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Synthesis and Optical Properties of Dye-Sensitized films based on Anatase Cu-TiO2 Nanoparticles

Semiconductor oxides have been applied extensively in photovoltaic devices. In DSSCs, semiconductor oxides have been applied as the main active layer as electron transport materials and as interfacial layers in solar cells [1, 2]. Titanium dioxide (TiO2) has received a great deal of attention due to its chemical stability, non-toxicity, low-cost, and other advantageous properties [3]. Nanocrystalline TiO2 is an interesting semiconducting material, because of its unique optical and electrical properties, which are suitable for solar energy conversion applications such as photocatalysts, photochromics and photovoltaics [4, 5]. Therefore, the stabilization of anatase phase has become a subject of interest to be studied. Numerous researchers had attempted various procedures to stabilize the anatase phase of TiO2 using physical approaches such as metal organic chemical vapor deposition [6], epitaxial growth using reactive sputter deposition [7], freeze-drying and chemical approaches such as solvothermal method [8-10], precipitation method [11, 12], sol–gel method [13-15], chemical vapor deposition [16], thermal decomposition of alkoxide [17] and impurity addition to modify the structure [18]. Sol-gel technique is being extensively used for the synthesis of advanced ceramics, production of nanocrystalline materials and for metallurgical treatment of ores and minerals to yield value-added materials. Sol–gel method is easy to produce relatively large quantities of nanoparticles at low-cost. Anatase TiO2 is commonly regarded as the most promising materials for DSSCs due to their wide band gaps, which are suitable for...
interfacial electron transport to take place. Such applications of titania have been found to depend strongly on the crystalline structure, size and morphology [19]. Therefore controlling novel morphologies and developing effective doping strategies are two important tasks for advancing TiO\textsubscript{2}-based nanomaterials [20]. In order to improve the photoefficiency of electronic process of TiO\textsubscript{2} under UV region several kinds of modification methods have been explored such as noble metal doping, composite semiconductors and doping with transition metals [21, 22, 35]. Doping is known as one of the effective ways to manipulate the internal properties of host material such as crystalline structure and crystallite size. The band-gap energies of anatase TiO\textsubscript{2} semiconductors are found to be around 3.75 eV. Many attempts have been made to tune band gap by varying the composition of transition metal ions. It could be possible to decrease the band gap of semiconductors by the use of right dopants. A few studies have been suggested that the TiO\textsubscript{2} doped with transition metal ions could possess interesting optical properties [23-27]. The origin of these improved photoreactivities is clearly related to the efficiencies of the doping centers to trap charge carriers and to intercede in the interfacial transfer. Trapping of either an electron or hole alone is not effective to increase photoefficiency, because of the recombination of immobilized charge species with mobile counterpart. Thus, metal ions can also serve as charge trapping sites and thus reduce electron–hole recombination rate [28, 29]. The band gap of TiO\textsubscript{2} could be reduced by incorporating an adequate amount of an appropriate transition metal such as Fe, Zn, Cu, Ni and V. Doping of these metals essentially lowers the band gap of anatase TiO\textsubscript{2} and reduces the recombination rate of photogenerated electron–hole pairs [30]. The effect of doping on the activity depends on many factors such as the method of doping, type and
the concentration of dopant [31, 32]. Therefore, seeking to simple and suitable methods for transition metal doping is necessary for the large-scale synthesis of metal-doped TiO$_2$ nano semiconductors. Several researchers have shown that the energy conversion efficiencies of photovoltaic devices are critically dependent on the morphologies of the semiconducting nanomaterials [33]. In the present work, the copper-doped TiO$_2$ semiconducting nanoparticles were prepared by sol-gel process using distillation method. The ultimate aim of the present study was to find out the effect of Cu doping on the structure and optical properties of anatase TiO$_2$. The slight increase in the crystallite size upon Cu doping leads to a small decrease in the band gap of anatase TiO$_2$.

