Chapter Three

Results and Discussion

3.1 Introduction:

The application of UV light induced photocatalytic process was studied in this work to deduce the chloroform degradation in water.

The results obtained in this study are presented here in two categories:

1) The dependence of the chloroform degradation on the irradiation time.
2) The dependence of the chloroform degradation on the amount of the Fe$_2$O$_3$.

The data concerning the behaviour of chloroform degradation were obtained using different amounts of Fe$_2$O$_3$ semiconductor (300, 400 and 700mg), and three different exposure times (15, 30 and 45 minute). Furthermore, data are presented as absorption spectra where the intensity of the absorption band of chloroform (281.7049 nm) is compared for both categories, before and after irradiation.

3.2 Spectrum of the pure distilled water:

The absorption spectrum of the pure distilled water was recorded as shown in the figure (3.1).
Figure (3.1) The absorption spectrum of the pure distilled water

The spectrum shows that the distilled water has a strong absorption band centered at 973.3387nm.

3.3 The absorption spectrum of chloroform in distilled water:
One ml of chloroform was added to 100ml distilled water, and the absorption spectrum of the mixture was recorded as shown in figure (3.2).
Figure (3.2) The absorption spectrum of 1 ml from chloroform in 100 ml distilled water.

Figure (3.2) shows that when the mixture of 1 ml chloroform and 100 ml of distilled water, has an absorption peak at 281.7049 nm. So when compared with figure (3.1) one can conclude that the new peak is an absorption band of chloroform.

3.4 Degradation of chloroform:
The absorption spectra of chloroform after irradiation by the UV source (365 nm) with output power 1200 mW for different times and different amounts of Fe₂O₃ catalyst were recorded and compared.
3.4.1 The spectra after adding 300mg Fe$_2$O$_3$ and irradiation with different times:

After adding 300mg from Fe$_2$O$_3$ to 1ml chloroform dissolved in 100ml of distilled water, the mixture was irradiated by the UV source for different times (15min, 30min and 45min). After that, the Fe$_2$O$_3$ was filtered out and the absorption spectra were recorded as shown in figure (3.3). The spectra were compared with that obtained before irradiation as shown in the figure.

![Absorption Spectra](image.png)

**Figure (3.3) The spectra of a mixture composed of 1ml chloroform and 300mg Fe$_2$O$_3$ irradiated for different times.**

Table (3.1) lists the results of the comparison between the intensities of the band at 281.704 in each case.
Table (3.1): The absorption intensities of the band at 281.704nm using 300mg from Fe$_2$O$_3$ for different exposure times.

<table>
<thead>
<tr>
<th>Weight of Fe$_2$O$_3$ (mg)</th>
<th>Exposure time (min)</th>
<th>Wavelength (nm)</th>
<th>Absorption band intensity (a.u)</th>
<th>Degradation percentage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without Fe$_2$O$_3$</td>
<td>No exposure time</td>
<td>281.7049</td>
<td>0.051</td>
<td>0.00</td>
</tr>
<tr>
<td>300</td>
<td>15</td>
<td>281.7049</td>
<td>0.045</td>
<td>11.77</td>
</tr>
<tr>
<td>300</td>
<td>30</td>
<td>281.7049</td>
<td>0.035</td>
<td>31.38</td>
</tr>
<tr>
<td>300</td>
<td>45</td>
<td>281.7049</td>
<td>0.029</td>
<td>43.14</td>
</tr>
</tbody>
</table>

Figure (3.4) shows the relation between the exposure time and the intensity of the absorption band of the chloroform and 300 mg Fe$_2$O$_3$ irradiated for different times.

Figure (3.4) The relation between the exposure time and absorption intensity of a mixture composed of 1 ml chloroform and 300 mg Fe$_2$O$_3$ irradiated for different times.
3.4.2 The spectra after adding 400mg Fe$_2$O$_3$ and irradiation with different times:

After adding 400mg from Fe$_2$O$_3$ to 1ml chloroform dissolved in 100ml of distilled water, the mixture was irradiated by the UV source for different times (15min, 30min and 45min). After that, the Fe$_2$O$_3$ was filtered out and the absorption spectra were recorded as shown in figure (3.5). The spectra were compared with that obtained before irradiation as shown in the figure.

