



The 4th International Online Conference on Nanomaterials (IOCN 2023)

Preparation of mesoporous bicrystalline N-doped TiO₂ nanomaterials for sustainable RhB degradation under sunlight

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May, 2023

Outline

- Introduction
- Methodology
- Result and discussion
- Conclusion
- Acknowledgment

Introduction

- Water is the prime necessity for *all living beings*.
- It covers **75%** of the earth's surface, less than **1%** of these can be utilized as drinking water....
- A growing *number of toxic contaminants* are being discharged to water supplies in *both developing* and *industrialized nations*.



14/03/2023



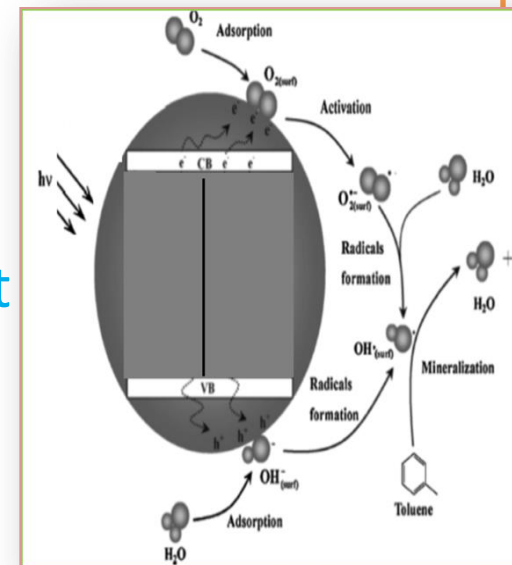
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The Promising material, Photocatalyst

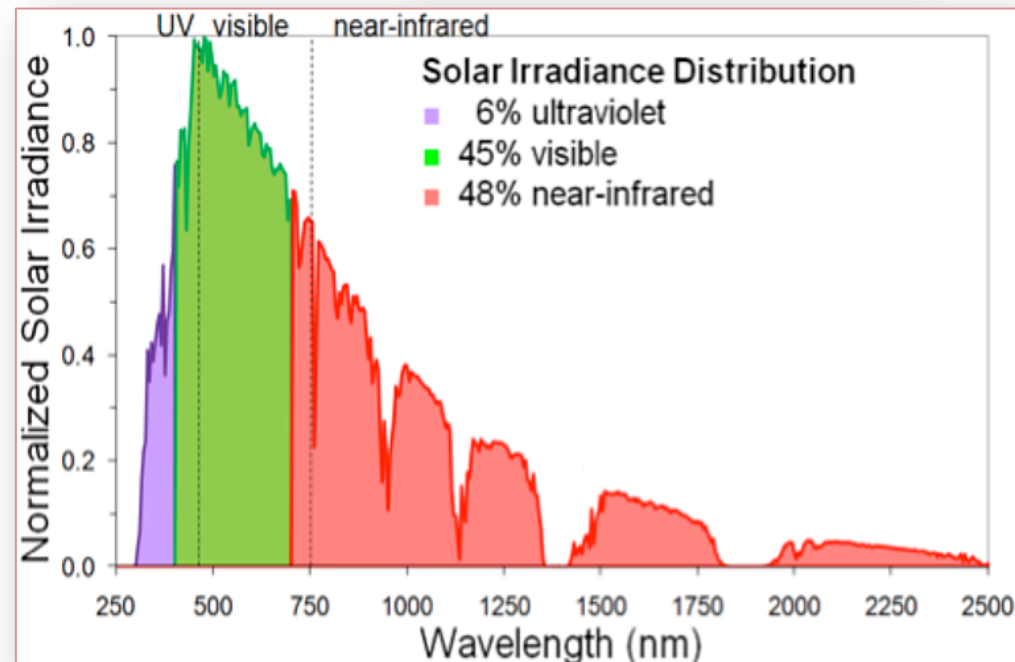
- **Photocatalysts** have received significant attention
 - ✓ are material can perform **light-induced chemical reactions**
e.g. TiO_2 , Fe_2O_3 , ZnO , WO_3 , CdS and SrTiO_3
- The advantages of nano-photocatalysts over conventional
 - ✓ **high specific surface area**
 - ✓ **faster adsorption equilibrium**
 - ✓ **small diffusion resistance**
 - ✓ **high retention reusability character**
 - ✓ **Use the cheapest source of energy, Sunlight**

❖ **How it works...**



Con't...

