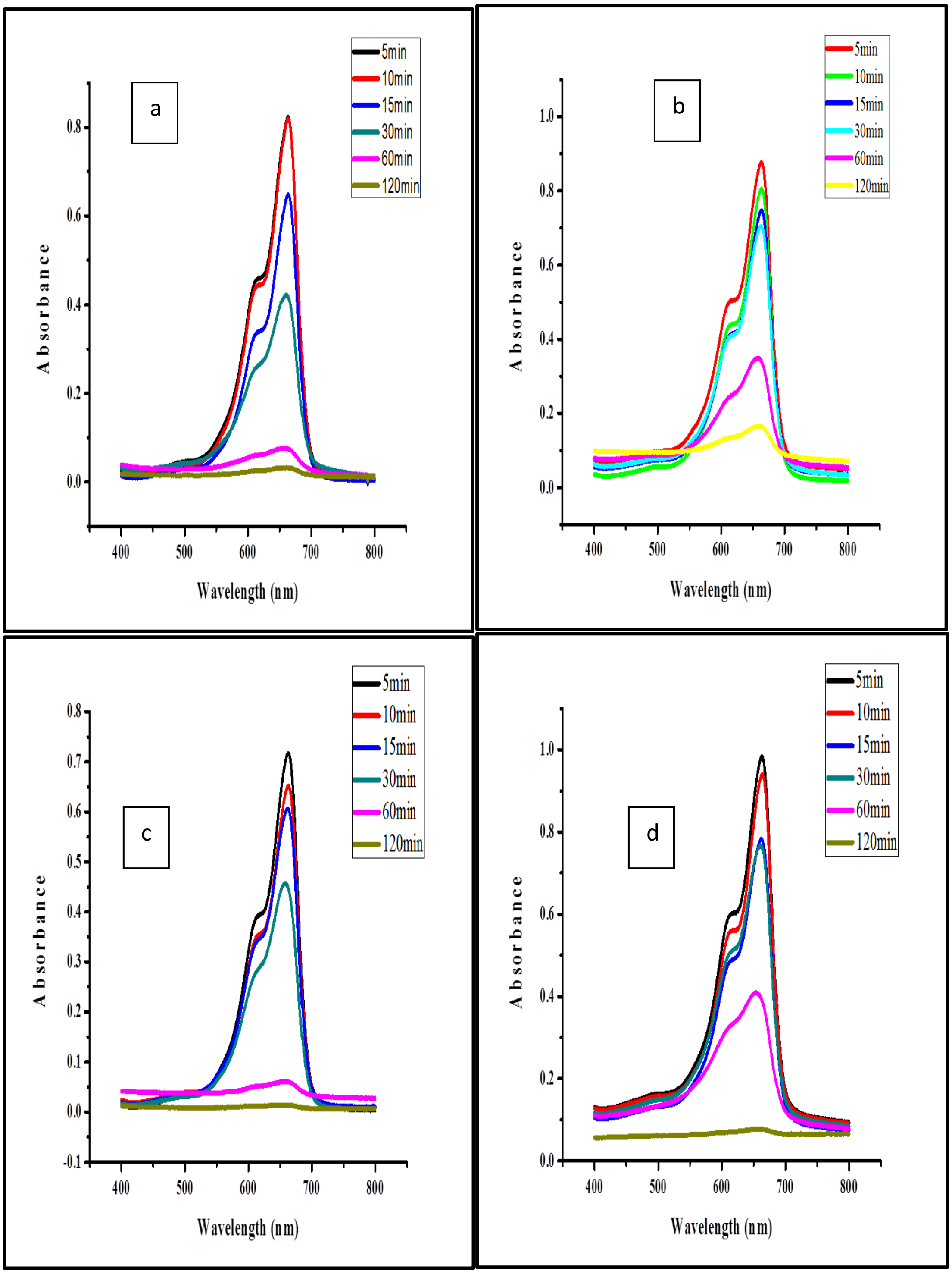
a
b
c
d
5min
10min
15min
30min
60min
120min
Absorbance
Wavelength (nm)