6. RESULTS AND DISCUSSIONS

6.1 XRD ANALYSIS

X-ray diffraction pattern gives information about crystalline structure, lattice strain and phase transformation of TiO$_2$. As can be seen from Fig. 6.1a, all the diffraction peaks in both the samples are assigned well to the tetragonal anatase crystalline phase of TiO$_2$ according to the standard XRD patterns (JCPDS card no. 86-1157). No impurity peaks have been observed, which indicates high purity TiO$_2$ sample. Although there is no change in the XRD pattern for anatase Cu-TiO$_2$ (Fig. 6.1b), the peak positions are shifted slightly towards the left as compared with that of pure anatase TiO$_2$. This shift in peak position exemplifies the incorporation of Cu into the lattice of anatase TiO$_2$. The peak intensity of anatase Cu-TiO$_2$ nanoparticles have been increased with a decrease in the full width half maximum (FWHM). This may be due to the fact that Cu doping alters the crystallinity but not the crystal structure of anatase Cu-TiO$_2$. The intensities of (1 0 3), (0 0 4) and (1 1 2) planes have been increased with Cu doping. This can be seen from the
enlarged view of the inset plot of Fig. 6.1. Cu doping leads to an expansion of lattice planes depending on the doping level [11, 23]. The lattice parameters for anatase TiO₂ and Cu-TiO₂ nanoparticles were calculated from the XRD data. The lattice constants \( a \) and \( c \) for undoped TiO₂ were found to be 0.37983 nm and 0.94756 nm, respectively. However, for Cu-TiO₂ the lattice constant \( a \) has been decreased to 0.37769 nm and \( c \) has been increased to 0.94895 nm (JCPDS card no. 86-1157). This revealed that, there is a perceptible change in lattice parameter \( a \), but an apparent increase in lattice parameter \( c \). This indicated clearly that the Cu doping distorted the \( c \) axis of TiO₂, which in turn caused the expansion of unit cell volume [34]. The reason for this increment is attributed to the substitution of Ti atoms by Cu atoms. This was confirmed by EDAX measurement. Also the addition of Cu has increased the crystallinity of the anatase TiO₂ nanoparticles. It can be seen that the addition of Cu into TiO₂ lattice resulted in an intensity enhancement and narrowing of \((1 0 1)\) peak, indicating a progressive growth of crystallites [35]. The slight increase in the crystallite size on doping with Cu leads to a small decrease in the surface area [36]. Moreover, no additional peaks of Cu were detected in sample (Fig. 6.1b), which suggests that the amount of Cu was very low and possibly Cu species were incorporated in anatase TiO₂ crystal [32, 36, 37, 42].

6.2 FESEM/EDAX ANALYSIS

The surface structure of pure anatase TiO₂ and Cu-TiO₂ nanoparticles were studied by FESEM (Fig. 6.2a and 6.2b). It can be seen that the crystallite size of pure anatase TiO₂ and Cu-TiO₂ semiconducting nanoparticles annealed at 550 °C were found to be 9 and 52 nm respectively. The pure anatase TiO₂ (Fig. 6.2a) and Cu-TiO₂ (Fig. 6.2b) nanoparticles exhibited uniform morphologies.
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Figure 6.1: XRD spectra of powder sample prepared at 550 °C (a) pure TiO$_2$, (b) Cu doped TiO$_2$. Inset shows the enlarge view of (a) and (b).

However, some aggregations were found in anatase Cu-TiO$_2$ nanoparticles. The rate of particle aggregation is a major factor that controls the morphology and crystalline structure of the final product [38]. It is well known that the Cu species dispersed in the anatase TiO$_2$ gel will aggregate during the annealing process [39]. This is because of the increase in supersaturation of the reaction products, which accelerates the crystal core forming reaction within a short time. Under these conditions the controlling step of the reaction is transferred from grain growth to crystal nucleus formation. At 550 °C, the phenomenon of "nuclear-aggregation" caused by the rapid formation of crystal nucleus resulted in the formation of aggregates among the crystal nucleus. This results indicated that the anatase Cu-TiO$_2$ powders have bigger crystallite size [1, 38]. The EDAX spectra
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of Cu-TiO₂ (Fig. 6.2c) show an evidence for the presence of copper, titanium and oxygen in the pure form. When Cu concentration is low, most of the Cu species are located at a depth exceeding the escape length of Cu-Kα radiation within the particles. Meanwhile the amount of aggregated Cu species is too small to be detected by the EDAX instrument [39, 40]. Nano-TiO₂ shows a peak around 0.2 keV and another intense peak appears at 4.5 keV. The intense peak is assigned to the balk TiO₂ and the less intense peak to the surface TiO₂. The peaks due to oxygen and copper were clearly distinct at 0.4 keV and 0.9 KeV, respectively. The presence of Cu is confirmed from the EDAX analysis (Fig. 6.2c). It can be correlated from the results of XRD that the Cu is successfully doped in the TiO₂ nanoparticles.

