

Microwave synthesis of Titanium Dioxide nanotubes for use in water treatment

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INTRODUCTION

Synthesis and engineering of the nanostructured semiconductors based on metal oxides have received considerable attention due to their unique physical and chemical properties, and their potential applications in industry and technology¹. While various methods have been used to synthesise Titanium Dioxide (TiO₂) (also known as Titania) nanoparticles hydrothermal synthesis in the presence of a base solution, has proved to be an effective approach to prepare 1D nanostructures of TiO₂. This is because the method utilises minimum reagents and produces relatively purer materials. However, the main attention is directed towards controlling structure and morphology by varying synthesis conditions such as temperature, pressure and time of processing during hydrothermal processing^{2,3}.

In our study, TiO₂ nanostructures are synthesised using conventional heating and microwave-assisted hydrothermal procedure. The effects of heating on the size, shape and crystallinity of materials are studied. Microwave heating is particularly interesting because of higher energy density and shorter reaction times leading to nanoparticles that are weakly agglomerated, with high crystallinity and narrow particle size distribution. The resultant product is used as a photocatalyst in the treatment of micro-organisms in water systems.

EXPERIMENTAL

- Nanotubes were synthesised by hydrothermal procedure under autogenous pressure using either conventional heating (autoclave) or microwave irradiation
- Ag/TNT (5 wt%) nanocomposite was prepared by incipient wetness procedure
- Materials characterisation was carried out to study the properties.



Figure 1: (a) High pressure system (autoclave) and (b) Microwave reactor unit for the synthesis of titania nanotubes (TNT) in a hydrothermal process

RESULTS AND DISCUSSION

Titania nanotubes (TNTs) were successfully synthesised via hydrothermal route (Figure 1) using conventional heating and microwave irradiation. The tubes by both procedures have diameter range of 8-10 nm and length of a few microns. Tubes tended to agglomerate forming bundles. The tubes were randomly distributed and have a specific surface area of about 300 m²/g. All tubes obtained were open ended. However, microwave irradiation generated tubes with relatively smoother surface.

A total of 5% of silver was loaded to both conventionally and microwave produced TNTs and the Silver (Ag) particles were well-dispersed on microwave synthesised TNTs. The two composite materials were tested for photocatalytic activity in microbial treatment and the microwave prepared composite showed better activity in killing the micro organisms.

