Infrared (IR)

Visible Light

Ultraviolet (UV)

ARISTOTLE UNIVERSITY OF THESSALONIKI

UMCS

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Titanate nanotubes for the selective cleavage of lignin inspired *β-O-4* linkages towards value-added aromatics under visible light

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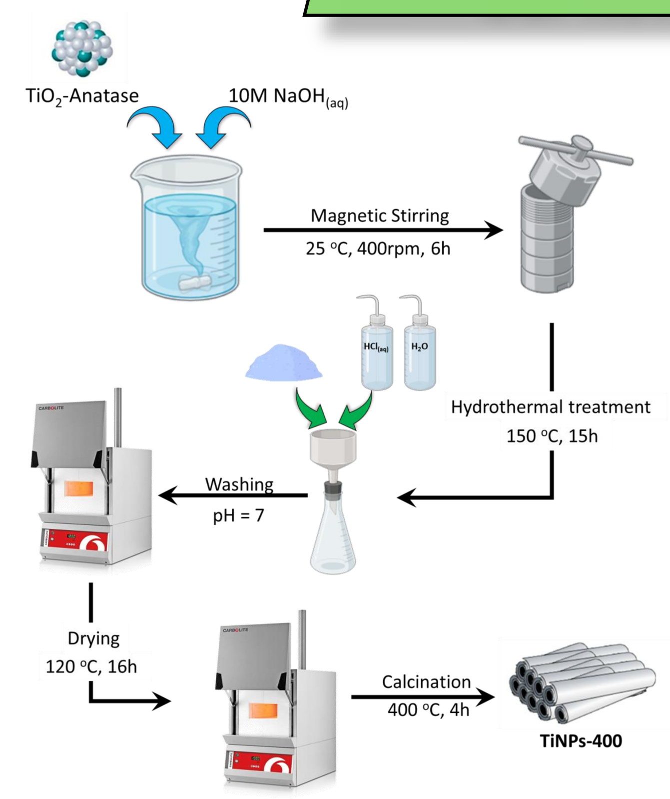
Introduction

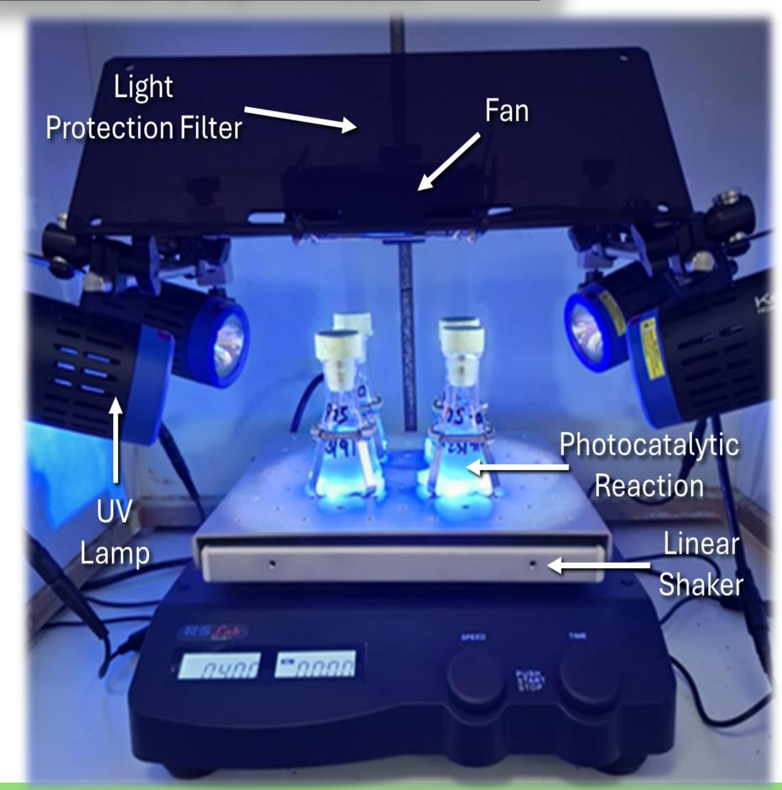
The endlessly expanding demand in chemicals, merchandises, materials, fuels as well as energy, dictates as an essential strategy to design and implement innovative and sustainable production methods, with (photo)catalytic biomass waste to value-added chemical conversion to be on its infancy.

The synthesis of a novel titanium-based nanomaterial that can selectively break lignin inspired *β-O-4* linkages (2-phenoxy-1-phenylethanol/PP-ol) towards value-added aromatics through a green technology (photocatalysis) is essential.