![Absorption spectra](image)

**Figure (3.5)** The spectra of a mixture composed 1ml chloroform and 400mg Fe$_2$O$_3$ irradiated for different times.

Table (3.2) lists the results of the comparison between the intensities of the band at 281.704 in each case.
Table (3.2): The absorption intensities of the band at 281.704nm using 400mg from Fe₂O₃ for different exposure times.

<table>
<thead>
<tr>
<th>Weight of Fe₂O₃ (mg)</th>
<th>Exposure time (min)</th>
<th>Wavelength (nm)</th>
<th>Absorption band intensity (a.u)</th>
<th>Degradation percentage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without Fe₂O₃</td>
<td>No exposure time</td>
<td>281.7049</td>
<td>0.051</td>
<td>0.00</td>
</tr>
<tr>
<td>400</td>
<td>15</td>
<td>281.7049</td>
<td>0.042</td>
<td>17.77</td>
</tr>
<tr>
<td>400</td>
<td>30</td>
<td>281.7049</td>
<td>0.029</td>
<td>43.14</td>
</tr>
<tr>
<td>400</td>
<td>45</td>
<td>281.7049</td>
<td>0.022</td>
<td>56.87</td>
</tr>
</tbody>
</table>

Figure (3.6) shows the relation between the exposure time and the intensity of the absorption band of the chloroform and 400 mg Fe₂O₃ irradiated for different times.

Figure (3.6) The relation between the exposure time and absorption intensity of a mixture composed 1 ml chloroform and 400 mg Fe₂O₃ irradiated for different times.
3.4.3 The spectra after adding 700mg Fe$_2$O$_3$ and irradiation with different times:

After adding 700mg from Fe$_2$O$_3$ to 1ml chloroform dissolved in 100ml of distilled water, the mixture was irradiated by the UV source for different times (15min, 30min and 45min). After that, the Fe$_2$O$_3$ was filtered out and the absorption spectra were recorded as shown in figure (3.7). The spectra were compared with that obtained before irradiation as shown in the figure.

Table (3.3) lists the results of the comparison between the intensities of the band at 281.704 in each case.
Table (3.3): The absorption intensities of the band at 281.704nm using 700mg from Fe$_2$O$_3$ for different exposure times.

<table>
<thead>
<tr>
<th>Weight of Fe$_2$O$_3$ (mg)</th>
<th>Exposure time (min)</th>
<th>Wavelength (nm)</th>
<th>Absorption band intensity (a.u)</th>
<th>Degradation percentage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without Fe$_2$O$_3$</td>
<td>No exposure time</td>
<td>281.7049</td>
<td>0.051</td>
<td>0.00</td>
</tr>
<tr>
<td>700</td>
<td>15</td>
<td>281.7049</td>
<td>0.032</td>
<td>37.26</td>
</tr>
<tr>
<td>700</td>
<td>30</td>
<td>281.7049</td>
<td>0.019</td>
<td>62.75</td>
</tr>
<tr>
<td>700</td>
<td>45</td>
<td>281.7049</td>
<td>0.000</td>
<td>100</td>
</tr>
</tbody>
</table>

Figure (3.8) shows the relation between the exposure time and the absorption intensity the band at 281.704 nm of the chloroform and 700 mg Fe$_2$O$_3$ irradiated for different times.

Figure (3.8) The relation between the exposure time and absorption intensity of a mixture composed of 1 ml chloroform and 700 mg Fe$_2$O$_3$ irradiated for different times.
Table (3.4) lists the results of the comparison between the intensities of the band at 281.704 nm for a mixture of chloroform and Fe$_2$O$_3$ (different weights) after irradiation with 15 min.

