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Preparation of some metal oxides nanoparticles using Chitosan as template

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ABSTRACT:

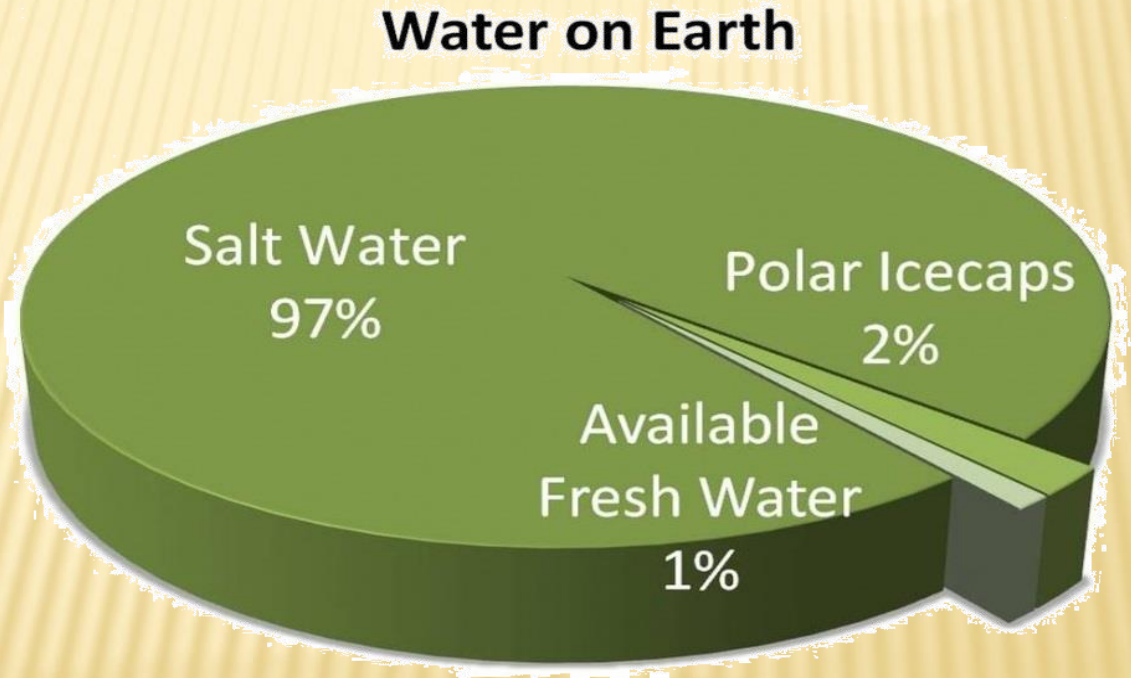
- Chitosan (CS) was included in the preparation of TiO_2 and ZrO_2 nanoparticles to improve their morphology.
- **Nanoparticles were prepared separately in presence of CS and / or Palladium (Pd).**
- **After characterization of the nanoparticles, their photocatalytic activity on MB and TH as model pollutants under visible light irradiation was investigated.**
- **The catalyst reproducibility has been also tested.**

INTRODUCTION

The most important thing on earth = life = WATER

INTRODUCTION

Clean water resources are very limited relative to total



- **Daily life activities bring pollutants and reduce the available clean water causing serious problems and diseases**

SOURCES OF POLLUTANTS

[Most of the dangerous pollutants are the Organic Pollutants]