A step forward is the tuning of materials' physicochemical properties and their photo-reactivity, and to understand the involved mechanisms and the role of different light wavelengths.

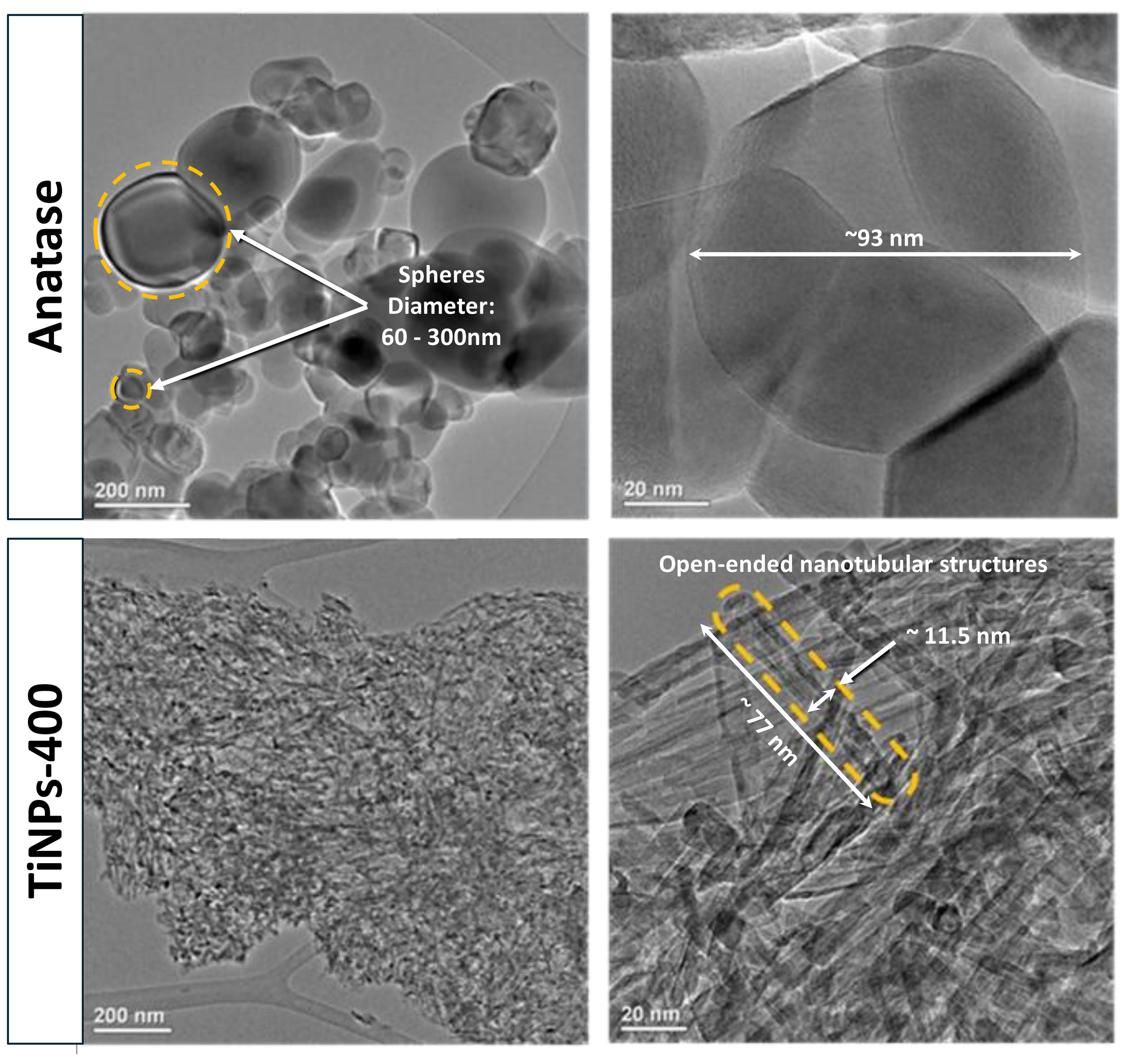
Our Novel Material's Synthesis



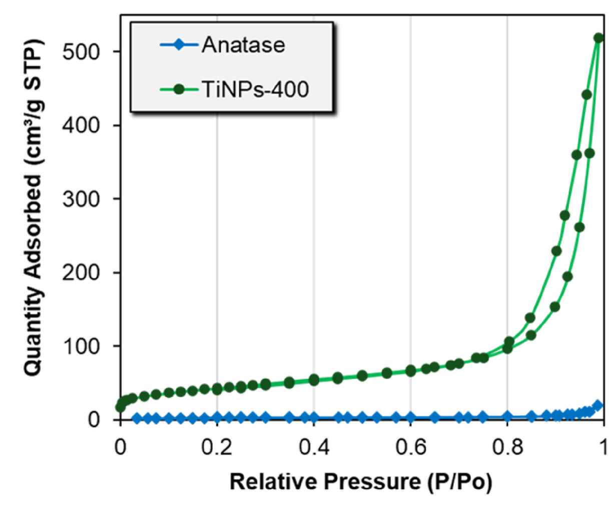


