Structural and Raman spectroscopic characterization of C-TiO2 nanotubes synthesized by a template-assisted sol-gel technique.

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INTRODUCTION

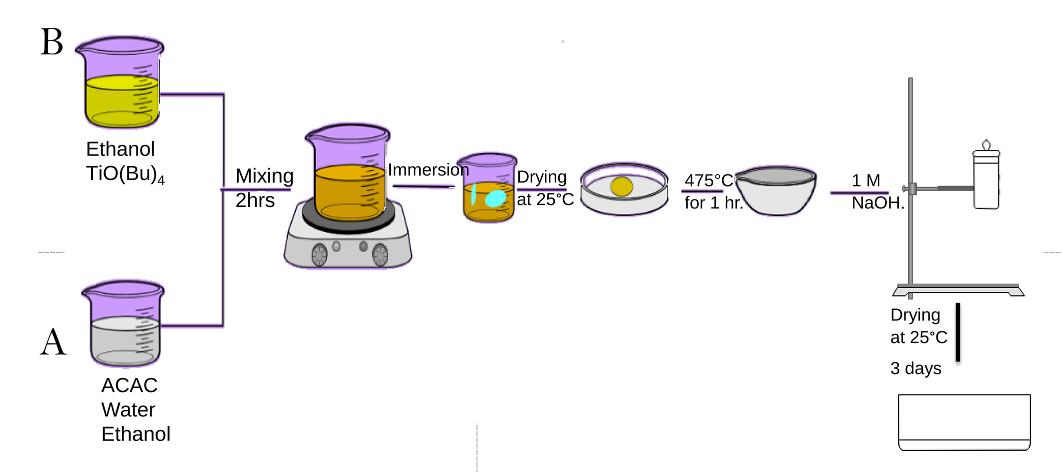
- Continuous breakthroughs in synthesis of TiO2 nanomaterials have brought applications such as photo-catalysis, photovoltaics (PV) , etc.
- In PV TiO2 has been used as a semiconductor material for dye sensitized solar cells (DSSC).
- DSSC photo-electrode utilizes TiO2 nanoparticles, resulting to low efficient collection of photo-generated electrons due to electron trapping sites and lack of response in the UV vis region.
- Carbon doped nanotubes are a promising solution to spectral loses .

OBJECTIVES

- Synthesis of undoped and carbon doped TiO2 sol-gel precursor solutions.
- Synthesis of undoped and carbon doped TiO2 nanotubes using AAM templates.
- Structural/ morphological, elemental and optical characterization of undoped and carbon doped TiO2 nanotubes using SEM, SEM-EDX, FTIR, XRD, and Confocal Raman spectroscopy