**Table (3.4): The absorption intensities of the band at 281.704 nm after irradiation with 15 min and different weights of Fe$_2$O$_3$.**

<table>
<thead>
<tr>
<th>Weight of Fe$_2$O$_3$ (mg)</th>
<th>Exposure time (min)</th>
<th>Wavelength (nm)</th>
<th>Absorption band intensity (a.u)</th>
<th>Degradation percentage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without Fe$_2$O$_3$</td>
<td>No exposure time</td>
<td>281.7049</td>
<td>0.051</td>
<td>0.00</td>
</tr>
<tr>
<td>300</td>
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<td>281.7049</td>
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<tr>
<td>400</td>
<td>15</td>
<td>281.7049</td>
<td>0.042</td>
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<tr>
<td>700</td>
<td>15</td>
<td>281.7049</td>
<td>0.032</td>
<td>37.26</td>
</tr>
</tbody>
</table>

Figure (3.9) shows the relation between the weights of Fe$_2$O$_3$ and the absorption intensity of the chloroform irradiated for 15 min.
Figure (3.9) The relation between the weights of Fe$_2$O$_3$ and the absorption intensity of a mixtures composed of 1 ml chloroform and different weights of Fe$_2$O$_3$ irradiated for 15 min.

Table (3.5) lists the results of the comparison between the intensities of the band at 281.704 nm with 30 min exposure time and different weights of Fe$_2$O$_3$.

Table (3.5): The absorption intensities of the band at 281.704 nm after irradiation for 30 min and different weights of Fe$_2$O$_3$.

<table>
<thead>
<tr>
<th>Weight of Fe$_2$O$_3$ (mg)</th>
<th>Exposure time (min)</th>
<th>Wavelength (nm)</th>
<th>Absorption band intensity (a.u)</th>
<th>Degradation percentage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without Fe$_2$O$_3$</td>
<td>No exposure time</td>
<td>281.7049</td>
<td>0.051</td>
<td>0.00</td>
</tr>
<tr>
<td>300</td>
<td>30</td>
<td>281.7049</td>
<td>0.035</td>
<td>31.38</td>
</tr>
<tr>
<td>400</td>
<td>30</td>
<td>281.7049</td>
<td>0.029</td>
<td>43.14</td>
</tr>
<tr>
<td>700</td>
<td>30</td>
<td>281.7049</td>
<td>0.019</td>
<td>62.75</td>
</tr>
</tbody>
</table>
Figure (3.10) shows the relation between the weights of Fe₂O₃ and the absorption intensity of the chloroform irradiated for 30 min.

Table (3.6) lists the results of the comparison between the intensities of the band at 281.704 nm with 45 min exposure time and different weights of Fe₂O₃.
Table (3.6): The absorption intensities of the band at 281.704nm after irradiation of 45min and different weights of Fe₂O₃.

<table>
<thead>
<tr>
<th>Weight of Fe₂O₃ (mg)</th>
<th>Exposure time (min)</th>
<th>Wavelength (nm)</th>
<th>Absorption band intensity (a.u)</th>
<th>Degradation percentage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without Fe₂O₃</td>
<td>No exposure time</td>
<td>281.7049</td>
<td>0.051</td>
<td>0.00</td>
</tr>
<tr>
<td>300</td>
<td>45</td>
<td>281.7049</td>
<td>0.029</td>
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<td>400</td>
<td>45</td>
<td>281.7049</td>
<td>0.022</td>
<td>56.87</td>
</tr>
<tr>
<td>700</td>
<td>45</td>
<td>281.7049</td>
<td>0.00</td>
<td>100</td>
</tr>
</tbody>
</table>

Figure (3.11) shows the relation between the weights of Fe₂O₃ and absorption intensity of the chloroform irradiated for 45 min.

