

## Synthesis and Characterization of TiO<sub>2</sub>/C Composite for Photocatalytic Degradation of Dyes.

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Titanium dioxide TiO<sub>2</sub> nanoparticles have moderate catalytic activity due to its wide band-gap and high rate of electron-hole recombination [1]. The TiO<sub>2</sub> electrical conductivity poor is improved by mixing with other materials. Therefore, TiO<sub>2</sub> based nanocomposites have been synthesized to increase the photocatalytic activity, as well as their structural characteristics and electrochemical performance, using dopants like carbon, nitrogen, sulfur and others. TiO<sub>2</sub>/C composite has been proven to be a promising photocatalyst for pollutants, due to C-doping, morphology, structure and mixed phases [1]. Carbon precursors are reported to synthesize TiO<sub>2</sub>/C, such as: glucose, oleic acid, carbon nanofiber, graphene oxide, activated carbon, carbon nanotubes, graphite, resorcinol and formaldehyde. Dyes used for industries, are significant sources of environmental pollution, because they are non-biodegradable [2]. Methyl orange (MO) and methyl blue (MB) have been used to help determine the activity of the photocatalyst [3].

The reagents used were: anatase powder 99.8% (metals basis), sucrose (99.5%), H<sub>2</sub>SO<sub>4</sub> (65% wt), distilled water, MO (MW=327.33g/mol) and MB (MW=319.85g/mol). TiO<sub>2</sub>/C composites were synthesized via infiltrating sucrose into anatase. In a typical synthesis, anatase (A) and sucrose (S) with molar ratio of A/S=6, sulfuric acid and distilled water were mixed completely. The mixture was then put in a drying oven, treated at 100°C for 6h and subsequently at 160°C for 6h. The resulting brown precursor powder was carbonized in a tubular furnace at 800°C for 1h in argon atmosphere. Rigaku D-Max 2200 diffractometer was used to obtain XRD patterns using Cu K $\alpha$  radiation. The surface morphology and the crystalline phases were examined with Field Emission Transmission Electron Microscope, JEM 2010F JEOL. The photocatalytic activity was tested for degradation of MB and MO with an initial concentration of 20 ppm, using 0 and 0.34 g/L of TiO<sub>2</sub>/C, under radiant flux provided by 175 W UV. The MB and MO concentrations were measured by UV-vis spectroscopy (Aiglet-Vis spectrophotometer).

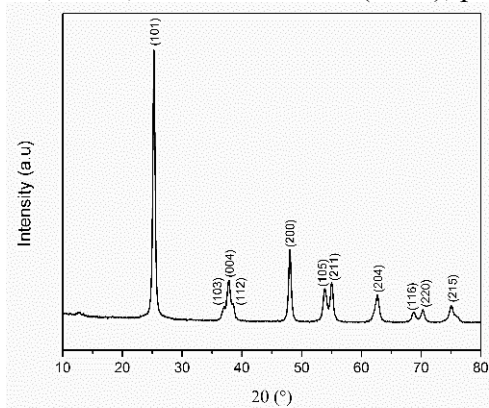
The XRD patterns of TiO<sub>2</sub>/C composite is shown in Fig. 2, in which all the characteristic diffraction peaks of the TiO<sub>2</sub>/C composite can be respectively indexed with planes of anatase phase of TiO<sub>2</sub> (JCPDS No. 21-1272). Moreover, a small peak at 11.5° is observed, characteristic of weakly ordered graphitic microstructure, indicating the trace amount of graphite in the composite [4]. No significant peaks of carbon or rutile are observed after the precursor powders are heat treated under argon atmosphere, which suggesting its amorphous nature and confirmed the high purity of the TiO<sub>2</sub>/C composite [5], because impurities have been reported due to the transformation from anatase to rutile phase [1]. The broad diffraction peaks indicate the sample's nanocrystalline nature. Fig. 3a shows the micrographs of TEM bright field of powders; it is clearly seen that the TiO<sub>2</sub>/C composite powders have mostly spherical morphology. Further, it can be estimated that the particle size of samples is of the microscale order with grain size of the range of 20-30nm. Fig. 3b shows the atomic structure and the crystallinity of TiO<sub>2</sub>/C composite through HRTEM. The insert image shown in Figure 3b give the corresponding Fast Fourier Transform (FFT) pattern of the anatase; this pattern displays (101) and (200) planes from the interplanar