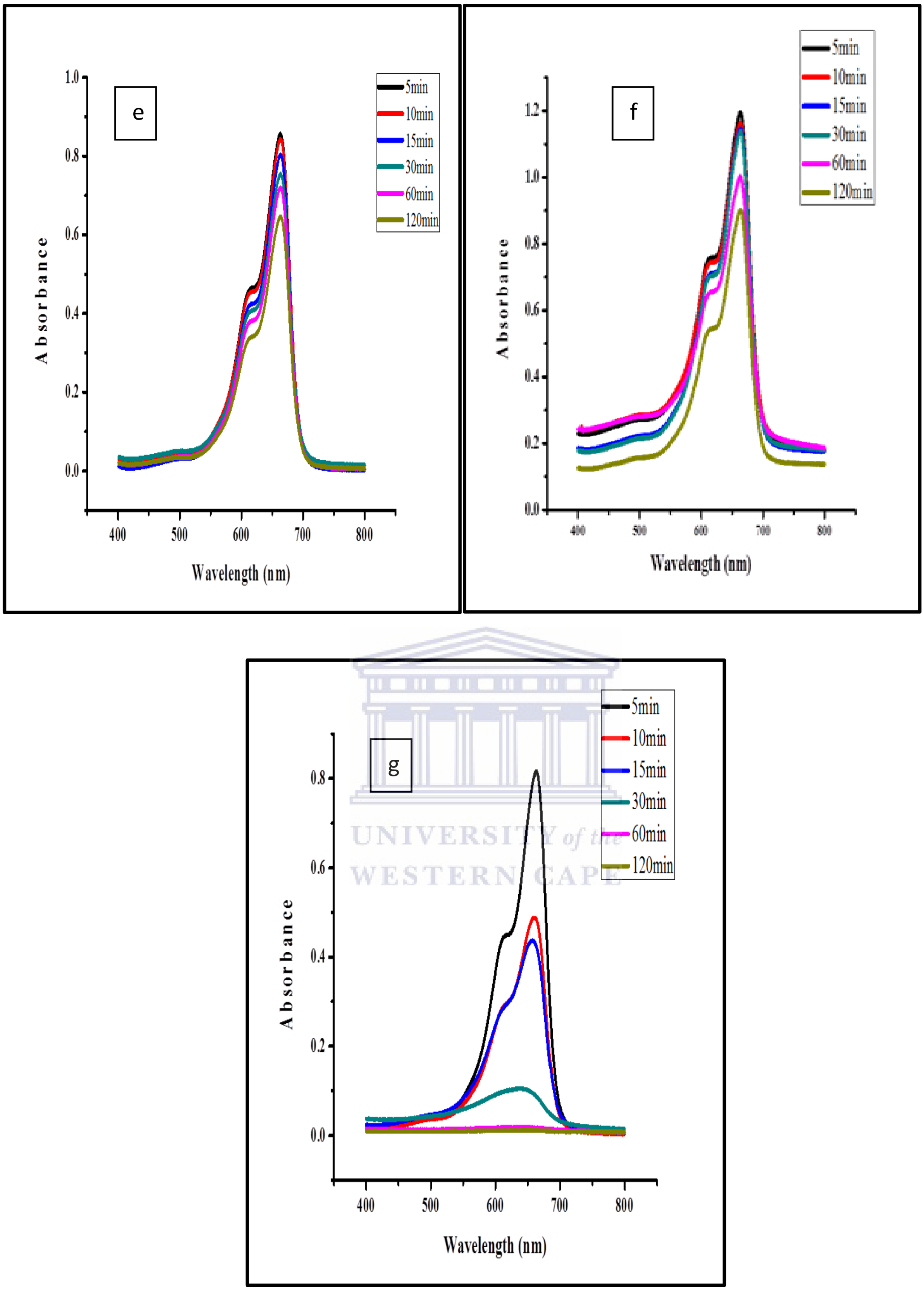

Figure 5.31: UV spectra of the photodegradation process over time using (a) blank, (b) Fe_3O_4, (c) $Fe_3O_4/SiO_2/TiO_2$, (d) Ag-$Fe_3O_4/SiO_2/TiO_2$ (e), Commercial TiO_2, (f) Com TiO_2 (no UV irradiation) and (g) $Fe_3O_4/SiO_2/TiO_2$ (no UV irradiation)

