Chapter Two

The Experimental Part

2.1 Introduction:

In this work the effects of CuO semiconductor and the exposure time of UV light on the photodegradiation of Malachite green in waste water were studied. This chapter presents the materials, the equipments, tools, and procedure.

2.2The materials:

The materials used in this experiment were:

2.2.1 Copper oxide:

Copper oxide is compound from the two elements copper and oxygen, It has two stable forms; cuprous oxide (CuO) and cupric oxide (CuO). These two oxides have very different colors. It is inorganic compound and used in wide range of applications. The figure (2-1) shows the CuO material in powder form.



Figure : (2-1) The CuO in powder form

2.2.1.1: The Physical and chemical properties:

Pure cupric oxide is a black solid with a density of 6.4g\cm. It also a high melting point of 1330° C, is insoluble in water and dissolve in alcohol or ammonia solution. Some as cuprous oxide, native copper vacancy in CuO makes it a p-type semiconductor, cupric oxide has an indirect band-gap 1.2eV, refractive index 2.63, and molar mass 79.545g\mol. Copper oxides are semiconductors that have been studied for several reasons such as; the easiness of production by Cu oxidation ;their nontoxic nature and reasonably good electrical and optical properties and can be prepared with simple method at very low cost (W. Longcheng, 2006).

CuO nanoparticles have been widely used as powerful heterogeneous catalysts because of its high activity and selectivity in oxidation/reduction reactions. the reactions at the CuO causing the degradation of dyes can be expressed as follows (A. B. Alabi et al, 2013).

$$CuO + hv \rightarrow CuO(e^{-} + h^{+})$$

$$CuO(h^{+}) + H^{2}O \rightarrow CuO + H^{+} + OH^{\circ}$$

$$CuO(e^{-}) + O^{2} \rightarrow CuO + O_{2}$$

$$O_{2} + H^{+} \rightarrow H_{2}^{\circ}$$

$$Dye + OH^{\circ} \rightarrow degradation \ products$$
 $Dye + h^{+} \rightarrow oxidation \ products$ $dye + e^{-} \rightarrow reduction \ products$

2.2.2 Malachite green:

Malachite green is an organic compound that is used as a dye for materials such as silk, leather, paper and pigment industry.

2.2.2.1 Physical and chemical properties:

Malachite green is a green crystal with a metallic luster; it is soluble in ethanol, methanol, and amyl alcohol and is very soluble in water, chemical formula (C₂₃ H₂₅ CIN₂). Neutral water solutions are blue-green, with an absorption maximum of 616.9 nm; aqueous solutions are yellow below pH 2. The compound has a molecular weight of 364.92. Figure (2-1) shows the chemical structure of Malachite green (B. Bryant, et.al, 2004).

Figure (2-2) Chemical structure of Malachite green.

Malachite green is hazardous dye which not just have lethal properties influencing the cells of mammals but is also a significant reason for making tumor in liver.

The dye released in water without being dealt with appropriately, affects the life-cycle of amphibian creatures and plants by hindering the infiltration of sunlight (B. Ranjit, 2001).

Malachite green is toxic to aquatic organisms and human. This dye can be absorbed by fish tissues when it is entering water cycles. It influence on the immune and reproductive system, it also carcinogenic, mutagenic, teratogenic, chromosomal fractures and also reduce fertility in fish (F. Abu Bakar, 2012).

2.3 Equipments, tools and setup:

The experimental setup that was used in this work consists of UV light source, magnetic stirrer, glass beaker, UV/Visible spectrophotometer and Quartz cell. Figure (2.2) shows a schematic diagram of the setup.

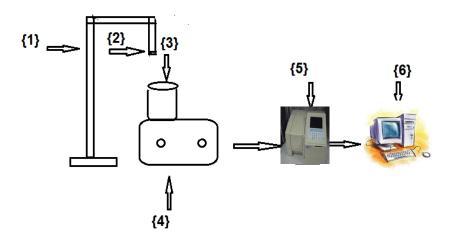


Figure (2.3) a schematic diagram of the setup.

{1} Holder, {2} The UV light source, {3} The glass beaker, {4} The magnetic stirrer, {5}The UV\ Visible spectrophotometer and {6} computer.

2.3.1 The UV Light Source:

The UV light source used in this work was composed of light emitting diode (LED) and power supply. The LED used in this work has wavelength of 365nm, forward current 700 mA, output power of 1200mW. It was manufactured by LED ENGIN. Figure (2.3) shows the UV light source used in this work.



Figure (2.4): the UV light source used in this work

2.3.2 Magnetic Stirrer:

The magnetic stirrer device is a laboratory device that is very common in experimental chemistry and biology. They are used to mix components (solids and liquids) to get homogeneous liquid mixtures. Magnetic stirrers mix solution using an external magnetic field that rotates a small stir bars that has been placed in the mixture which provides the stirring action. In this work this device was used to make homogeneous solution by mixing the semiconductor powder with the Malachite green solution. The magnetic stirrer used have was manufactured by Scott science and

healthcare limited its speed between 1-10 rpm. Figure (2.5) shows The magnetic stirrer.



Figure (2.5) The magnetic stirrer.

2.3.3 The Glass beaker:

A beaker is a simple glass container for stirring and mixing liquids commonly used to hold the sample during irradiation with capacity 100 ml.

2.3.4 The UV/Visible 1240 Spectrophotometer:

Spectrophotometer deals with visible light ,near-ultraviolet, and near – infrared. This device was used to measure the absorption of the solution before and after irradiation by UV light, in wavelength region from 190-1100 nm with auto lamp switch from visible to ultraviolet, wavelength Display 0.1nm, Accuracy \pm 1.0 nm, wavelength Repeatability \pm 0.3nm. The UV/Visible spectrophotometer used in this work was supplied from SHIMADZU company and contains quartz cell of thickness 1cm as sample holder. Figure (2-6) shows a photo for this device.



Figure (2.6) UV\Visible 1240 spectrometer.

2.3.5 The quartz cell:

The quartz cell is employed as a liquid sample container. Its dimensions are 1cm and width, height 4.2 cm manufactured by Jenway. Figure (2.7) shows the quartz cell



Figure (2.7) The quartz cell

2.3.6 The experimental method:

The experimental work was done in steps as follows:

- five mg from Malachite green was added to 100ml of distill water.
- 300mg from catalyst(CuO) was added to the first sample,while
 500mg from CuO was added to the second sample and 1000 mg of
 CuO was added to the third sample .
- A small portion from 5mg Malachite green(MG) in 100 ml of distilled water was put in the quartz cell and placed in the UV spectrophometer and the absorption spectrum was recorded and used as reference.
- The first sample was irradiated with UV light source with wavelength of 365nm and output power of 1200 mW for 10 minutes, then small portion was filtered off and placed in a quartz cell. The absorption spectrum of this sample was recorded using UV spectrometer.
- The above sample was irradiated with 20 minute and then 30 minutes. The absorption spectra were recorded for the two sample.
- The above steps were repeated for the second sample.
- The above steps were repeated for the third sample but with irradiation times; (10, 20, 40) minute. Then the absorption spectra were record for each sample.
- The degradation of Malachite green was estimated from its absorption intensity in the spectra.