![Figure 6.2: FESEM images of pure samples as-prepared at 550 °C (a) pure TiO₂, (b) Cu doped TiO₂ (c) EDAX spectra of Anatase Cu doped TiO₂.](image)

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6.3 HRTEM ANALYSIS

The surface morphology evaluated by HRTEM studies have clearly indicated a significant change in crystalline growth that effectively leads to an increase in the crystallite size (Fig. 6.3). In addition, more uniform and homogeneous distribution has been obtained by doping Cu into the anatase TiO$_2$ nanoparticles. The presence of small amount of Cu ions influences the growth process resulting in elongated nanocrystals. The HRTEM image shows that the sample consists of plenty of irregular particles. Although the nanoparticles are slightly agglomerated, their grain boundaries are clearly distinguishable. Crystal images of the product were clearly observed, indicating that these nanoparticles composed of fused nanoparticles had high crystallinity. Since the particles are aligned perfectly, the orientations of crystals of fused nanoparticles were completely aligned. Fig. 6.3b also reveals that the doping of Cu does not leave any change in the shape of the nanoparticles.

![HRTEM images](image_url)

**Figure 6.3:** HRTEM images of pure samples as-prepared at 550 °C a) pure TiO$_2$, (b) Cu doped TiO$_2$. 
6.4 UV-VIS SPECTRAL STUDIES

UV-Vis spectrometry is the most reported technique used to evaluate the optical properties and to examine the doping effects on the host metal oxide matrix [1]. Doping of impurity induces substantial modification of electrical and optical properties in the semiconducting materials. By doping with different impurities, the structural and optical properties of anatase TiO₂ can be modified. The optical absorbance spectra of anatase TiO₂ and Cu-TiO₂ particles were measured in the region of 300–600 nm (Fig. 6.4). It can be found that the absorption edge of TiO₂ is shifted from 330 to 344 nm after Cu doping. The shift in the optical band gap edge towards longer wavelength region may be pointed to the decreasing band gap. The presence of Cu in the doped sample has been confirmed by EDAX technique, which suggested relatively high dispersion of Cu component in the samples [25, 39, 43]. The red shift caused by the metal dopants in the Cu-TiO₂ (Fig. 6.4b) could be attributed to the charge transfer between 4d orbital electrons of Cu ions and the conduction or valence band of TiO₂, which could lead to the reduction in the number of electron–hole pairs and increase in the photon efficiency. Consequently, doping with Cu might be more favorable for the separation of photo-induced electron–hole pairs. The absorbance peak of the doped sample becomes broader with a lower energy shift (Fig. 6.4b) and broadening to the lower energy region takes place most considerably for the Cu doping. It gives a direct evidence for the decrease of band gap (Eₔ) [41]. An increase in Cu doping beyond 0.1 wt % shows a shift toward longer wavelength region (red shift) due to an increase in the crystallite size. The band gap is important in preventing the electron-hole recombination and ultimately to enhance the photo efficiency [42, 32]. The optical band gap energy (Eₔ) of the samples is calculated using the formula, $E_g = \frac{hc}{\lambda}$,
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Fig. 4 shows the UV-Vis absorption spectra of anatase TiO$_2$ and Cu-TiO$_2$ nanoparticles. The corresponding band gaps were found to be 3.75 eV and 3.58 eV respectively. Broad absorption peak between 310 - 440 nm for anatase Cu-TiO$_2$ (3.58 eV) is attributed to the presence of Cu species. These absorption bands could be due to the absorption of metallic Cu which led to a reduction of its inherent band gap from 3.75 eV to 3.58 eV. For Cu-TiO$_2$ nanoparticles, only one peak was observed in the region (310 - 440 nm), which suggested relatively high dispersion of Cu component in the sample and good contact with anatase TiO$_2$ particles [24, 26, 27]. This indicated that the band gap energy in Cu-TiO$_2$ is lower than that of pure anatase TiO$_2$ [45]. Since the radius of Cu$^{2+}$ ion is larger than that of Ti$^{4+}$ ion, introduction of Cu$^{2+}$ has distorted the crystal lattice and consequently created disorderness and hence responsible for the optical band gap reduction [46].