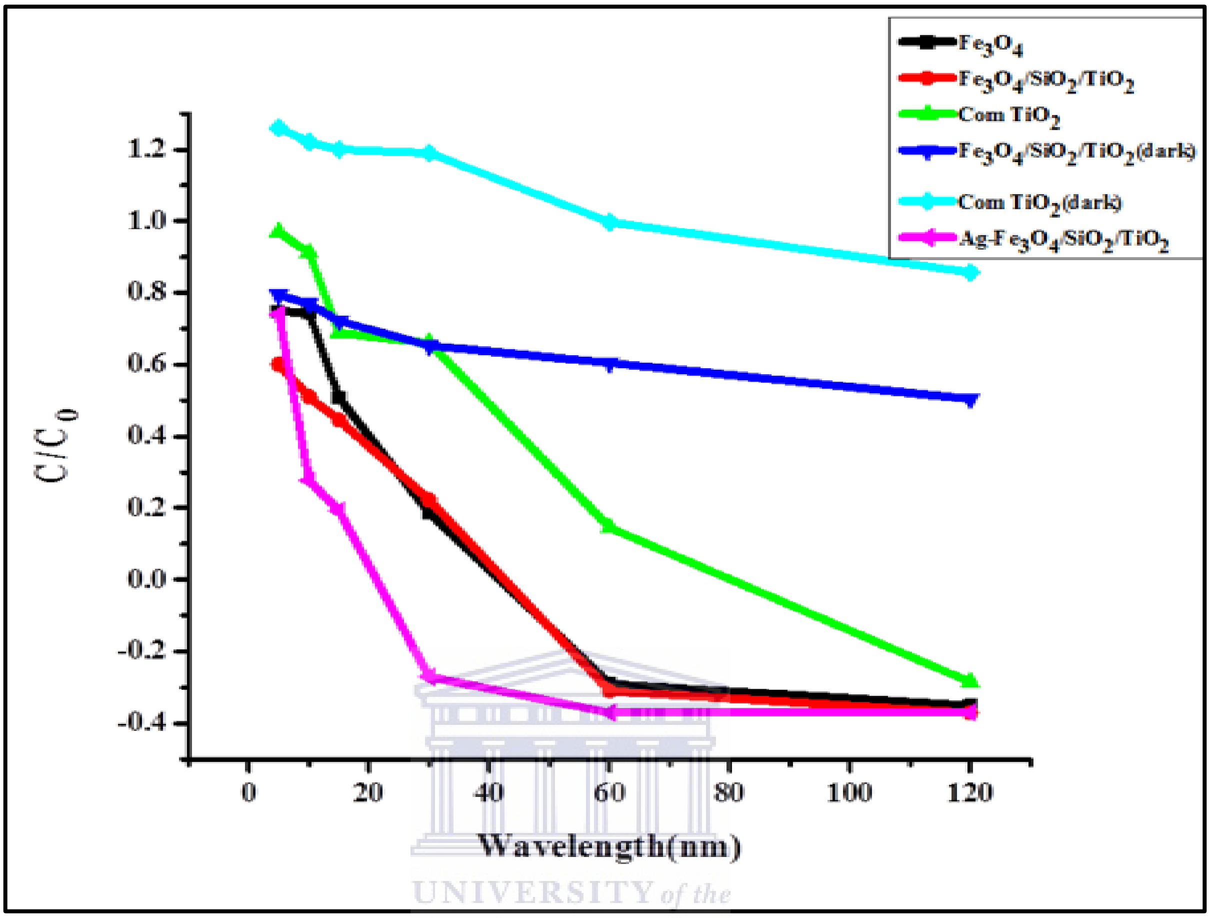

Figure 5.32: UV spectra of the photodegradation process over time using

Commercial TiO_2 (Degussa P25 titanium dioxide), which is known to be the best photocatalyst commercially available, was used as a reference to qualitatively assess the photocatalytic performance of the as-synthesized samples. Figure 5.5 shows the degradation of methylene under UV light in the presence of Fe_3O_4, $Fe_3O_4/SiO_2/TiO_2$, and Ag-$Fe_3O_4/SiO_2/TiO_2$ and in the absence of a catalyst. A rapid decrease in the absorption of methylene blue was observed at 664nm. The degradation rate is fast at the beginning of the reaction and then became slow. A sharp decrease of the major absorption band within 30 min indicates that this sample exhibits excellent photocatalytic activity for the degradation of MB.