- **TiO₂** is the most dominant and promising, due to
 - chemically stable, photostability
 - nontoxic, abundant and cheap
 - suitable band alignments
- Commercially as P25 (Degussa)
- However, TiO₂ owns
 - ✓ large band gap- needs UV
 - ✓ low solar energy usage
 - ✓ slow charge mobility
 - ✓ high recombination rate
- Limits its practical applications



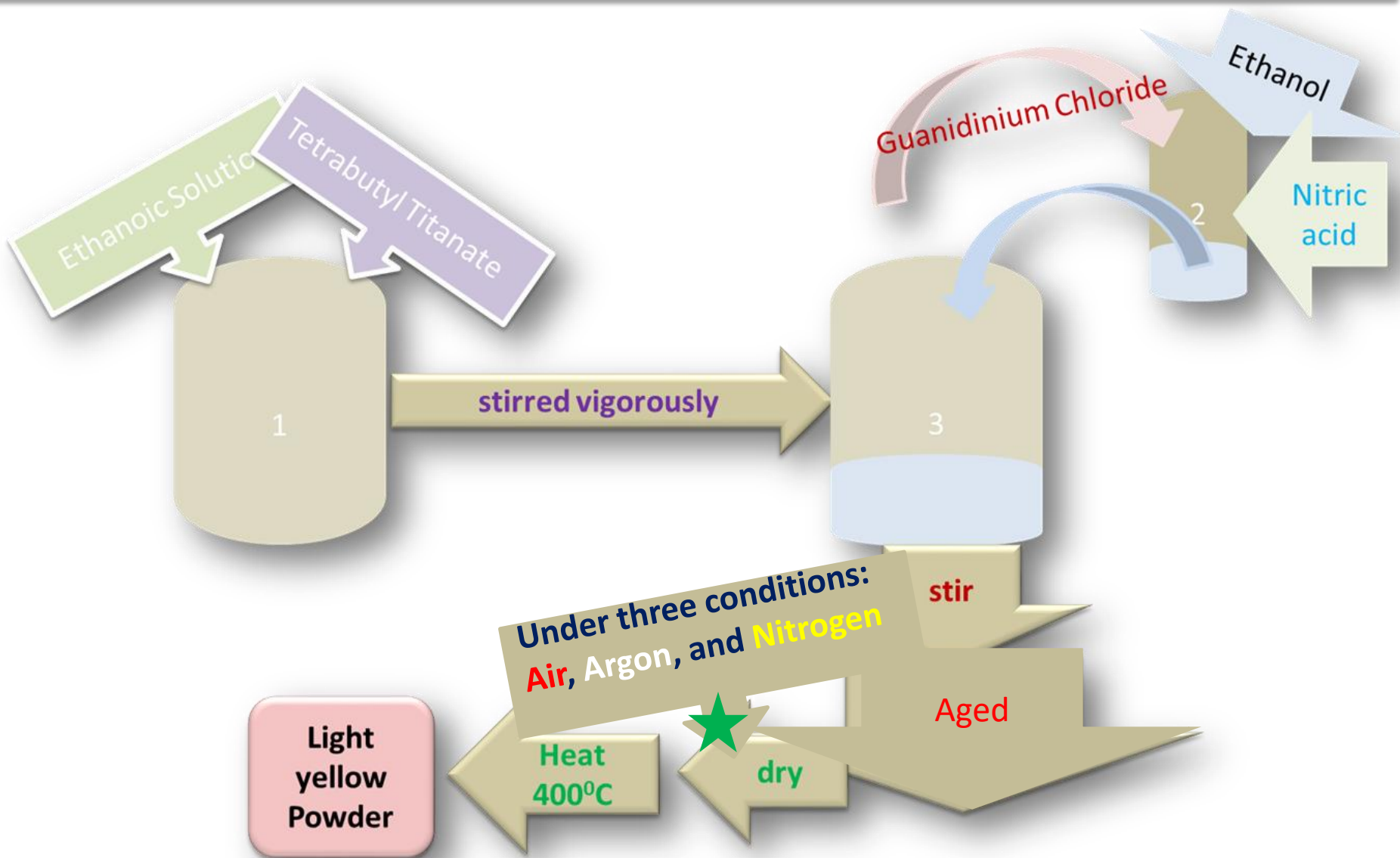
Con't...