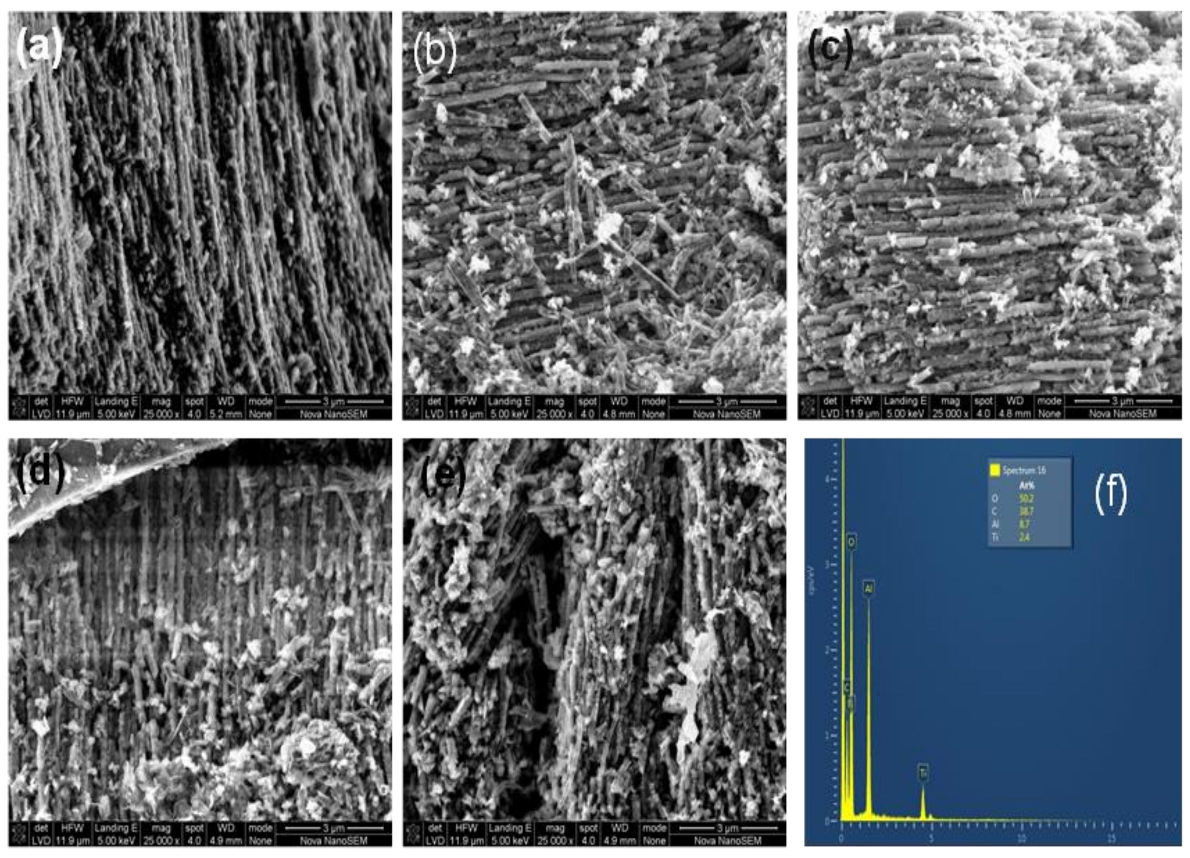
Template-assisted sol-gel technique

$$Ti[O(CH_2)_3(CH_3)]_{4(aq)} + 2H_2O_{(aq)} \xrightarrow{Heat} TiO_{2(s)} + 4C_4H_9OH_{9(g)}$$