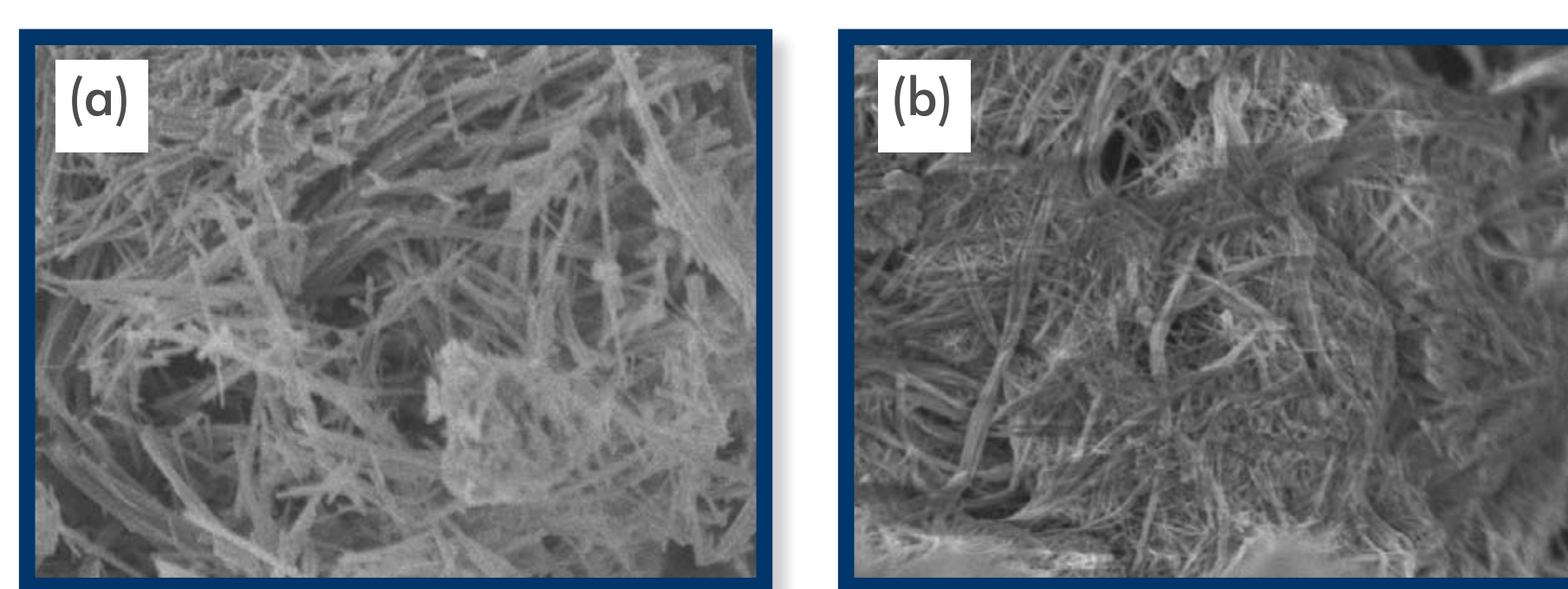


Figure 2: Scanning Electron Microscopy images of (a) TNTs produced by conventional heating and (b) TNTs produced by microwave irradiation

The use of conventional route yielded tubes primarily composed of a titanate structure, Potassium Titanate (KTiO₂(OH)) whereas microwave irradiation led to the formation of tubes predominantly comprised of anatase and rutile phases (TiO₂). The proportion of rutile is more than that of anatase phase. Microwave produced sample showed relatively better crystallinity than conventional though the particle sizes were similar.

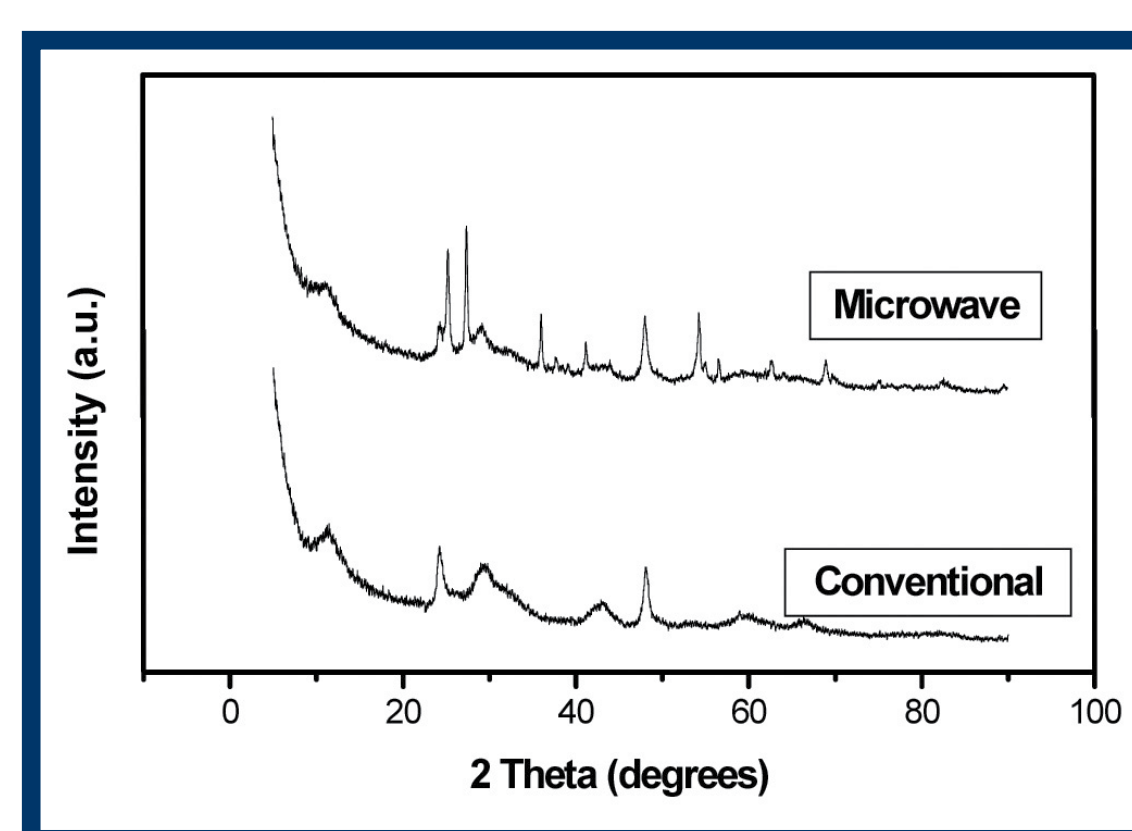


Figure 3: XRD patterns of conventionally produced TNTs and microwave produced TNTs