Physicochemical Characterizations

Transmission Electron Microscopy

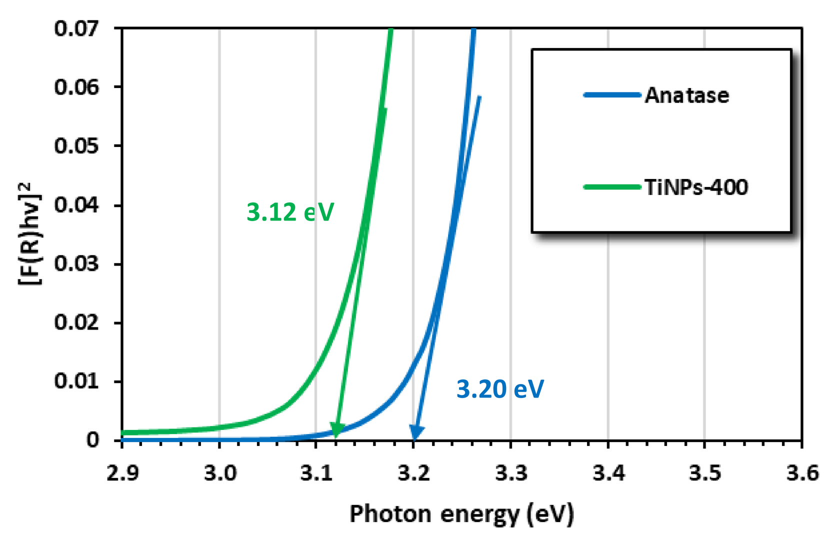


Nitrogen Adsorption

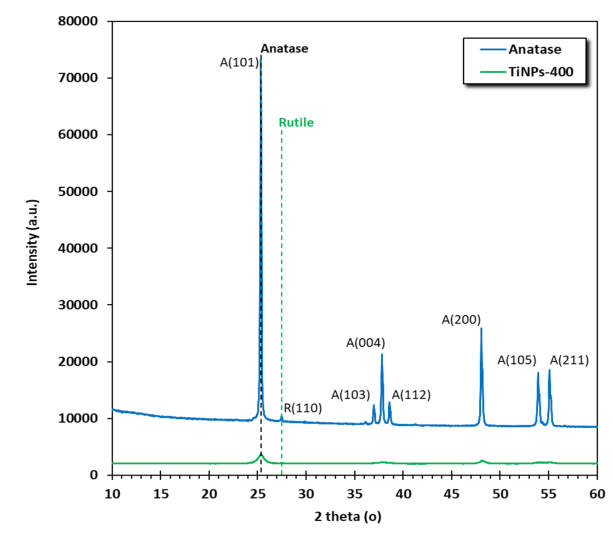


Material	BET Surface Area, m²/g	Total Pore Volume, cm³/g	Micropore Volume, cm³/g
Anatase	10	0.03	0.0
TiNPs-400	154	0.8	0.01