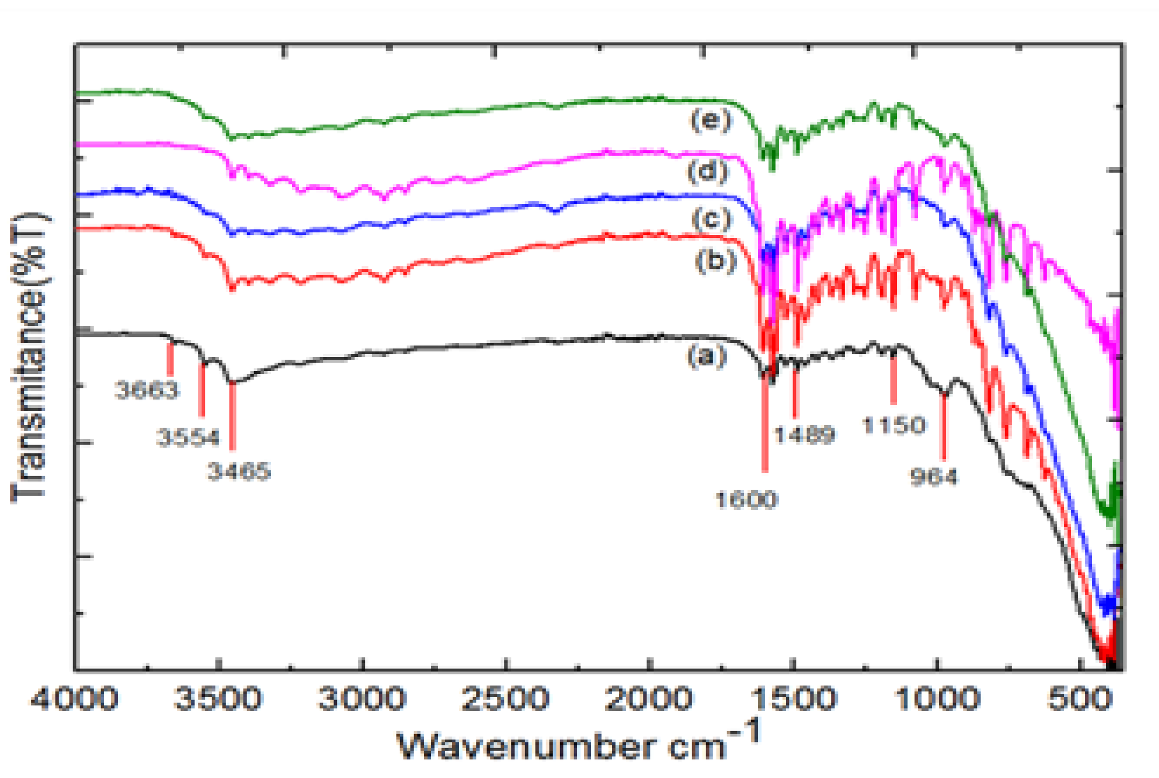
RESULTS AND DISCUSSION

Scanning Electron Microscopy



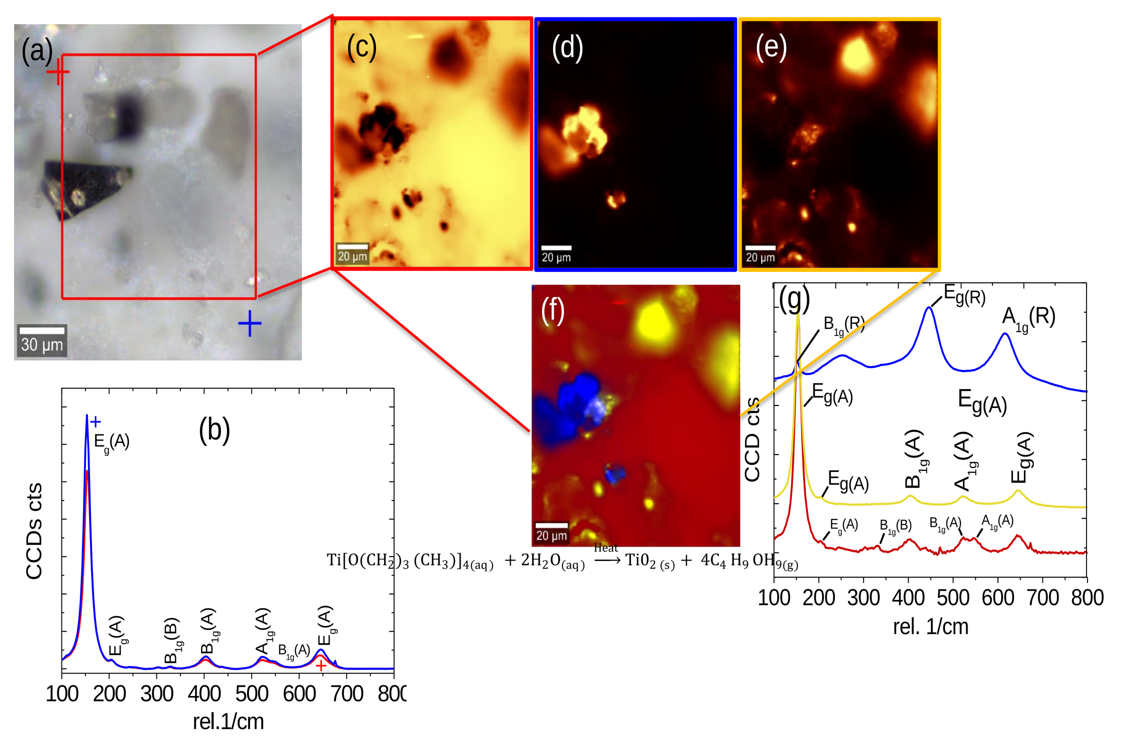
- SEM revealed that the un-doped TNTs are closely-packed in the form of bundles.
- Upon doping the TNTs become loosely packed
- EDX shows presence of Ti and O in the spectra confirms the successful hydrolysis of (Ti (OBu) 4)