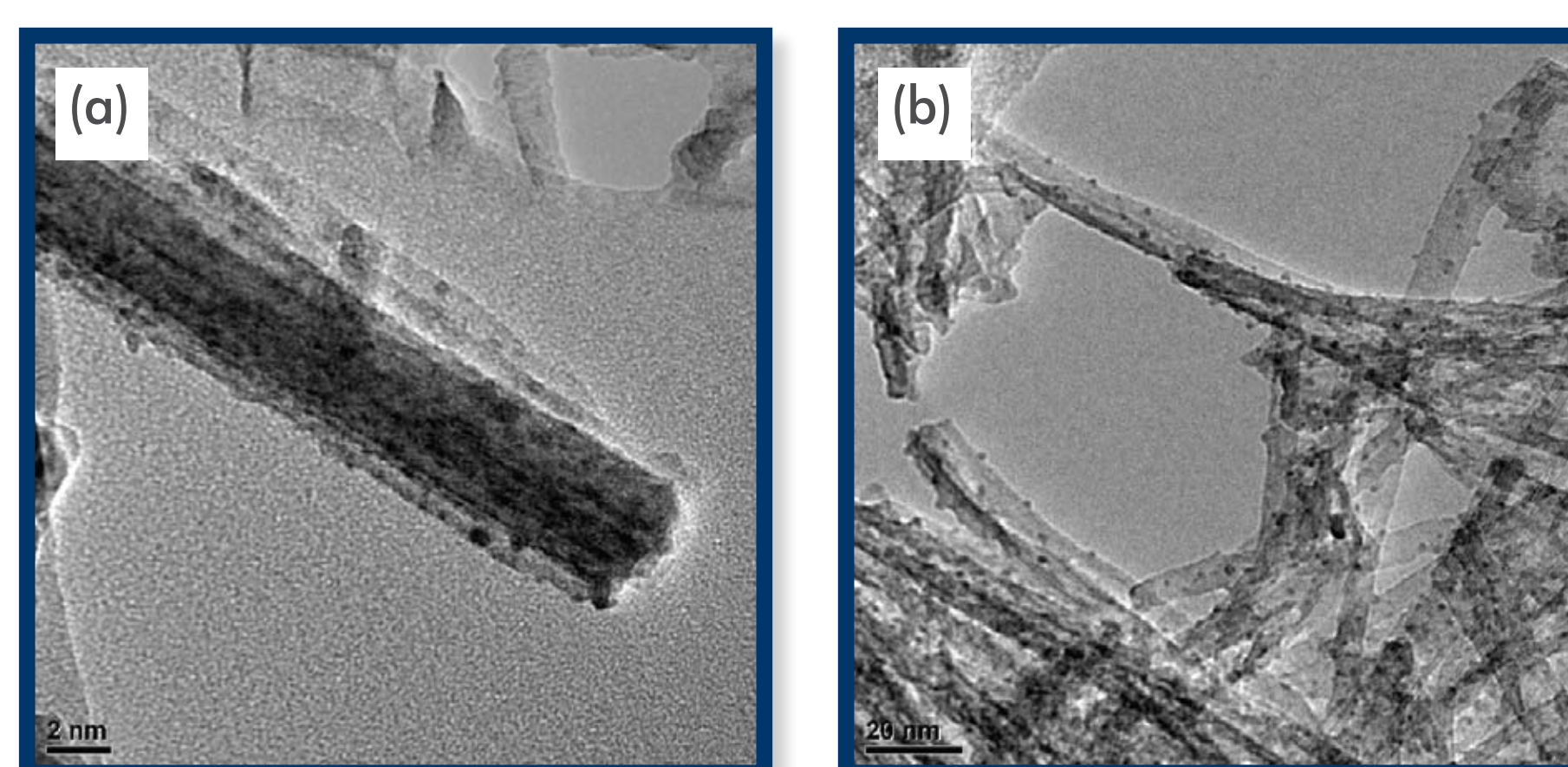


Figure 4: Transmission Electron Microscope images of Silver loaded on titania nanotubes (Ag/TNT) (5%) produced by (a) conventional route and (b) microwave irradiation

Table 1: Physical properties of Ag/TNTs produced by conventional route and microwave irradiation

Composite	Surface area (m ² /g)	Particle size (nm)
Ag/TNT (conv)	286	1.6
Ag/TNT (MW)	220	2.0

CONCLUSION

- TNTs with large specific surface areas with controlled crystal structure have been synthesised and used as catalyst support for Ag
- TNTs supported Ag catalyst showed good crystallinity and was utilised for water treatment for its photocatalytic activity.

REFERENCES

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COLLABORATIONS

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*Water treatment:
Nanotechnology's
promise and
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