- The development of **visible light TiO₂ photocatalyst**
 - **Band Gap Engineering...** Eg. **doping**, coupling, Sensitizing
 - **Surface Engineering...** Eg. defect formation

❖ The main research focus are

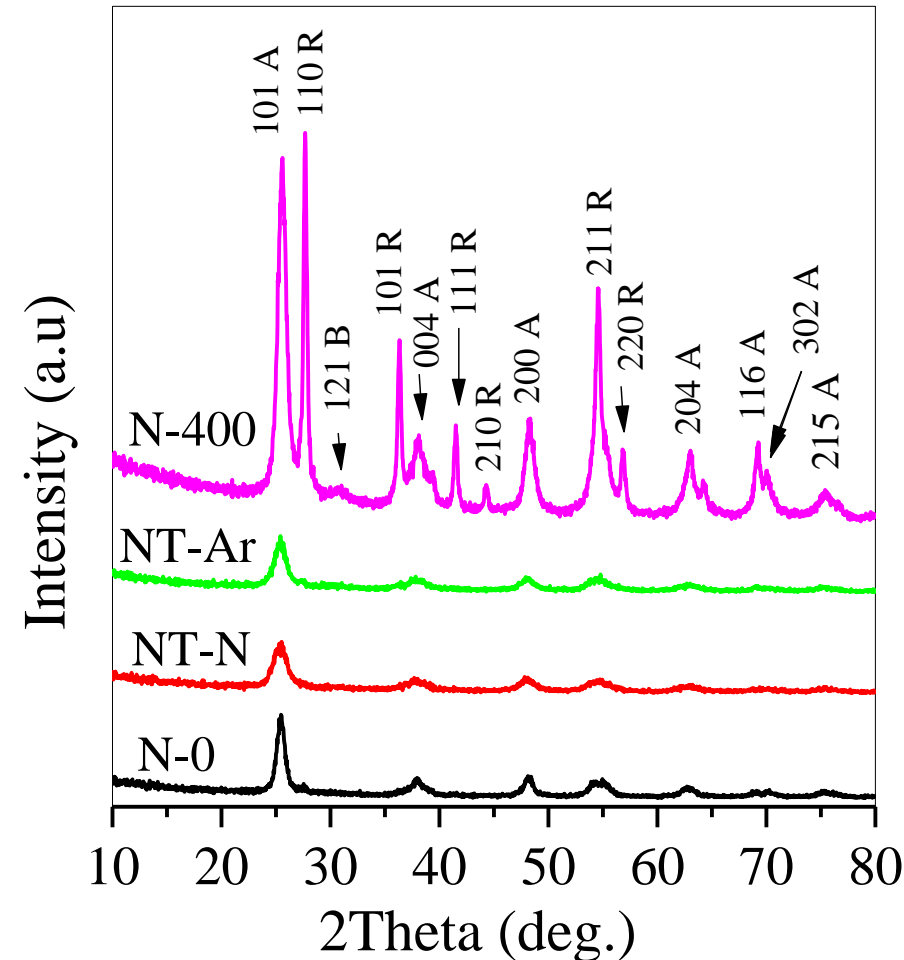
- ✓ To **prepare visible active N-doped TiO₂ nanomaterial**
- ✓ To study the **effect of annealing gas type** on physicochemical properties and photocatalytic active against **Rhodamine B (RhB)**

Preparation Methodology



Result and Discussion

XRD



- Both **NT-Ar** and **NT-N** (which were prepared in Ar, and N₂ gas, respectively) have **98% anatase phase** (JCPDS: 21-1272) like N-0.
- Whereas **NT-A**, which was prepared in atmospheric air, has a mixture of **53% anatase** and **44% rutile** phases.
- The difference in gas environment influences **the degree of crystallinity** and **particle size**.