- ***Household or domestic and industrial but mostly industrial***

SOURCES OF POLLUTANTS

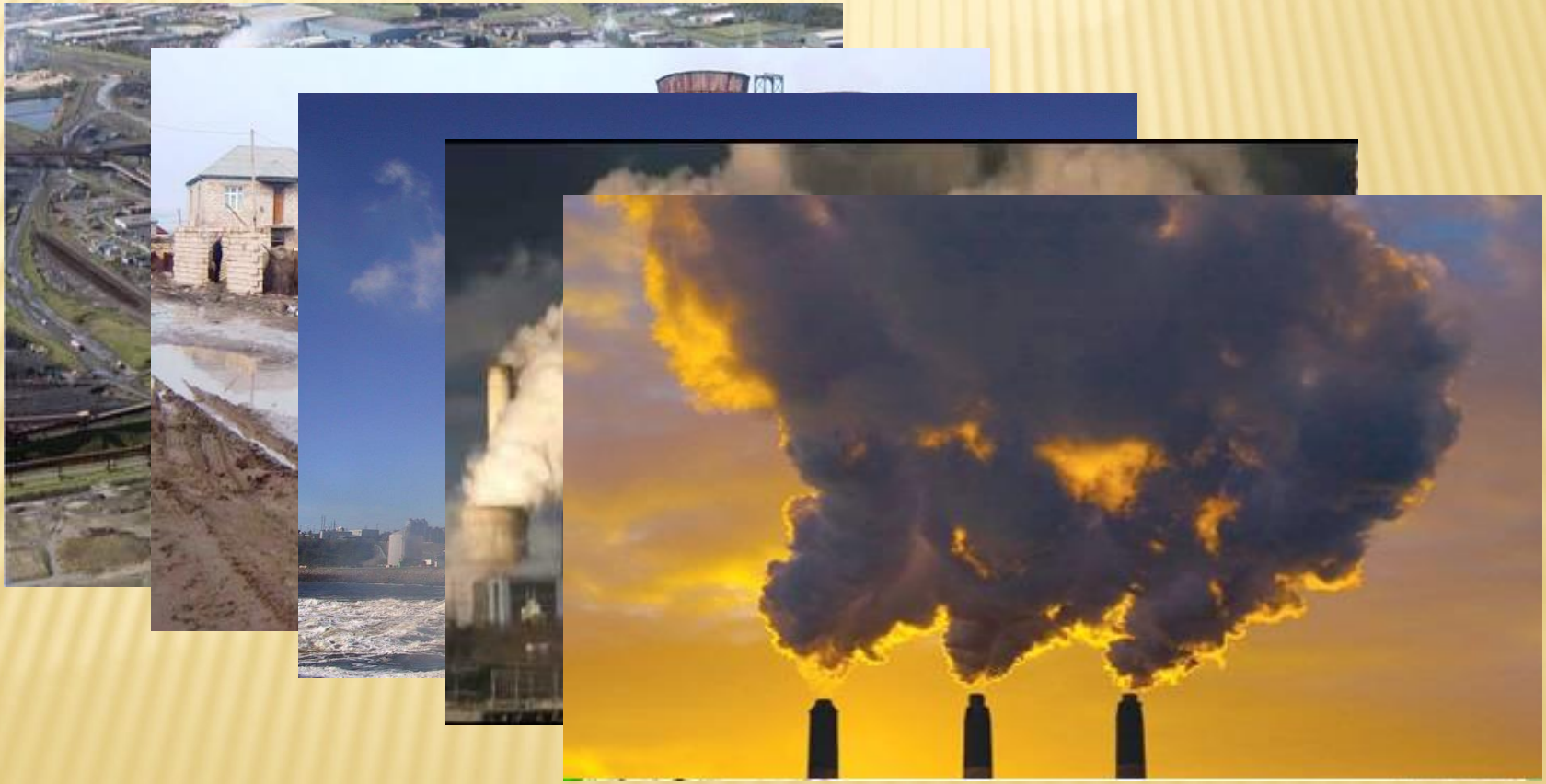
- ***Food and Cosmetic Industry***

SOURCES OF POLLUTANTS

- ***Dyes and Textile Industry***

SOURCES OF POLLUTANTS

- *Chemical Industries*



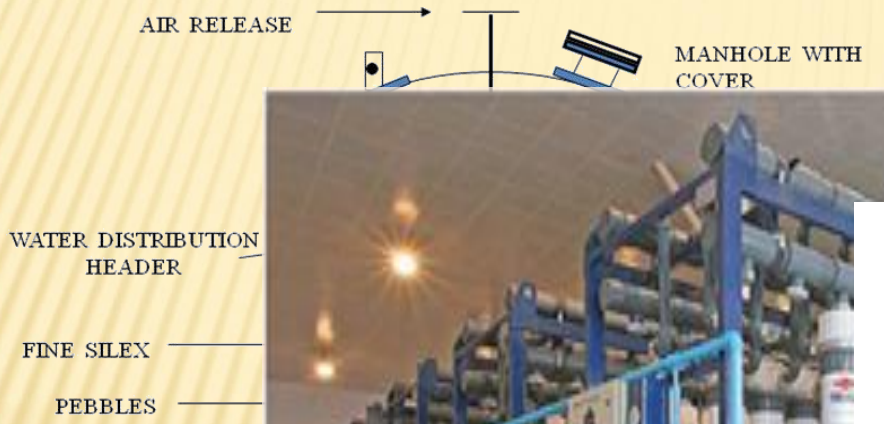
WHAT IS THE SOLUTION?!