Figure (3.11) shows the relation between the weights of Fe₂O₃ and absorption intensity of the chloroform irradiated for 45 min.
3.5 Discussion:

As shown in figures (3.3), (3.4) and (3.5), the chloroform degradation is observable and that is due to many reasons. For example in figure (3.3), when the amount of Fe$_2$O$_3$ was 300mg and the exposure time was increased from 15min, 30min to 45min, the intensity of the chloroform absorption peak (at 281.7049 nm) was decreased significantly with increasing the exposure time, which increase the amount of produced charge carriers. That is also noticed in figures (3.4) and (3.5), where the amount of Fe$_2$O$_3$ was 400mg and 700mg respectively, and the exposure time increased from 15min to 30min and 45min, the intensity of the chloroform absorption peaks decreased significantly with increasing the exposure time. The degradation percentage of the chloroform was increased by increasing the exposure time and the weight of Fe$_2$O$_3$.

As one can see in figures (3.3), (3.4) and (3.5), and tables (3.1), (3.2) and (3.3), the intensity of the chloroform absorption peak (at 281.7049 nm) was decreased significantly with increasing the Fe$_2$O$_3$ weight and this peak was completely disappeared when the Fe$_2$O$_3$ weight reach 700mg and the exposure time was 45min by 100% degradation percentage as shown in figure (3.5) and table (3.3). Increasing the semiconductor weight lead to more production of electron-hole pairs and that means increasing the carrier charges. When the semiconductor particles exposed to the UV source, where its energy is higher than the band gap of Fe$_2$O$_3$, the production of electron-hole pairs is increased and these species are capable to reduce.

A possible reaction pathway for the photocatalytic degradation of chloroform is as follows (W. Abu Bakar, R. Ali and M. Othman, 2010):

$$ CHCl_3 + hv + Fe_2O_3 \rightarrow Cl^- + CHCl_2^+ \rightarrow CCl_2^+ + HCl $$
\[ H_2O \xrightarrow{hv + Fe_2O_3} OH^* + H^* \]

\[ CHCl_2^+ + OH^* \rightarrow OCl^+ + CH_2Cl^+ \]

\[ CH_2Cl^+ + H^* \rightarrow CH_3Cl^+ \]

\[ Cl^* + CHCl_2^+ \rightarrow CCl_3^+ + HCl \]

\[ CHCl_3 + OH^* \rightarrow H_2O + CCl_3^+ \]

\[ CCl_3^+ + O_2 \leftrightarrow CCl_3O_2^+ \]

\[ 2CCl_3O_2^+ \rightarrow 2COCl_2 + O_2 + 2Cl^* \]

\[ CHCl_2^+ + O_2 \rightarrow CHCl_2O^+ \]

\[ 2CHCl_2O^+ \rightarrow 2CHCl_2O^+ + O_2 \]

\[ CHCl_2O^+ \rightarrow HCOCl + Cl^* \]

\[ COCl_2^+ \xrightarrow{hv + Fe_2O_3} CO + 2Cl^* \]

\[ COCl_2^+ + H_2O \rightarrow 2HCl + CO_2 \]

\[ 2Cl^* \rightarrow Cl_2 \]

\[ OH^* + H^* \rightarrow H_2O \]
3.6 Conclusions:

From the results obtained in this work the follows can be concluded:

1. The objectives of this study were achieved successfully.
2. Degradation of chloroform is increased with increasing the exposure time.
3. The degradation of chloroform is increased with increasing the semiconductor catalyst amount.
4. The degradation percentage of chloroform is 100%, in case of 700mg Fe₂O₃ irradiated with UV source for 45 minute.

4.7 Recommendations:

The following ideas can be recommended:

1. Study the photodegradation process for other organic compounds which can be considered as water pollutants such as aston and cresol.
2. Using other semiconductors such as WO₃, TiO₂ and NiO.
3. Study the photodegradation process under solar irradiation.
References:


J. Gupta, (2007), *Semiconductors and Devices*; S.K. KATARIA and SONS.


L. Simin, (2010), *Photocatalytic Hydrogen Production with Iron Oxide Under Solar Irradiation*; Faculty of Science and Technology, Queensland University of Technology, Brisbane.


M. Herrman, (2005), Heterogeneous photocatalysts: state of the art and present applications. *Topics in catalysis*, pp. 49-65.