Figure 1 XRD data of as-prepared nanomaterials (A: anatase, R: rutile).

Morphology

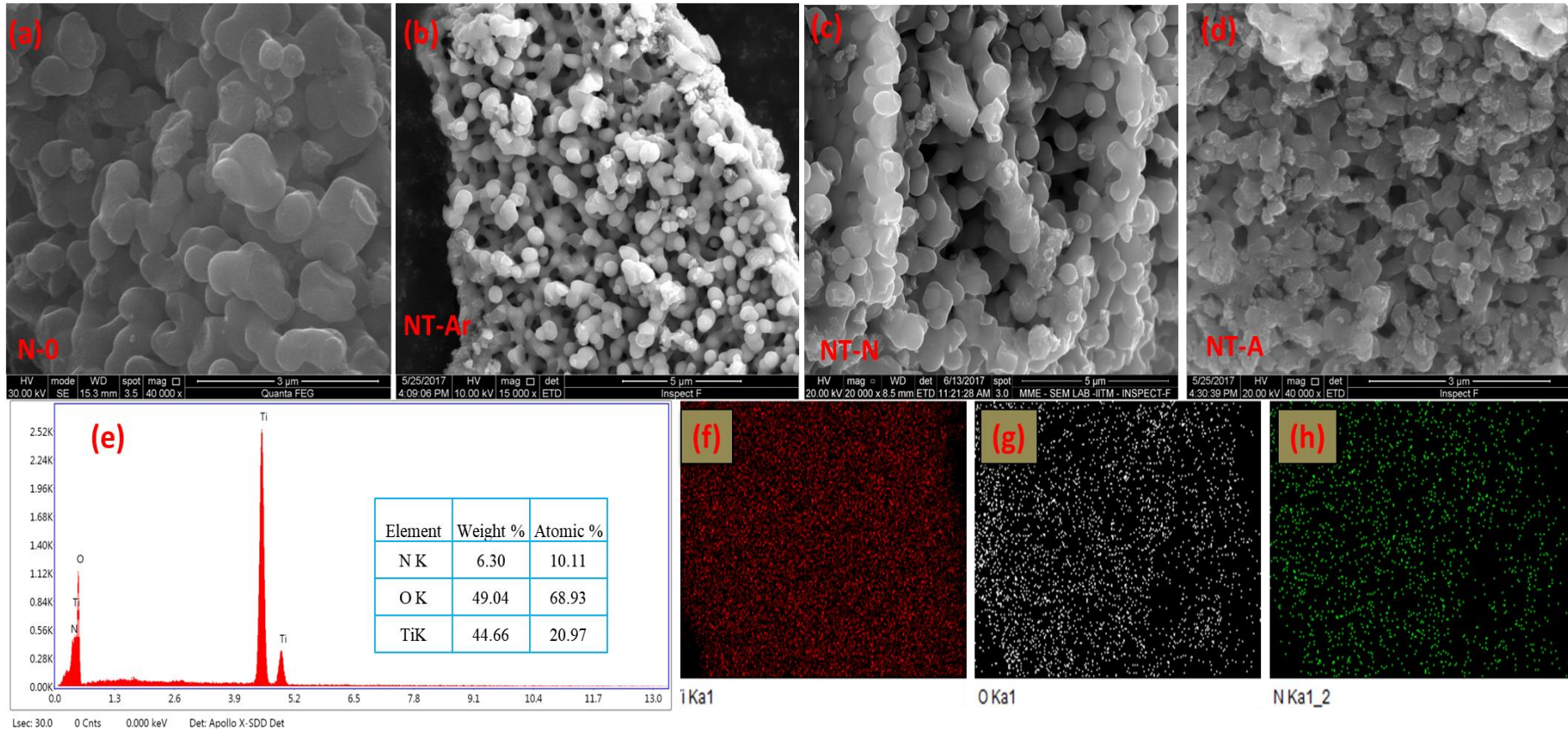
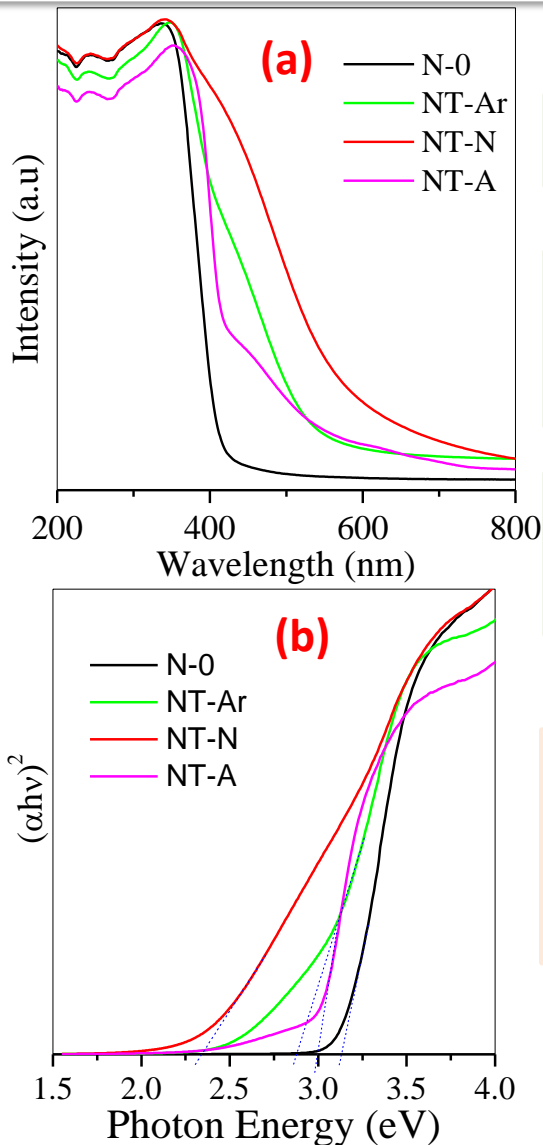