Flourier Transform Infrared Spectroscopy



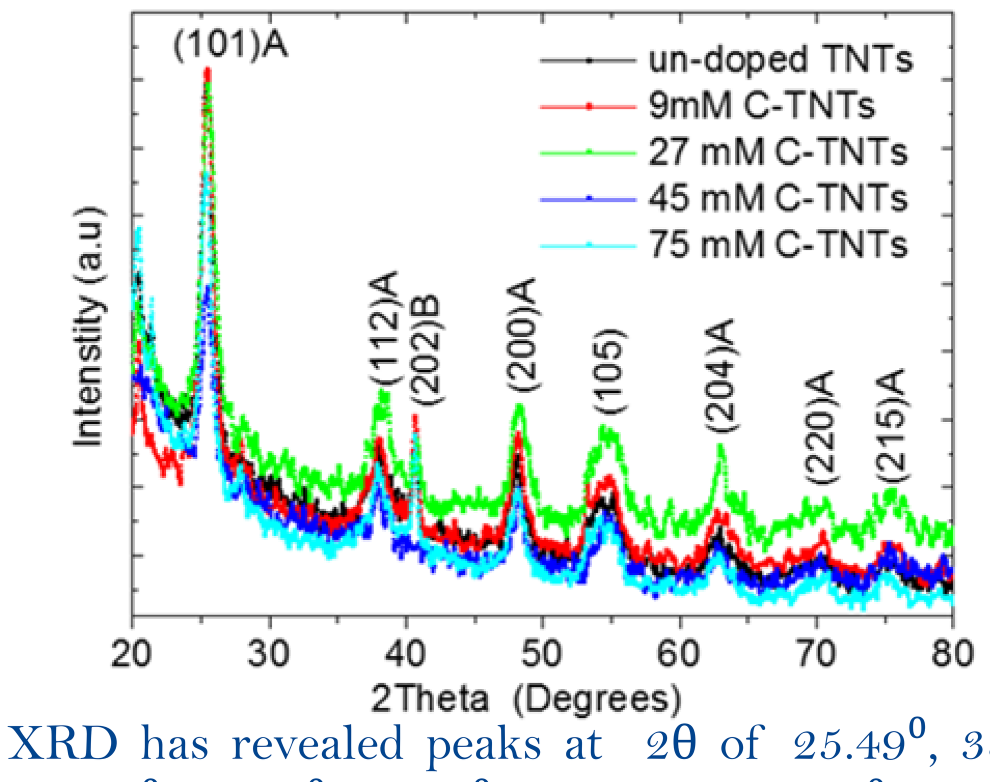
- FTIR Reveals a characteristic vibrational mode in the region 580-660 cm-1 which is associated with Ti-O bonds in TiO2 for all the synthesized samples