- ***Get more resources of clean water ?!*** ***(Difficult and costing)***
- ***Minimize daily life activities those are impacting the environment … … !!!!***
- ***Reduce the consumption of clean water … !!***
- ***Cleaning contaminated water***

(mainly from Organic Pollutants)

HOW?!!

TECHNIQUES FOR CLEANING OR DECONTAMINATING WATER



Reverse Osmosis (RO)

They are non-destructive and just transferring the contaminants from one phase to another

Adsorption on activated Carbon

Ultrafilteration

TECHNIQUES FOR CLEANING OR DECONTAMINATING WATER

- ***Chlorination and ozonization are also ineffective and expensive.***
- ***Heterogeneous catalytic degradation known also as Advanced Oxidation Process (AOP) of the Organic Pollutants.***
- ***Photocatalysis is one of AOP used to overcome the thermal issues but needs for irradiation mostly in UV region which has many limitations.***
- ***Photocatalysis would be promising if its effectiveness could be shifted to the visible light range of irradiation.***

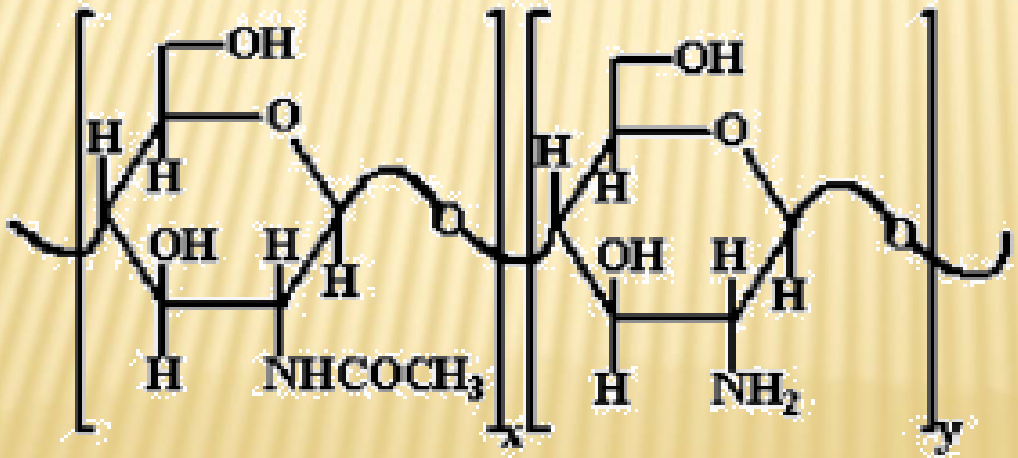
NANOPHOTOCATALYSTS OF TITANIUM AND ZIRCONIUM OXIDES

((PROBLEM HIGHLIGHTING))

- *TiO_2 and ZrO_2 are known as good and effective catalysts with high band gap energy (> 3.0 eV).*
- *This means they are applicable in UV region.*
- *Pd is a promising dopant to extend the photo-response range.*
- *TiO_2 and ZrO_2 and their Pd-doped version are good photocatalysts especially on the nanoscale of the particle size to increase the **EFFECTIVE** surface area.*
- *The main problem is the **AGGLOMERATION** of nanoparticles during calcination leading to **REDUCTION** in the effective surface area of the catalyst.*

CHITOSAN:
A DERIVED NATURAL POLYMER

- ***Chitosan is known as the most common existing natural polymer after cellulose. It is a cationic polymer rich in reactive hydroxyl and amino groups.***
- ***It has good ability to absorb and chelate several heavy metal ions and also for drug delivery systems.***

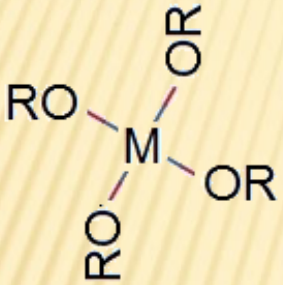


Chitosan

Hence, Chitosan will be used in the preparation of the nanophotocatalysts

EXPERIEMENTAL:

* Preparation of MO_2 & Pd/MO_2 Nanoparticles



- Ethanol
- HCl
- dd H_2O

→ **Clear Sol** → Pour onto aq. NaOH → **$M(OH)_4$** ↓

- Separate
- Wash well with dd H_2O
- Dry 24h at 100°C

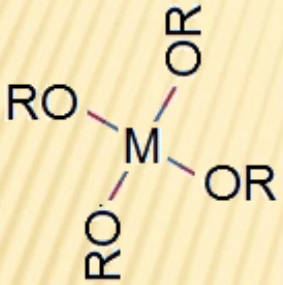
← **Dry $M(OH)_4$** ← Calcination at 550°C/5h ← **MO_2 Nanoparticles (Sample I)**

- $PdCl_2$ suspension in H_2O
- Stirring (1h) (Impregnation)
- Evaporation while stirring
- Calcination at 550°C/5h

→ **Pd/MO_2 Nanoparticles (Sample II)**

EXPERIEMENTAL:

* Preparation of CS-Modified Nanoparticles



- Ethanol
- HCl
- dd H_2O

→ **Clear Sol**

- Simultaneous mixing while stirring
- Aq. NH_4OH to pH 9

→ **Mixed Precipitate**

Chitosan

- Dil. aq. Acetic acid
- Stirring for 10h

→ **Clear Solution**

Mixed Precipitate →

- Separate
- Wash well with dd H_2O
- Dry 24h at 100°C
- Calcination at 550°C/5h

→ **MO_2-CS Nanoparticles (Sample III)**

$PdCl_2$ as before →

Pd/MO_2-CS Nanoparticles (Sample IV)

EXPERIEMENTAL:

* Preparation of CS-Modified Nanoparticles

Now we have:

* TiO_2 Nanoparticles
* Pd/TiO_2 Nanoparticles
* TiO_2-CS Nanoparticles
* Pd/TiO_2-CS Nanoparticles