Figure FESEM images of N-0 (a), NT-Ar (b), NT-N (c), NT-A (d); and EDAX of NT-A (e) with its elemental mapping of Ti (f), O (g), N (h).

- ❖ N/TiO₂ has a **spherical shape** with some aggregation
- ❖ N was **effectively doped** and **homogeneously** distributed in the crystal

Con't...

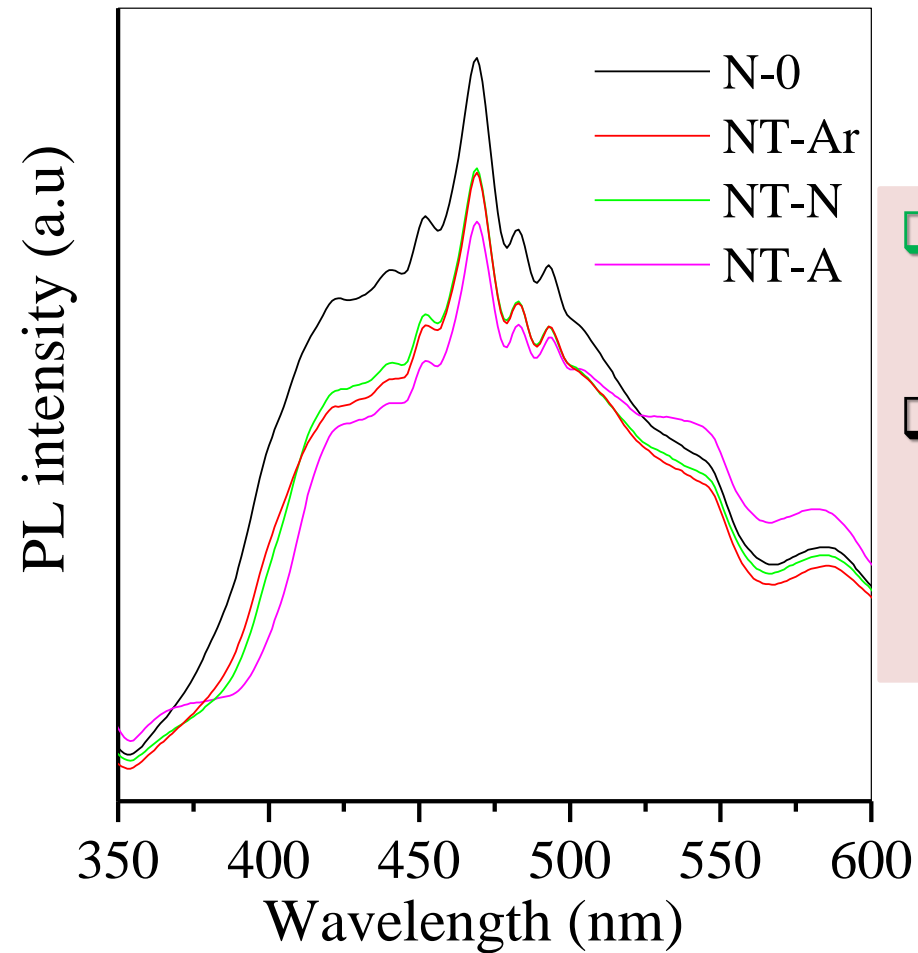


Optical response

- The N-0 absorbs in the UV region (~ 400 nm).
- The N/TiO₂ have two peaks: at ~ 420 nm, and 420-600 nm, enhancing their visible absorption
- The N/TiO₂ samples showed a red shift unlike the undoped.
- K-M plot (b) all the N/TiO₂ materials exhibited lower band gap energy than TiO₂; NT-N particularly demonstrated the lowest of 2.35 eV.

Figure UV-Vis spectra (a), K-M plot (b) of obtained N/TiO₂ nanomaterials.

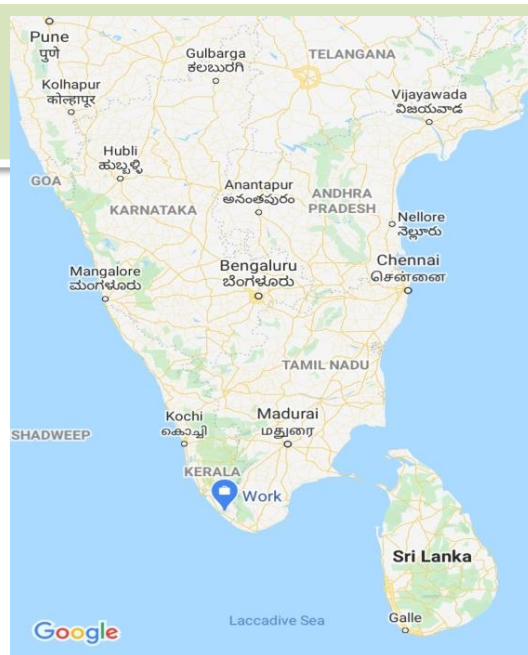
Photoluminescence