![Figure 6.4: UV-Vis spectra of as-prepared at 550 °C (a) pure TiO$_2$, (b) Cu doped TiO$_2$](image)
6.5 FTIR ANALYSIS

FT-IR spectra of anatase TiO$_2$ and Cu-TiO$_2$ nanoparticles are shown in Fig. 6.5. As can be seen in Fig. 6.5 both samples had similar spectra although their peak heights were different at different wave numbers. The absorption peak at 3450 cm$^{-1}$ is attributed to the OH stretching vibration and its corresponding bending vibration occurs at 1600 cm$^{-1}$, due to the presence of adsorbed water molecules. Additionally, in Cu-TiO$_2$ catalyst the OH group concentration in the 3500 - 3000 cm$^{-1}$ region are slightly reduced [27, 42]. The band around 480 - 700 cm$^{-1}$ in both the spectra are attributed to Ti-O stretching vibration (Fig. 6.5a and 6.5b), which shifts to the higher frequency after the addition of Cu. The IR spectra of the modified samples did not show any band corresponding to Cu. It might be due to the reason that the content of incorporated metal is slight; therefore the intensity of the band related to Cu-O-Ti is very low. In addition, the bending frequency of Cu-O might have overlapped with Ti-O bending frequency below 1000 cm$^{-1}$ and therefore the overlapping peaks are observable in this region [31, 42]. These results revealed that the dopant was successfully incorporated into the lattice of anatase TiO$_2$. The result of FT-IR analysis substantiated the XRD results.

6.6 XPS ANALYSIS

X-ray photoelectron spectroscopy is a non-destructive technique to measure the surface chemistry of any inorganic compounds, metal alloys, semiconductors, polymers, or catalysts. Fig. 6.6 represents typical XPS spectrum which gives valuable information on the surface composition. XPS is a very useful technique especially for multi component samples such as the doped metal oxides. The Cu 2p$_{3/2}$ and Cu 2p$_{1/2}$ spin orbital splitting photoelectrons were located at binding energies of 934.4 and 954.0 eV, respectively.
The relative binding energies for Ti 2p$_{3/2}$ and Ti 2p$_{1/2}$ in TiO$_2$ nanoparticles were found to be 459.4 and 465.0 eV (Fig. 6.6A). These bands were assigned to a typical Ti$^{4+}$. However, upon introducing Cu into the TiO$_2$ lattice, the binding energy for both the Ti 2p$_{3/2}$ and Ti 2p$_{1/2}$ shifted towards higher energy, as shown in Fig. 6.6A. According to the shift of XPS peaks (Fig. 6A), the Ti 2p$_{3/2}$ and Ti 2p$_{1/2}$ binding energy of the Cu-TiO$_2$ (460.0 eV) sample is increased comparing with that of pure TiO$_2$ (459.4 eV) [24] as shown in Fig. 6.6A. This binding energy shift might be attributed to the presence of the second metal atom in the TiO$_2$ lattice and the change in the coordination number of the metal by the formation of a Cu-O-Ti bond. The presence of the second metal will change the electronic environment as well as the binding energy of the system. This analysis indicates that the Cu atom was substituted for some Ti atoms in the TiO$_2$ lattice. The surface Cu amount was very low. When scanning the Ti 2p and Cu 3d XPS regions,
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significant differences are observed at the binding energies between the pure TiO₂ and the modified system. The small shift of binding energy of the Ti 2p₃/₂ and Ti 2p₁/₂ peak is attributed to the change of the valence state of Ti⁴⁺. The 1s XPS spectra of oxygen also shows significant changes upon Cu incorporation, with the peak shifted to a higher binding energy than the pure oxygen peak in TiO₂. It gives a good indication of Cu incorporated into the crystal lattices of TiO₂ [11, 20, 24].

Figure 6.6: XPS spectra of as prepared samples TiO₂ and Cu-TiO₂ (A) Ti 2p spectra of TiO₂ (peak a) and Cu-TiO₂ (peak b), (B) Cu 2p of Cu-TiO₂, (C) O 1s of TiO₂ (peak a) and Cu-TiO₂ (peak b), (D) XPS survey spectrum of TiO₂ (peak a) and Cu-TiO₂ (peak b).
6.7 UV-Vis spectral studies of Dye-Sensitized Cu-TiO₂ films

Figure 6.7: The optical absorption spectra of anatase Cu-TiO₂ films before and after sensitization with Eosin B (EB).