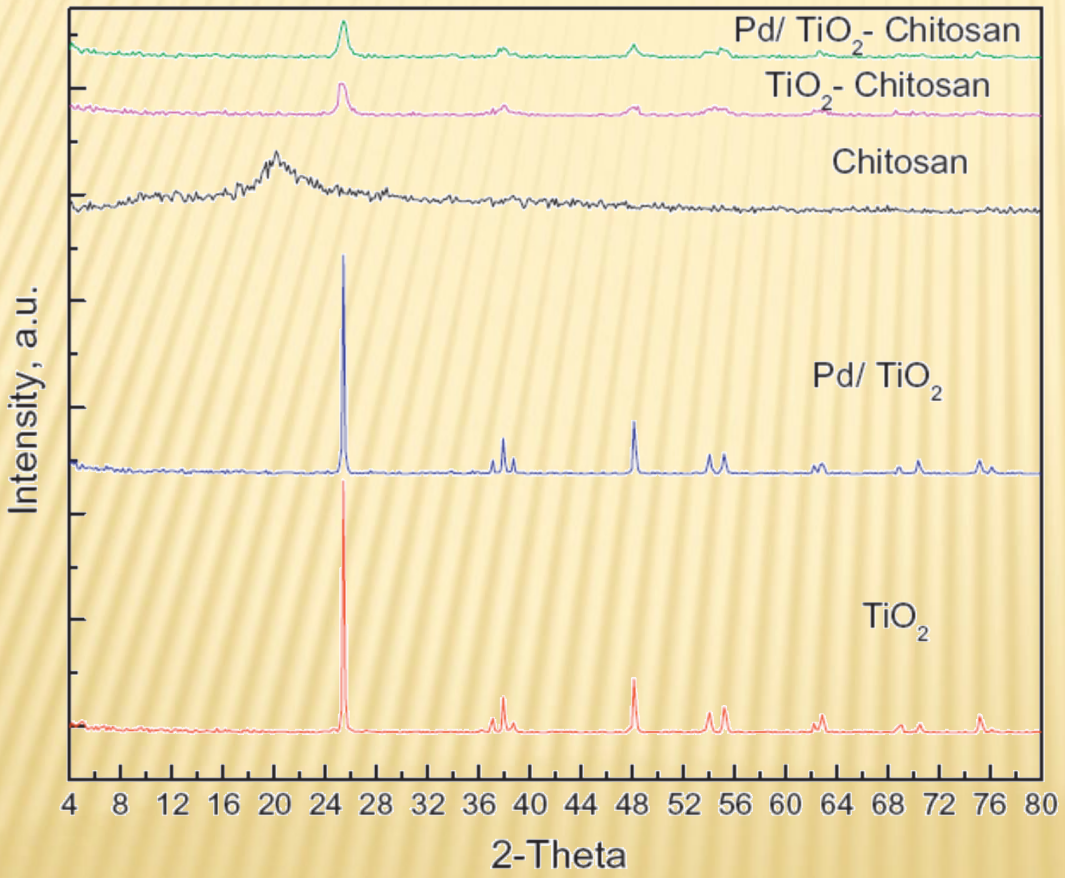
---

* ZrO_2 Nanoparticles
* Pd/ZrO_2 Nanoparticles
* ZrO_2-CS Nanoparticles
* Pd/ZrO_2-CS Nanoparticles

ready for Characterization & Application

CHARACTERIZATION:

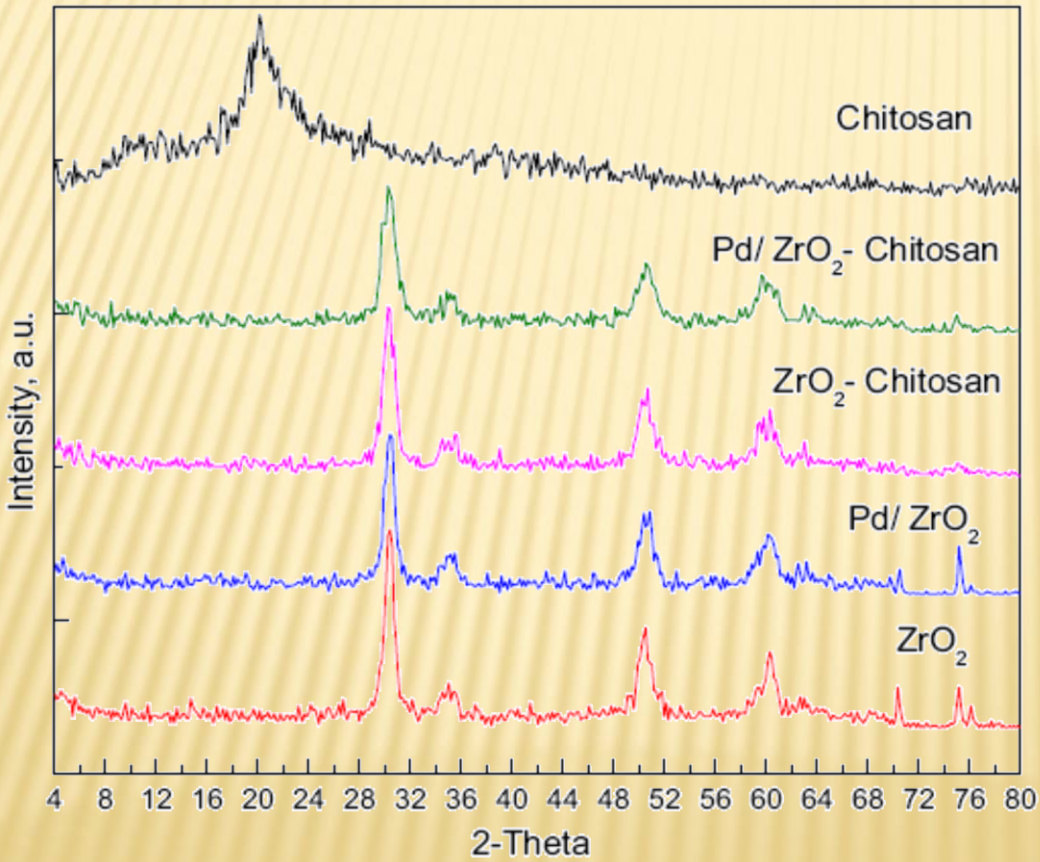
1. X-Ray Diffraction analysis (XRD)



All samples consist of *anatase* phase which demonstrate no obvious influence on crystallization process of TiO_2 upon the addition of Chitosan

Fig. 1. XRD patterns of CS, TiO_2, Pd/TiO_2, TiO_2–CS and Pd/TiO_2–CS.