- ❑ All the N/TiO₂ samples have lower PL peak than the pure TiO₂
- ❑ revealing the introduction of the N species in the TiO₂ crystal lowers the photo-generated charge carriers' recombination rate

Figure Photoluminescence spectra of obtained N/TiO₂ nanomaterials.

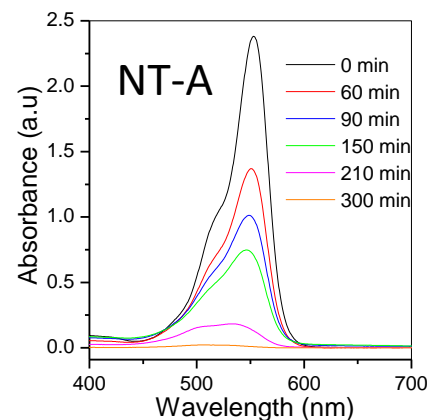
Photocatalytic Activity



Conditions:

- Catalysts Weight: 50 mg
- Rh B Concentration: 20 ppm
- MB Volume: 200 mL
- Light Source: Sun light irradiation at CSIR-NIIST Trivandrum ($8^{\circ}31' N$, $76^{\circ}56' E$) the month of *January* (having Solar irradiance $\sim 240 W m^{-2}$, Temp. $25.5^{\circ}C$, and 66.5% humidity)
- Photoreaction Time: 30 min adsorption and 5 Hrs (11 AM to 4 PM)

NB: The change in the characteristic absorption band of **Rh B 552 nm** was used to calculate the photodegradation efficiency



Con't...