Figure 6.7 shows the optical absorption spectra of anatase Cu-TiO₂ films before and after sensitization with Eosin B (EB), Fast Sulphon Black F (Fig. 6.8) and Solochrome Black T (Fig. 6.9) at different concentration. It is found that the λ_max for Eosin B is about 532 nm while those of Fast Sulphon Black F and Solochrome Black T are about 617 and 501 nm, respectively. The results are shown in Fig. 6.7a. No absorption peak for the TiO₂ is observable above 340 nm. In general, auxochromes influence the intensity of the dye, but they can also provide a site by which the dye can chemically bond to the Cu-TiO₂. To understand the efficiency enhancement of DSSCs with Cu-TiO₂ nanostructures, the optical absorbance of the samples after being dye-sensitized for 30 min has been measured. It can be seen from Fig. 6 that all of these nanostructure show a wide
absorbance band in the visible region (370 - 650 nm). It can be believed that the increase in photo absorption is the main reason for the improved conversion efficiency. The efficiency of cell sensitized by the Eosin B was significantly higher than that sensitized by Fast Sulphon Black F and Solochrome Black T. This is due to a broader range of the absorption by the dye on Cu-TiO\(_2\) and the higher interaction between those. Nanosized semiconductor particles exhibit pronounced size effects on electronic structure and related optical properties. As the size of the semiconductor crystal changes, different facets and surface steps may be created. These different surface species induced by different diameters are expected to have varied electron injection capabilities. Small TiO\(_2\) nanoparticles have a high surface area/volume ratio, which gives rise to a lot of defects. Different diametered TiO\(_2\) may have different defect densities on the surface. Note that the TiO\(_2\) quantum size effect, that is, the exciton confinement effect, appears only at a diameter of less than 1.5 nm, so the quantum effect is not likely in the present case because the diameter of Cu-TiO\(_2\) is larger than 18 nm. Dye aggregation is not good for achieving high performance of DSSCs [47, 48]. In general, aggregation can change the nature of the excited state of organic molecules. The optical spectrum of Cu-TiO\(_2\) and dye-sensitized TiO\(_2\) is recorded and shown in Fig. 6.6. It can be seen from figure that before sensitization, the Cu-TiO\(_2\) shows an absorption band extending from 320 to 370 nm. Further, sensitization with Eosin B imparts widening of spectral response covering complete visible region (370 - 600 nm) and extrapolation of the peak absorption edge results a substantial decrease in band gap down to 2.28 eV (542.2 nm) [48]. It does mean that sensitization with Eosin B imparts an enhancement in photoconductivity and broadening of photoaction spectrum towards low photon energy. The absorption spectra
of Eosin B sensitized Cu-TiO₂ exhibit obvious red shift (Fig. 6.7d) in comparison with that of other two dyes (Fig. 6.8 and 6.9). The "head-to-tail" transition dipole arrangement on Cu-TiO₂, which is distinguished by a sharp intense red-shifted absorption band. In case of Eosin B dye, an absorption peak of photo anode is broader than that of the pure dye solution (Fig. 6.7c). The Increase of absorption spectrum from 370 to 600 nm may be due to the change of energy level for the Cu-TiO₂ [48-50] and the shift is due to the change in the energy of the lowest unoccupied molecular orbital (LUMO) of the dye. Upon photoexcitation of the dye by visible light, electrons are excited from the ground state of the dye to the excited state, and then electron injection occurs from the excited dye into the conduction band of the semiconductor film. The injected electrons relax to the bottom of the conduction band, and then the electrons are trapped in a defect site below the conduction band, followed by diffusion of electrons in the film toward the transparent conductive electrode. Electron injection is an important primary process in dye-sensitized solar cells. To achieve a high quantum yield for electron injection from the excited state of the dye, the dye ideally needs to be in intimate contact with the semiconductor surface, which indicates a close overlap of the ligand orbitals and the titanium 3d orbitals. The choice of suitable sensitizer dye energetic is essential to achieve suitable matching to the metal oxide. The excited-state oxidation potential must be sufficiently negative to achieve efficient electron injection into the Cu-TiO₂ conduction band. Typical rates of dye excited-state decay to ground state are in the range 10⁷-10¹⁰ s⁻¹. The rate of electron injection depends on the electronic coupling between the dye excited-state LUMO orbital and accepting states in the Cu-TiO₂. However it should be noted that fast electron injection dynamics require both strong electronic coupling of
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the dye LUMO orbital to the metal oxide conduction-band states. Several groups studying the transient absorption of the excited and oxidized states of the dye in the visible and near-IR have reported the evidence for ultrafast charge separation and electron injection. In the DSSCs, the sensitizer is strongly adsorbed on the semiconductor surface, resulting in a very large electronic coupling between the \( \pi^* \) orbital of the excited state of the sensitizer and the conduction band of TiO\(_2\), which consists of the unoccupied 3d orbital of Ti\(^{4+}\). In addition, the conduction band of the semiconductor has a continuous and relatively large density-of-states. Thus, electron injection from the photosensitizer to the semiconductor occurs at a higher rate. A broad metal-to-ligand charge transfer (MLCT) absorption is observed at lower energy region in Fig. 6.7d (370-650 nm). When the dyes were adsorbed on the Cu-TiO\(_2\) film, a red shift in the peak was observed. The average value of the shift is about 180 nm, which means that the interaction between the dyes and the cationic Ti surface was observed. The interaction was formed through a chemical bond, the Cu-O-Ti bond as discussed in the literature. In the case of Eosin B, an absorption peak of photoanode is broader than that of the dye solution, with a shift to a higher wavelength. The difference in the absorption peak is due to the binding of dye on the oxide surface. It is generally accepted that the chemical adsorption of these dyes takes place due to condensation of alcoholic-bound protons with the hydroxyl groups on the surface of nanostructured Cu-TiO\(_2\). Therefore, this chemical attachment affects the energy levels of the highest occupied molecular level (HOMO) and the lowest unoccupied molecular level (LUMO) of these dyes, which eventually affects the band gap of these materials and a shift in the absorption peak of the absorption spectra. The shift in the absorption maxima in the samples could be considered as an evidence for the
attachment of these molecules on the surface of Cu-TiO\(_2\) for fast electron injection. This difference, however, was not found in the case of other dyes (Fig. 6.8 and 6.9). Hence, the Eosin B dye significantly improved the optical absorption ability of the Cu-TiO\(_2\) semiconductor electrode [51-53]. The excitation, electron injection and dye regeneration is represented in Fig. 6.10. The excitation, electron injection and dye regeneration can be expressed as follows.

