

Chapter Two

The Experimental Part

2.1 Introduction:

In this work the effect of semiconductor (Fe_2O_3) and the exposure time of UV light (365nm) on the photodegradation of chloroform (CHCl_3) in waste water were studied. This chapter presents the materials, the equipments, tools and setup that were used in addition to the experimental procedure.

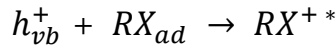
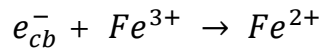
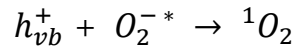
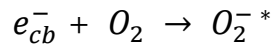
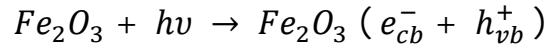
2.2 The materials:

The materials used in this work were:

2.2.1 Iron oxide (Fe_2O_3):

Iron oxides are among the most chemically reactive components of suspended matter in aquatic systems and can be easily prepared in laboratory conditions (A. Helena, Y. Valentin, 2015). Additionally, they are considered non-toxic and environmentally friendly compounds, similarly to free iron ions (A. Helena, Y. Valentin, 2015). Most of them reveal semiconductor properties and then may also act as photocatalysts, even though their overall efficacy can be impaired by a very efficient hole–electron recombination (A. Helena, Y. Valentin, 2015). Possible semiconducting mechanisms involved in iron oxide can be summarized as follows. In the first step, a photon with energy equal to or greater than the material's band gap, which separates the conductance band (CB) and valence band (VB), is absorbed by a semiconducting particle of iron oxide. This gives rise to the generation of the electron/hole pair (A. Helena, Y. Valentin, 2015). Although the excited electron/hole pair can recombine and release the energy as heat, some of the excited electrons and holes can contribute to redox reactions on the surface of a semiconducting particle of iron oxide (A. Helena, Y. Valentin, 2015). The most relevant redox processes,

which take place after the photo-generation of electrons (e^-_{cb}) and holes (h^+_{vb}) in semiconducting particles of iron oxide suspended in aqueous medium, containing also an organic substance (RX), are summarized in next equations (A. Helena, Y. Valentin, 2015).



2.2.1.1 Physical properties:

Ferric oxide has semiconducting properties which make it an attractive material to be used as a photoactive catalyst. Fe_2O_3 is widely used for air purification, deodorization, sterilization and anti-fouling. Activity of Fe_2O_3 depends on its surface area, porosity and acid-basic properties. The pristine Fe_2O_3 is only active upon ultraviolet light, its band gap 3.2 eV which is large enough to make a photodegradation of an organic substance when illuminated with UV Light Emitting Diode (LED) (J. Gupta, 2007).

2.2.1.2 Chemical properties:

Iron(III) oxide or ferric oxide is the inorganic compound with the formula Fe_2O_3 . As the mineral Known as hematite, Fe_2O_3 is the main source of iron for

the steel industry. Fe_2O_3 is ferromagnetic, dark red, and readily attacked by acids. Fe_2O_3 insoluble in water (www.ox.ac.uk, 2015).

2.2.2. Chloroform (CHCl_3):

2.2.2.1 Physical properties:

Chloroform is a colorless liquid that is not very soluble in water and is very volatile. The chemical formula for chloroform is CHCl_3 and it has a molecular weight of 119.38g/mol (www.ox.ac.uk, 2015).

2.2.2.2 Chemical properties:

Chemical names are Chloroform, Trichloromethane, Trichloroform, Formyl thrichloride, Methenyl thrichloride and Methane thrichloride. Chloroform is a commonly used laboratory solvent. The primary use of chloroform today is in industry, where it is used as a solvent and in the production of the refrigerant Freon (www.ox.ac.uk, 2015).

2.2.2.3 Overview:

Chloroform is released into the environment as a result of its manufacture and use; its formation in the chlorination of drinking water, municipal and industrial waste water, and swimming pool and spa water; and from other water treatment processes involving chlorination. Most of the chloroform released into the environment will eventually enter the atmosphere. In the atmosphere, chloroform may be transported long distances before ultimately being degraded by indirect photochemical reactions with such free radicals as hydroxyl (www.ox.ac.uk, 2015).

The general population is exposed to chloroform by ingesting water and food, inhaling contaminated air, and possibly through dermal contact with chloroform-containing water (www.ox.ac.uk, 2015).

2.2.2.4 Effect on humans:

In general, chloroform elicits the same symptoms of toxicity in humans as in animals. In humans, anaesthesia may result in death as a result of respiratory and cardiac arrhythmias and failure. Renal tubular necrosis and renal dysfunction have also been observed in humans (E. Ohanian, H. Galal-Gorchev, et al, 2004).

Individual studies around the world proved that the exposure to chloroform in wastewater have an effects in humans, the results of those studies showed that the chloroform can effected the humans by making colorectal cancer, bladder cancer, colon cancer and rectal cancer (E. Ohanian, H. Galal-Gorchev, et al, 2004).

2.3 Equipments, tools and setup:

The setup used in this work was composed of UV source (LED), magnetic stirrer, beaker and UV/visible spectrometer. Figure (2.1) shows the arrangement of the setup.