DR-UV-Vis



XRD



Photocatalytic β-O-4 cleavage results

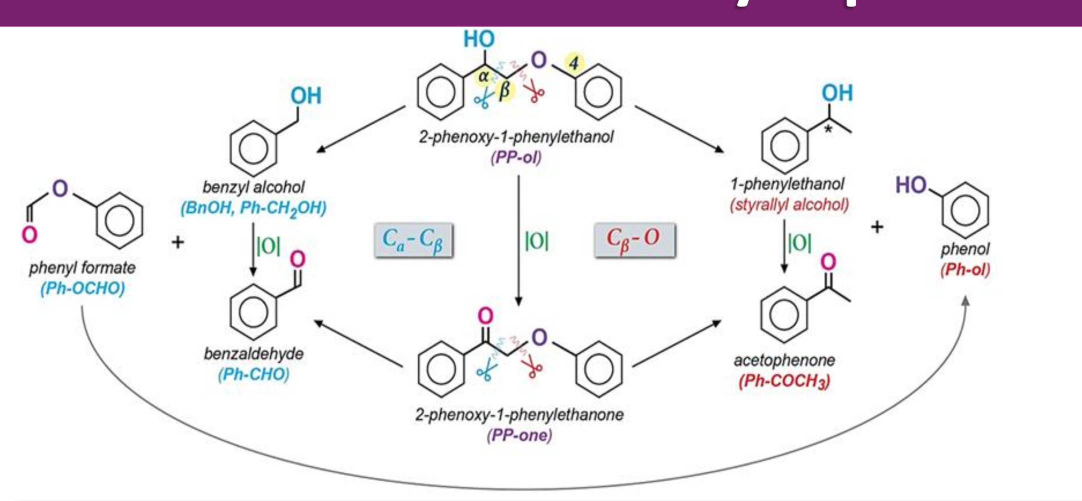


Figure 1: All the possible reaction pathways for the depolymerization and oxidation of the *β-O-4* linkage of the lignin-inspired model compound 2-phenoxy-1-phenyl-ethanol (PP-ol) towards the formation of the desired monoaromatics.

Qayyum A, et al. Selective (sono)photocatalytic cleavage of lignin-inspired β-O-4 linkages to phenolics by ultrasound derived 1-D titania nanomaterials. 2024. https://doi.org/10.1016/j.ultsonch.2024.106829.

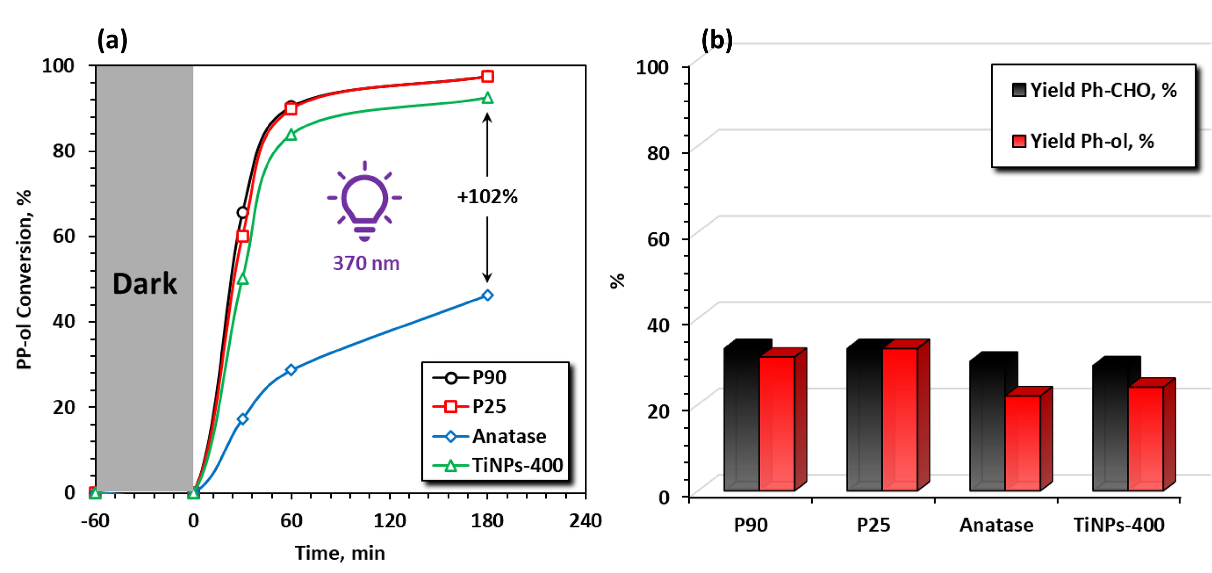


Figure 2: 2-phenoxy-1-phenylethanol (PP-ol) conversion (a); benzaldehyde (Ph-CHO) and phenol (Ph-ol) yield after **180min** UV-370nm irradiation with commercially available nano TiO_2 (P90, P25, and Anatase) and our novel synthesized material (TiNPs-400)/(T<40°C, atm Pressure, 400rpm, Cat. Rate = 1g/L).