In the absence of a catalyst the degradation rate shows the same phenomenon however the degradation of the dye maintains the same rate even after 30min, suggesting the addition of catalysts increases the degradation of MB. The photocatalytic activity of Ag-coated Fe_3O_4/TiO_2 nanoparticles is much higher than that of Degussa P25 titania, Fe_3O_4 and the Fe_3O_4/TiO_2 nanoparticles. The deposition of metallic Ag nanoparticles on the surface of the Fe_3O_4/TiO_2 nanocomposites increases the photocatalytic activity, which indicates that the Ag–TiO_2 heterojunction can improve electron-charge separation efficiency.

In order to investigate the efficiencies of mineralization for MB in these three catalysts, photocatalytic performance was also measured during the reaction. The figures above show degradation efficiencies of dye waste water after 2hrs. From these results, it is noteworthy that the Ag-coated $Fe_3O_4/SiO_2/TiO_2$ microspheres are the strongest at mineralizing MB among the catalysts.

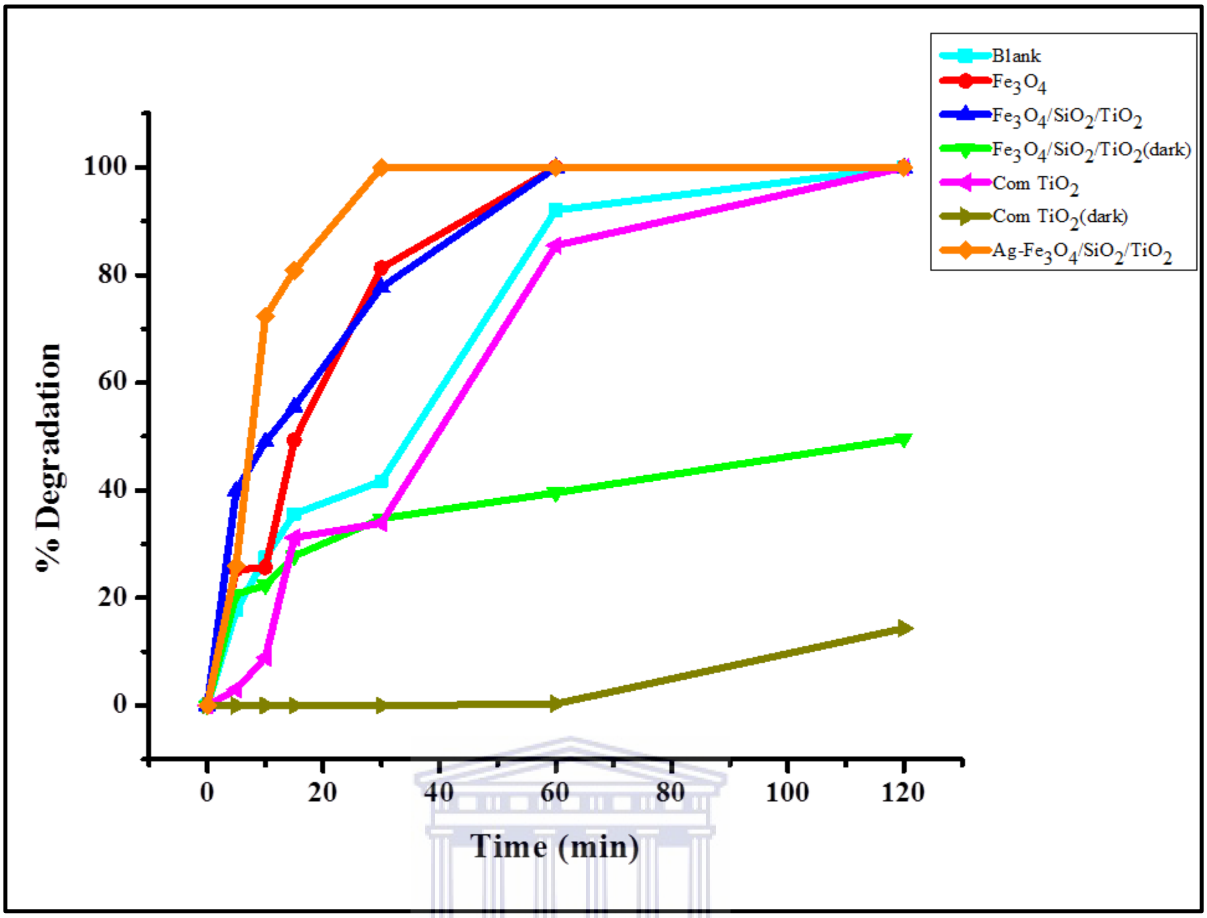

Figure 5.33: Degradation percentage of MB dye over time

The degradation was calculated using the formula

$$\%\text{degradation} = \frac{Co - Cf}{Co} \times 100$$

Where C_o is the initial concentration

C_f is the final concentration

The maximum MB degradation efficiency for the Ag coated Fe_3O_4/TiO_2 nanoparticles is about 81%, and complete disappearance of colour in 15min and good activity of silver doped titanium dioxide catalyst have been reported by (Sung-Suh, *et al.*, 2004; Sobana, *et al.*, 2006. The Fe_3O_4 and $Fe_3O_4/SiO_2/TiO_2$ reached 92% and 77.8% in 60 and 30 minutes respectively. This proved that the titanium dioxide shell improved the photocatalytic performance of Fe_3O_4 nanoparticles. The commercial TiO_2 catalyst under UV irradiation showed 85.5% degradation of MB dye after 60min, which is less than that achieved by Fe_3O_4. The $Fe_3O_4/SiO_2/TiO_2$ catalyst and commercial TiO_2, in the absence of UV irradiation, only achieved 49.6 and 14.3% degradation of MB after two hours showing that the use of UV light also enhances the photodegradability of MB dye. It is also evident that the as-synthesized catalyst had better photocatalytic activity than commercial TiO_2 and this has also been reported (Gao,*et al.*, 2003; Shi , *et al.*, 2012).

Chapter 6: Conclusions, future work and recommendations