Photocatalytic Activity

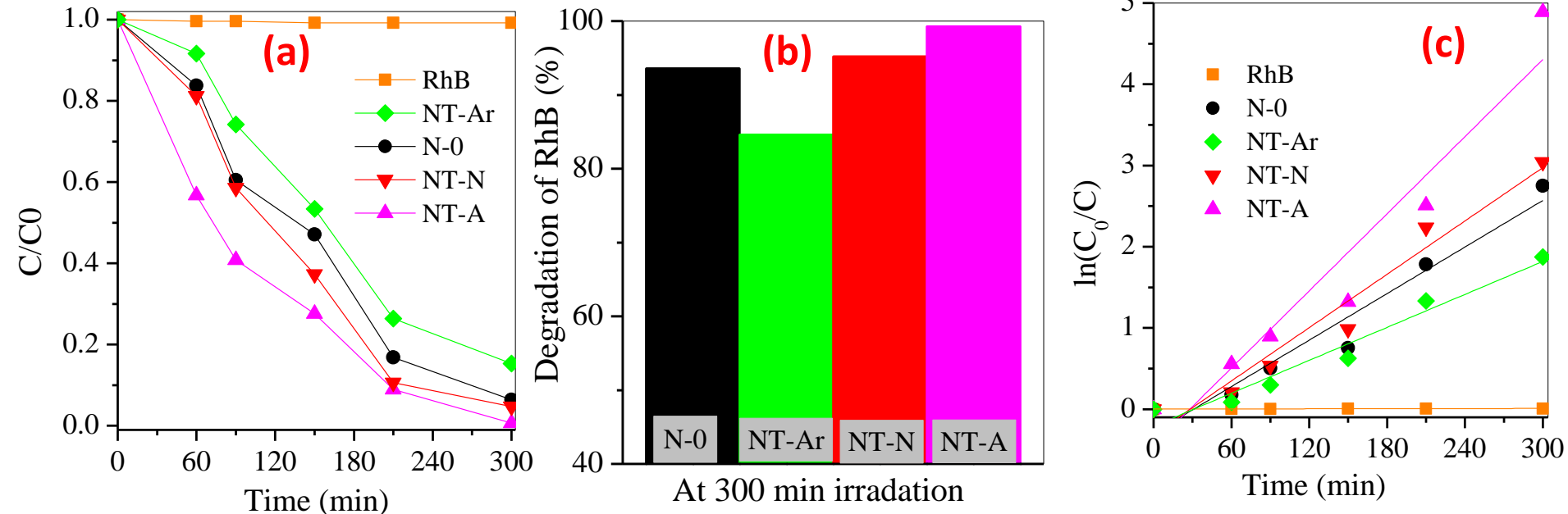


Figure (a) RhB degradation rate under sunlight; (b) RhB photodegradation performance under 300 min irradiation; (c) pseudo first order of as-synthesized catalysts

- ❖ The concentration and characteristic RhB peak intensity are reduced as the function of irradiation time.
- ❖ Particularly, NT-A (the catalyst annealed in air) displayed the highest photocatalytic efficiency (99%) within 300 min sunlight irradiation.
- ❖ The RhB degradation performance was in the following order:
NT-A (in air) > NT-N (in nitrogen) > N-0 > NT-Ar (in argon)

Conclusion

- The N/TiO₂ powders were optimized at different annealing gas types (air, argon, nitrogen) which profoundly influenced their physicochemical and photocatalytical properties.
- Notably, the sample prepared in air demonstrated the highest degradation performance (99%) with the highest apparent rate constant (0.0158 min⁻¹) which is twice faster than the undoped TiO₂.
- Such outstanding performance is attributed to the synergistic effect of N doping and its optimal anatase/rutile phase which led to higher specific surface area, higher light absorption capacity, lower band gap energy, and lower charge carriers recombination.
- Such visible active dual phase N/TiO₂ photocatalysts will have practical applications in photocatalysis, photoelectrochemical, and photoelectric areas.

Acknowledgment



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