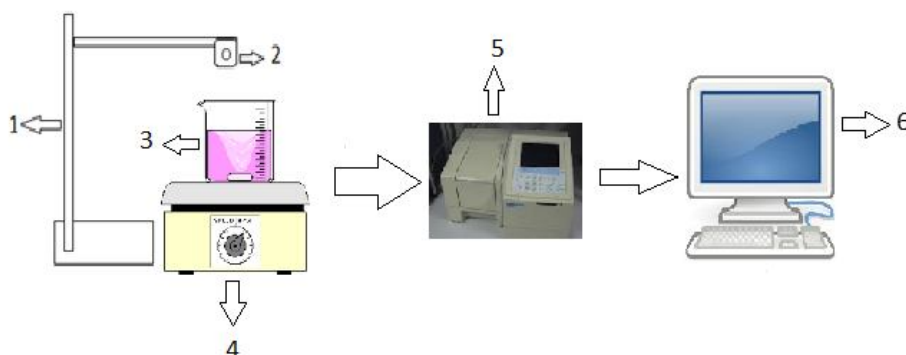


Figure (2.1) Block diagram of the setup used in this work:

- 1) Stand.
- 2) UV light source (LED).
- 3) Beaker.
- 4) Magnetic stirrer.
- 5) UV/Visible spectrometer.
- 6) Computer.

Figure (2.2) shows a photo of the setup.



Figure (2.2) photo of the setup.

The following articles describe the equipment and tools used.

2.3.1 The UV source:

The UV light source used in this work is a type of incoherent semiconductor light source. It is a p-n junction diode, which emits light when activated. When a suitable voltage is applied to the leads, electrons are able to recombine with electron holes within the device, releasing energy in the form of photons. This effect is called electroluminescence, and the color of the light (corresponding to the energy of the photon) is determined by the energy band gap of the semiconductor (www.ox.ac.uk, 2015).

The LED used in this work has an illumination color in ultraviolet with wavelength of 365nm and output power of 1200mW supplied from forward current 700mA and forward voltage 16.4V. figure (2.3) shows a photo of this source.

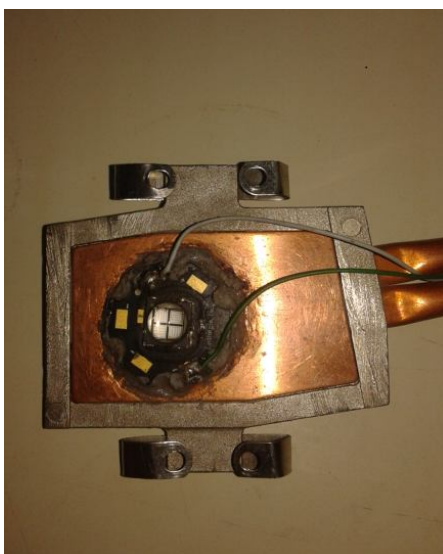


Figure (2.3) The LED with 365nm.

2.3.2 The magnetic stirrer:

A magnetic stirrer, or magnetic mixer, is a laboratory device that employs a rotating magnetic field to cause a stir bar (also called “flea”) immersed in a liquid to spin very quickly, thus stirring it. The rotating field may be created either by a rotating magnet or a set of stationary electromagnets, placed beneath the vessel with the liquid (www.ox.ac.uk, 2015).

The magnetic stirrer used in this work was manufactured by Scott Science and Healthcare limited Germany with speed of 1-10 rpm.

The magnetic stirrer used in this work, shown in figure (2.4), was used to mix the chloroform with water and the semiconductor, to make a homogeneous liquid during irradiation to make sure that all the solution is irradiated by the UV light.



Figure (2.4) The magnetic stirrer.

2.3.3 UV/visible spectrometer:

Ultraviolet-visible spectroscopy or ultraviolet-visible spectrophotometry (UV-Vis or UV/Vis) refers to absorption spectroscopy or reflectance spectroscopy in the ultraviolet-visible spectral region. This means that it uses light in the visible and adjacent (near-UV and near-infrared [NIR]) ranges. The absorption or reflectance in the visible range directly affects the perceived color of the chemicals involved in this region of the electromagnetic spectrum, molecules undergo electronic transitions. This technique is complementary to fluorescence spectroscopy, in that fluorescence deals with transitions from the excited state to the ground state, while absorption measures transitions from the ground state to the excited state (www.ox.ac.uk, 2015).



Figure (2.5) UV/Visible spectrometer.

The UV-Visible spectrometer used in this work is shown in figure (2.5) it was designed and manufactured in U.K by Jenway company, its wavelength is ranged from 190 to 1100nm. The spectrometer model was 1240mini.

2.4 The experimental procedure:

The experimental work was done in steps as follows:

- 1) The absorption spectrum of the distilled water was recorded using UV/Visible spectrometer.
- 2) One ml from chloroform was added to 100ml of distilled water, then it was mixed by the magnetic stirrer for 15minunte to make sure that all the chloroform was dissolved in water.
- 3) The absorption spectrum of water-chloroform mixture was recorded using UV/Visible spectrometer. This spectrum was used as a reference for other samples.
- 4) 300mg, of Fe_2O_3 was added to the water-chloroform mixture and stirred by the magnetic stirrer to make a homogenous solution for irradiation .

- 5) The solution then was irradiated for 15min, 30min and 45min by the UV source.
- 6) After that, the Fe_2O_3 was filtered out using the filtered paper and the absorption spectrum was recorded by UV/Visible spectrometer.
- 7) The above steps were repeated for 400mg and 700mg from Fe_2O_3 , and the spectra were recorded for 15, 30 and 45 minutes irradiation times.
- 8) The degradation of chloroform was deduced via the comparison between the absorption intensity in the spectra, specially the band at 281.7048nm.
- 9) The degradation percentage of chloroform was calculated.