Raman Spectroscopy



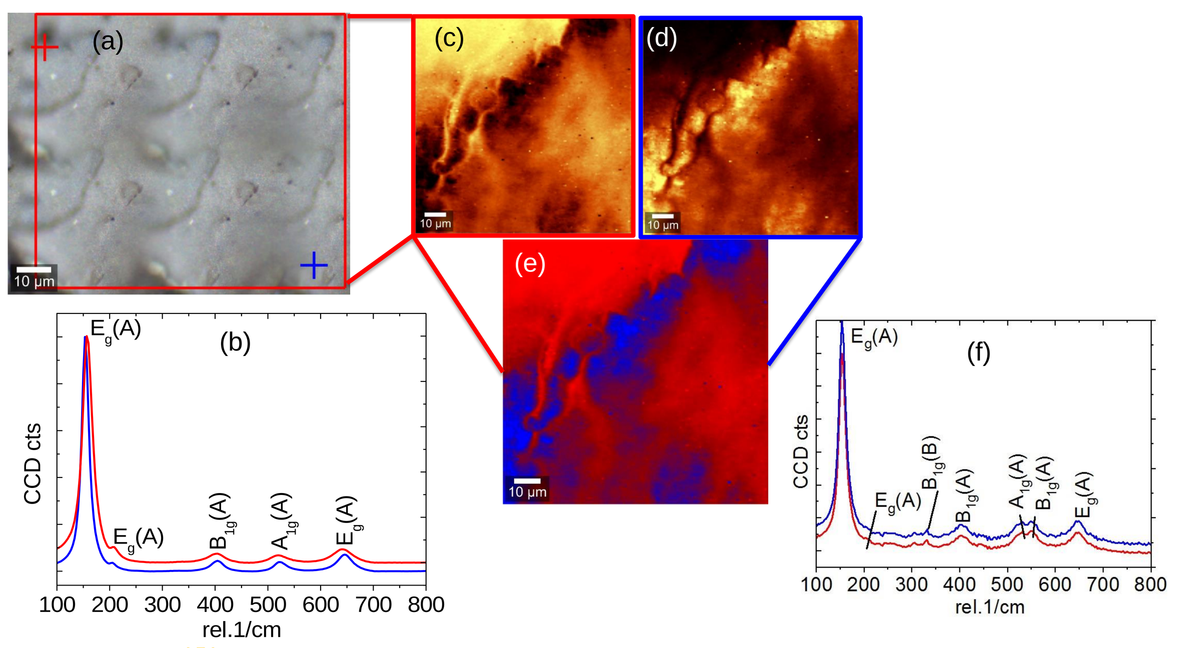
- Raman revealed the distribution of Anatase, Brookite and Rutile phases of TiO2 in undoped samples.
- Anatase, (Eg1) at 209.08 cm-1 and 644.59 cm-1(Eg3).

X-Ray Diffraction



- XRD has revealed peaks at 2θ of 25.49^0, 38.11^0, 48.14 ^{0}, 54.58^0, 63.00^0, 70.33 and 75.66^0 (Anatase JCPDS No. 21- 1272).
- Brookite peak at 40.6 ^{0}(JCPDS No. 29-1360).
- The lattice constant increases with the increase in carbon concentration along the maximum elongation of c-axis is about 0.687 Å, from 9.143 Å (un-doped TNTs) to 9.830 Å (75 mM TNTs)

Raman Spectroscopy



- The Eg1 mode of the undoped as TNTs has shown a red shift of Δ9.78 cm-1 relative to the bulk.
- This was attributed to decrease in particle size, presence of Brookite and phonon confinement.
- Upon doping the 9 mM C-TNTs, 27 mM TNTs and 75 mM TNTs have shown a red shift of Δ0.09 cm-1, Δ1.39 cm-1 and 1.81 cm-1 respectively, suggesting incorporation of carbon in the TiO2 compared to the undoped TiO2.

Conclusion

- SEM micrographs show changes in morphology of samples as the dopant level increases.
- SEM-EDX has revealed the presence to strong Ti and O signals.
- FTIR has confirmed presence of Ti and O bonds at 580-660 cm-1
- XRD has shown existence of Brookite and Anatase phase TiO2.
- Raman spectroscopy analysis confirmed the presence of Anatase and Brookite phases of TiO2 .
- The Eg1 mode of Anatase TiO2 has shown a red shift as the dopant level increase. Suggesting successful doping of TiO2, which is in mutual agreement with XRD which has shown increase in lattice parameters along c-axis as the dopant level increases

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