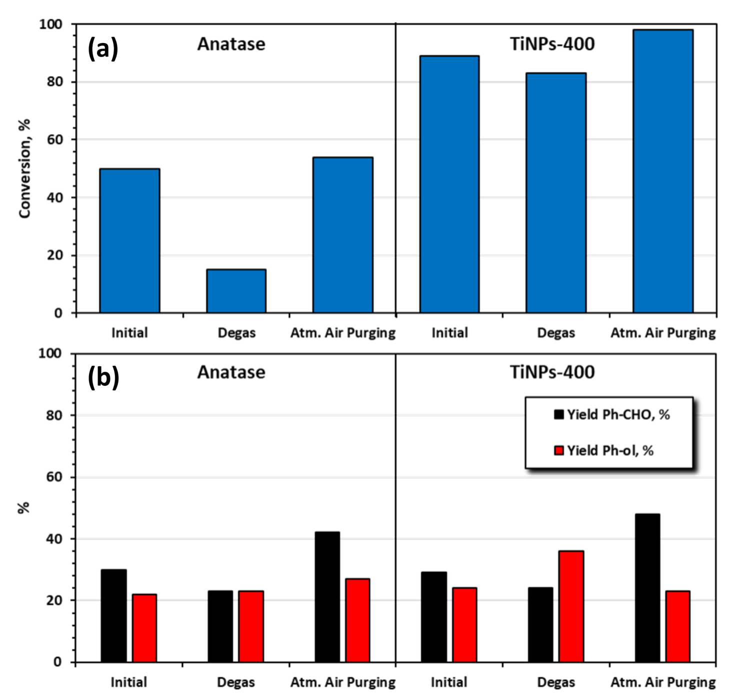


Figure 3: PP-ol conversion (a) and Ph-CHO and Ph-ol yield after **3h** UV-370nm irradiation with Anatase and TiNPs-400. Explore oxygen's role in the system/(T< 40 °C, atm Pressure, 400rpm, Cat. Rate = 1g/L).

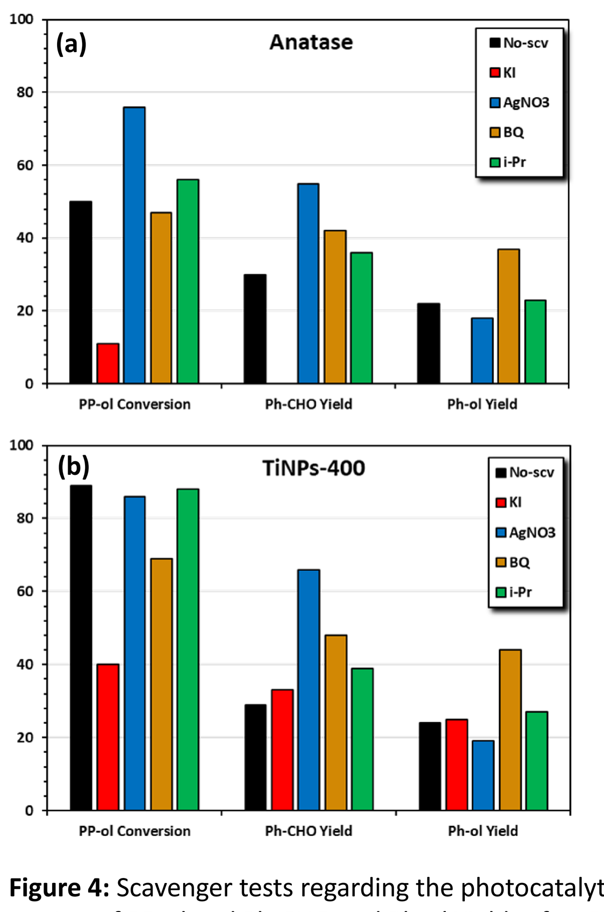


Figure 4: Scavenger tests regarding the photocatalytic conversion of PP-ol and Ph-CHO and Ph-ol Yields after **3h** UV-370nm irradiation with Anatase and TiNPs-400/(T< 40 °C, atm Pressure, 400rpm, Cat. Rate = 1g/L, Scavenger Conc. 1mM)

