists in passing the water to be treated through a sand which, in view of its filtering properties, is able to retain a large number of solid particles. A fine biological layer (bio-film) forms on the surface of the sand, which is responsible for removing most of the microorganisms that exist in the water. A sand filter requires continuous cleaning to protect satisfactory and acceptable processing capabilities. [8]

There are two categories of sand filtration:

##### *Slow filtration (slow sand filters):*

Slow filtration is a biological purification method which involves passing the water to be treated through a bed of filter material (sand) at a relatively low speed of 2 to 6 m / day. Its main goal is the elimination of pathogenic organisms contained in raw water, so we will produce bacteriologically pure water [21] [22].

##### *Fast filtration (fast sand filters):*

Rapid filtration is a physical (use of pump) and sometimes chemical (use of chemicals) process which involves filtering water through different layers of sand in order to remove the solids suspended in the water at a very high speed. high from 4 to 25 m / h; These sand filters can be gravity or pressurized [10].

#### *Sawdust*

It powdery particles of wood produced by sawing.

A researcher from the National Research Center in Egypt has found that waste from furniture making workshops, known as "sawdust", is used to purify water in a way that turns it into harmful waste for the environment. environment as a means of combating pollution.

This discovery is part of a study conducted by Amal Abdel Aziz, a researcher in the Preparations and equipment for cellulosic fibers division of the textile industries division, on "the conversion of certain waste into materials used to eliminate certain industrial dyes which pollute the environment",

because industrial wastewater contains large quantities of them.

In special statements to the correspondent of the Anadolu Agency, the researcher said that the idea of using "sawdust" in water purification is due to its high content of "cellulose", which has a role in the absorption of chemical dyes.

The researcher explained that the mechanism of use is based on the transformation of sawdust into small granules by crushing them, placing them in the drainage path of factories, and absorb industrial dyes.

The study tested the effectiveness of "sawdust" in absorbing three industrial dyes, namely red, green and blue acid. The results showed the ability of "sawdust" to absorb at rates of 86, 70, and 82 mg/gram for the three dyes, respectively [18].

#### Filter Paper

It's a paper intended to purify liquid and atmospheric substances. It is made from a derivative of cellulose, which is a substance present in the membrane of plant cells, depending on the use that the paper will have, its texture and diameter will be different. Its components make it a sufficiently resistant paper to allow the passage of an insoluble solution and insulation, which in this case would be impurities. Therefore, it is a semi-permeable paper, in addition to being able to withstand any kind of material, it can also withstand high and low temperature, this kind of paper is commonly used in analysis labs for the purpose of distilling liquid materials and obtaining a clean substance separated from the contaminated residue, however, this is not the only filter paper present. The design of this paper differs depending on what needs to be filtered, in that you can find filter papers for professional use such as those used in the chemistry lab, and for personal use [20].

#### Types of filter paper

As mentioned earlier, the different proportions, sizes, and roughness of filter paper differ, and this difference is created by the need. There is a type of filter for every use. In some cases, the choice of which filter paper to use must be made based on the components that will be analyzed and the components that make up the filter paper. In other cases, it will depend on the device or mechanism that needs filter paper to function properly.

Here are the most popular types of filter paper:

- Qualitative filter paper

This type of filter paper is used for a qualitative analysis, which serves to identify the particles present in the filtered solution. It is manufactured with 100% cellulose and is designed to withstand a PH from 0 to 12. It is also able to withstand temperatures up to 120 ° C. There are 13 different grades of qualitative filter paper. The largest is Grade 4 and the smallest is Grade 602 hours. Also, there are different diameters [20].

- Quantitative filter paper

This type of filter paper is made of 100% cellulose cotton and contains 0.01% ash. It is used to perform gravity analysis and monitor the environment. The goal is that by separating the present materials, a quantitative analysis can be done. Therefore, the characteristic that this filter paper stands out with is not that its components possess the materials to be analyzed. Like qualitative filter paper, quantitative filter paper also has different grades and diameters [20].

- Chromatographic filter paper

This filter paper is used by chemists to separate the components, a technique known as electrophoresis. It is also functional to conduct qualitative and quantitative analysis. They are characterized by the presence of a certain rate of water flow and fast absorption, which provides accurate analysis [20].

#### RESULT

	Oil content	CO D	BOD 5	SS	PH
production waste water upgrade pool exit	3.51	67.8	23	52	8.61
sedimentation	10.2	1039	125	117	7.51
Clean water basin for reclamation water	4.86	59.4	19	66	7.59
Oil separating pool exit low	28.9	2120	150	101	7.79
Oil separating pool exit high	18.1	1272	133	321	7.76

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## Abstracts

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The organization of discharges varies according to the nature, age and size of the refineries, but it is recommended to use a provision (and increasingly observed) of separation of effluents into at least three circuits:

- “contaminated” rainwater;
- Process water ;
- deballasting water.

This segregation becomes essential if we seek to recycle as well as possible, after treatment, the non-saline effluents to the refrigeration and fire circuits, to the process, or even to LP or MP - HP boilers.

Our objective in this thesis is to study the case of the Adrar refinery to treat and recycle industrial effluents. We will seek to optimize the treatment of wastewater to promote its reuse.

**Keywords:** Adrar refinery, recycle, treat, water

يختلف تنظيم مياه الصرف وفقاً لطبيعة المصافي وعمرها وحجمها، ولكن يوصى (بشكل متزايد وملاحظ) بفصل النفايات السائلة إلى ثلاث أصناف على الأقل:

- مياه الأمطار "الملوثة".
- المياه المعالجة؛
- المياه الفاسدة.

يصبح فصل النفايات ضرورياً إذا سعينا إلى إعادة التدوير، بعد معالجة النفايات السائلة غير المالحة إلى دوائر التبريد والحريق، أو في العملية، أو حتى إلى غلايات LP أو HP - MP

هدفنا في هذه الأطروحة هو دراسة حالة مصفاة أدرار لمعالجة وإعادة تدوير النفايات الصناعية السائلة. سنسعى إلى تحسين معالجة المياه العادمة لتعزيز إعادة استخدامها.

**كلمات مفتاحية:** مصفاة أدرار، إعادة تدوير، معالجة، مياه

## **Le Résumé**

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L'organisation des rejets est variable suivant la nature, l'âge et la taille des raffineries mais elle est recommandée d'utiliser une disposition (et de plus en plus fréquemment observée) de séparation des effluents en trois circuits au moins :

- Eaux pluviales « contaminées » ;
- Eaux de procédés ;
- Eaux de déballastage.

Cette ségrégation devient indispensable si l'on cherche à recycler au mieux, après traitement, les effluents non salins vers les circuits de réfrigération, d'incendie, vers le process, voire vers les chaudières BP ou MP – HP.

Notre objectif dans ce mémoire est d'étudier le cas de la raffinerie d'Adrar pour traiter et recycler les effluents industriels. Nous allons chercher à optimiser le traitement des eaux usées pour favoriser leur réutilisation.

**Mots clés :** raffinerie d'Adrar, recycler, traiter, eau