\[
Dye + h\nu \rightarrow Dye^* \quad (1)
\]

\[
Dye^* + e^- \rightarrow Dye^- + e^- \quad (2)
\]

\[
Dye^- + e^- \rightarrow Dye \quad (3)
\]

Figure 6.8: The optical absorption spectra of anatase Cu-TiO\(_2\) films before and after sensitized with Fast Sulphon Black F (FSF).
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Figure 6.9: The optical absorption spectra of anatase Cu-TiO₂ films before and after sensitized with Solochrome Black T (SBT)

Figure 6.10: Electron transport in nanocrystalline oxide electrodes, in which photo excited electrons are injected from the dye to the conduction band (CB).
6.8 CONCLUSION

➢ The synthesized anatase semiconducting nanoparticles were shown to possess high crystallinity and size uniformity, all of which are beneficial properties for improved photovoltaic performance.

➢ The results revealed that the Cu doped semiconducting nanoparticles have bigger crystallite size than that of pure anatase TiO₂. The particle size also affects the electron transport and recombination. By controlling the particle size of the semiconductor, the photophysical and photochemical properties can be changed.

➢ The absorption spectrum of anatase Cu-TiO₂ shows a shift in the absorption edge towards longer wavelength region, which was likely caused by the increase in crystallite size, crystallinity and decrease in band gap energy.

➢ Thus the Cu doping can be used as a method to control the optical and structural properties of anatase TiO₂ nanoparticles. XPS showed, the excessive oxygen vacancies and Cu species can become the recombination centers for photoinduced electrons and holes.

➢ The Eosin B dye on Cu-TiO₂ particles imparts widening of Spectral response covering complete visible region through J-type aggregation.
REFERENCES