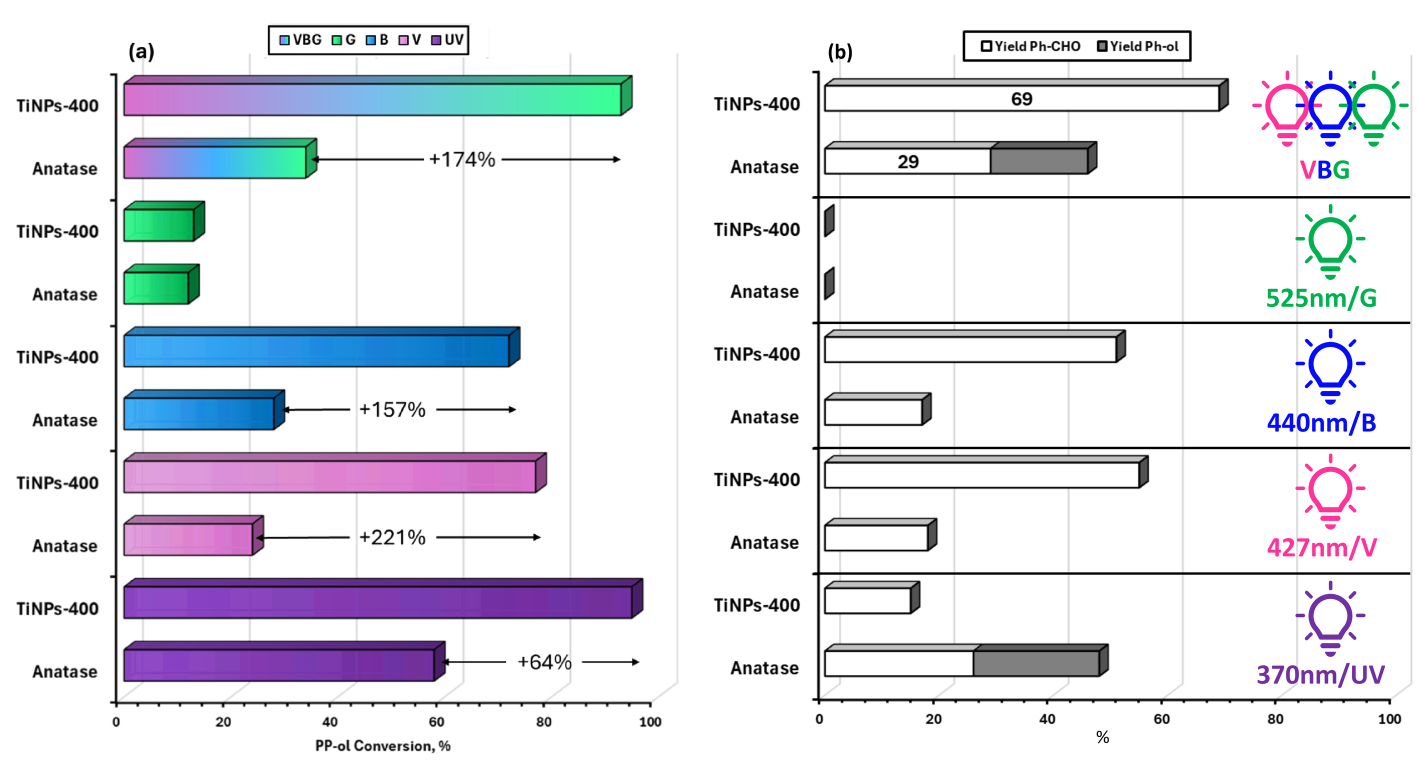


Figure 5: 2-phenoxy-1-phenylethanol (PP-ol) conversion (a), benzaldehyde (Ph-CHO) and phenol (Ph-ol) yield (b) after **24h irradiation** of various monochromatic wavelengths (UV-370nm, Violet/V-427nm, Blue/B-440nm, Green/G-525nm) and their combination (VBG) /(T< 40 °C, atm Pressure, 400rpm, Catalyst Rate = 1g/L).

Take home messages

PP-ol Conversion

- Cleavage of the C_a-C_b bond of 2-phenoxy-1-phenylethanol
- Selective production of benzaldehyde (Ph-CHO) and phenol (Ph-ol)
- Intermediate products, benzyl alcohol ($Ph-CH_2OH$) and phenyl formate (Ph-OCHO), were not detected.

Materials Properties

- Synthesis of 1D porous titanate nanotubes through the hydrothermal method.
- Compared to the commercial benchmark precursor:
  - The textural features were significantly higher (S_{BET} up to 154 m²/g)
  - The band gap was slightly decreased
  - Material with an elevated nanosized nature

Photocatalytic Performance

- TiO_2-Anatase:
  - Absence of O_2 decreases the material's performance
  - Main active species: holes (h^+)
- TiNPs-400:
  - The highest PP-ol conversion under all wavelengths or irradiations.
  - Main active species: holes (h^+)
  - Higher Ph-CHO Yield during visible light wavelength irradiations.
  - **69%** Ph-CHO Yield under visible (**VBG**) irradiation.