spacing of 0.352 and 0.189 nm respectively (JCPDS No. 21-1272). The results of XRD concurred with the electron diffraction pattern created by FFT from HRTEM. The degradation percentage for MO and MB were 99.95 and 99.99 % at 15 min on TiO<sub>2</sub>/C whereas 86 and 81 % at 90 min for MB and MO in the absence of catalyst is illustrated in Fig. 5.

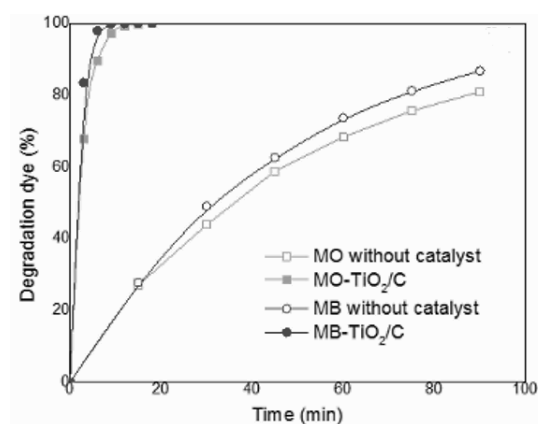
TiO<sub>2</sub>/C composite was synthesized using anatase and sucrose by obtaining a precursor powder at low temperature (160°C), which is carbonized at 800°C in argon atmosphere. The XRD analysis reveals that the TiO<sub>2</sub>/C composite is a phase anatase unique structure with high crystallization, which had no impurities, but it showed a trace amount of graphite in the composite. The TiO<sub>2</sub>/C composite were found to be efficient catalyst for the photodegradation of MB and MO dyes under UV irradiation. The reaction was found to follow pseudo-first order kinetics described it well. This method could be extended to synthesize a variety of other composites for photocatalytic degradation of dyes.

#### References:

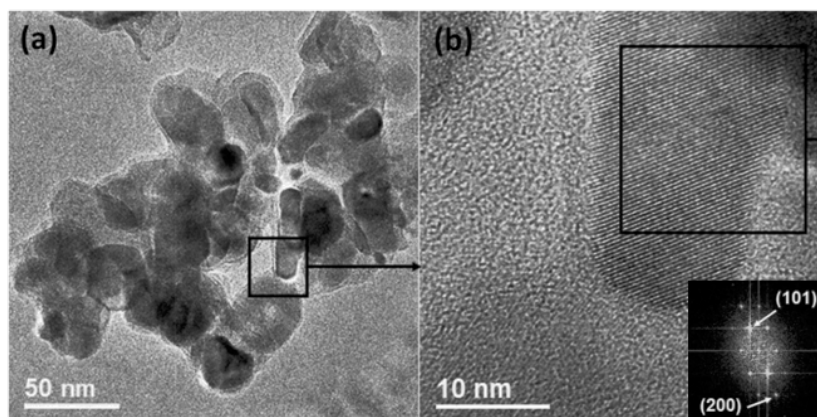
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**Figure 1.** XRD pattern of TiO<sub>2</sub>/C composite.



**Figure 3.** Degradation percent of MO and MB on TiO<sub>2</sub>/C.



**Figure 2.** (a) TEM micrograph of TiO<sub>2</sub>/C composite nanoparticles, (b) HRTEM image of TiO<sub>2</sub>/C composite. The insert in (b) shows the corresponding electron diffraction by FFT pattern.