Conclusion

A silver doped magnetite titanium dioxide nanocomposite was successfully synthesized using sol-gel and hydrothermal techniques. The as-synthesized catalysts were characterized using FTIR, SEM, TEM, XRD, BET, TGA, and UV. The TEM measurements showed cubic shaped particles and spherical shaped Ag particles whose average size were 66 nm, 74nm and 61nm for Fe_3O_4, $Fe_3O_4/SiO_2/TiO_2$ and Ag-$Fe_3O_4/SiO_2/TiO_2$. The synthetic techniques which were used were fast and give uniform distribution of crystallites of $Fe_3O_4/SiO_2/TiO_2$ confirmed by XRD studies.

The catalysts were evaluated for their photocatalytic performance for the degradation of a standard methylene blue solution. The as-synthesized catalysts showed good degradation activities compared to commercial P25 TiO_2. Their performance showed degradation efficiency in the order Ag-$Fe_3O_4/SiO_2/TiO_2$> $Fe_3O_4/SiO_2/TiO_2$ > Fe_3O_4> Degussa P25 TiO_2. Silver nanoparticles on the TiO2 surface behave as sites for electron accumulation; increasing the electron-whole separation (reducing recombination reactions). This promotes efficient channelling of charge carriers into useful reduction and oxidation reactions enhancing photocatalytic activity.

Furthermore the nanocomposite maintained good magnetic properties and could be separated easily from reaction mixture making use of an external magnet. The photocatalysts was recovered without any or negligible mass loss. The results of the present work and the referenced literature show that photocatalytic treatment of textile industry waste-wat er is a promising tool for the decolourization and possible demineralisation of coloured wastewaters.

Future work and recommendations

The future work is to investigate the reusability and application of the catalyst for other dyes under varied reaction conditions such as MB solution concentration, catalyst loading. Silver deposition on the TiO_2 layer is suggested to decrease the bandgap of TiO_2thus enhancing the photocatalytic performance of the nanocomposite. The bandgap studies of the nanocomposite and varying the Fe_3O_4/SiO_2/TiO_2 and Ag ratios. The catalysts synthesized in the study can also be evaluated for photocatalytic activity using visible light instead of ultra-violet irradiation.

Chapter 7: References

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