CHARACTERIZATION:

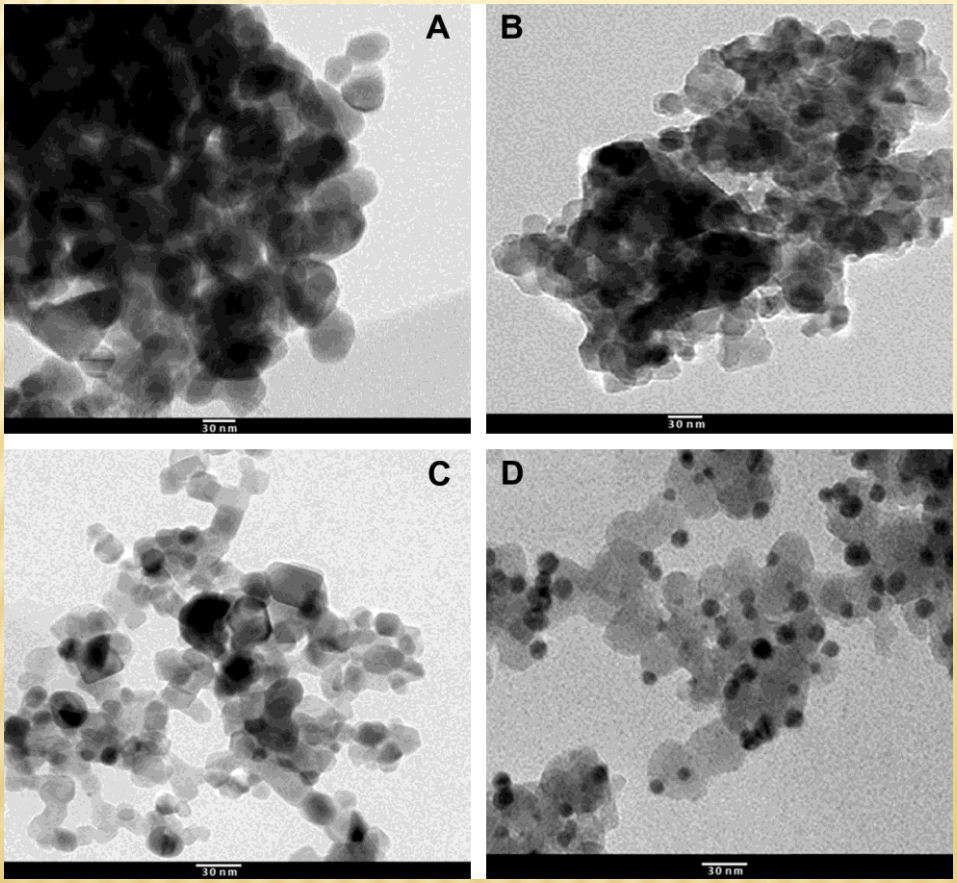


All the samples consist of *zirconia* phase which demonstrate no obvious influence on crystallization process of ZrO_2 upon the addition of Chitosan

Fig. 2. XRD patterns of CS, ZrO2, Pd/ZrO2, ZrO2–CS and Pd/ZrO2–CS.

CHARACTERIZATION:

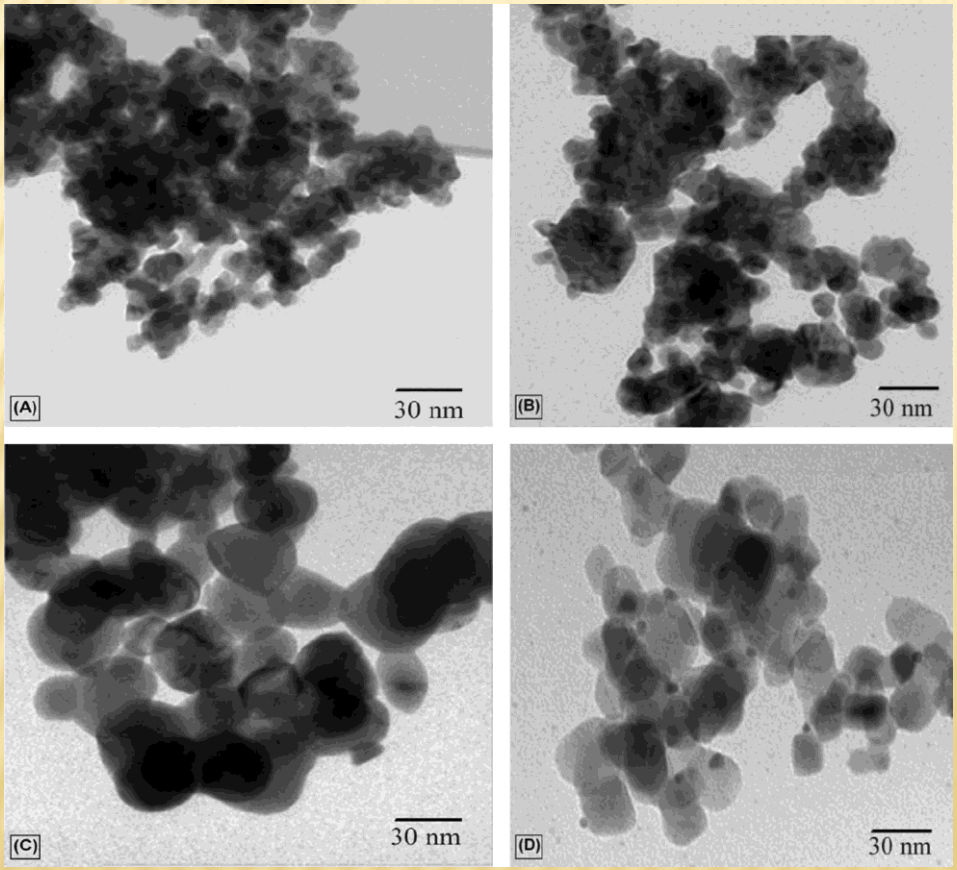
2. Transmittance Electron Microscopy (TEM)



- TiO_2-CS nanoparticles were relatively uniform while showed serious agglomeration for TiO_2 nanoparticles.
- Pd/TiO_2 nanoparticles showed agglomeration while Pd/TiO_2-CS showed dispersion of Pd over the uniform of TiO_2 nanoparticles.
- Particle size was estimated as 45, 40, 18 & 16 nm for TiO_2, Pd/TiO_2, TiO_2-CS & Pd/TiO_2-CS nanoparticles, respectively.

Fig. 3. TEM Images of TiO_2 (A), Pd/TiO_2 (B), TiO_2-CS (C) and Pd/TiO_2-CS (D).

CHARACTERIZATION:



- ZrO_2 nanoparticles showed serious agglomeration while ZrO_2-CS nanoparticles are relatively uniform.
- The same was for Pd/ZrO_2 and Pd/ZrO_2-CS nanoparticles where Pd was dispersed over uniform of ZrO_2 nanoparticles.
- It reflects the role of CS in the distribution uniformly of ZrO_2 and Pd/ZrO_2 nanoparticles.

Fig. 4. TEM Images of ZrO_2 (A), Pd/ZrO_2 (B), ZrO_2-CS (C) and Pd/ZrO_2-CS (D).

CHARACTERIZATION:

3. UV/Vis-Diffuse Reflectance Spectroscopy

UV/Vis-Diffuse Reflectance Spectroscopy for both kinds of photocatalysts proved that band gap energy is getting lower by modification in the order of: Oxide > Pd/Oxide > Oxide-CS > Pd/Oxide-CS.

Table 1: The band gap energy, eV, Ti- and Zr- nanophotocatalysts

Sample	Band gap Energy , eV (Change %)	
	Ti-Photocatalysts	Zr-Photocatalysts
MO_2	3.23	3.12
Pd/MO_2	3.04 (5.26)	2.99 (4.17)
MO_2-CS	2.72 (15.79)	2.88 (7.69)
Pd/MO_2-CS	**2.60 (19.50)**	**2.80 (10.26)**

- ***Band gap energy was lower for Pd/TiO_2-CS than that for Pd/ZrO_2-CS. It means more shift to visible region in case of Ti – nanophotocatalyst.***

CHARACTERIZATION:

4. Photoluminescence Emission Spectroscopy (Pl)

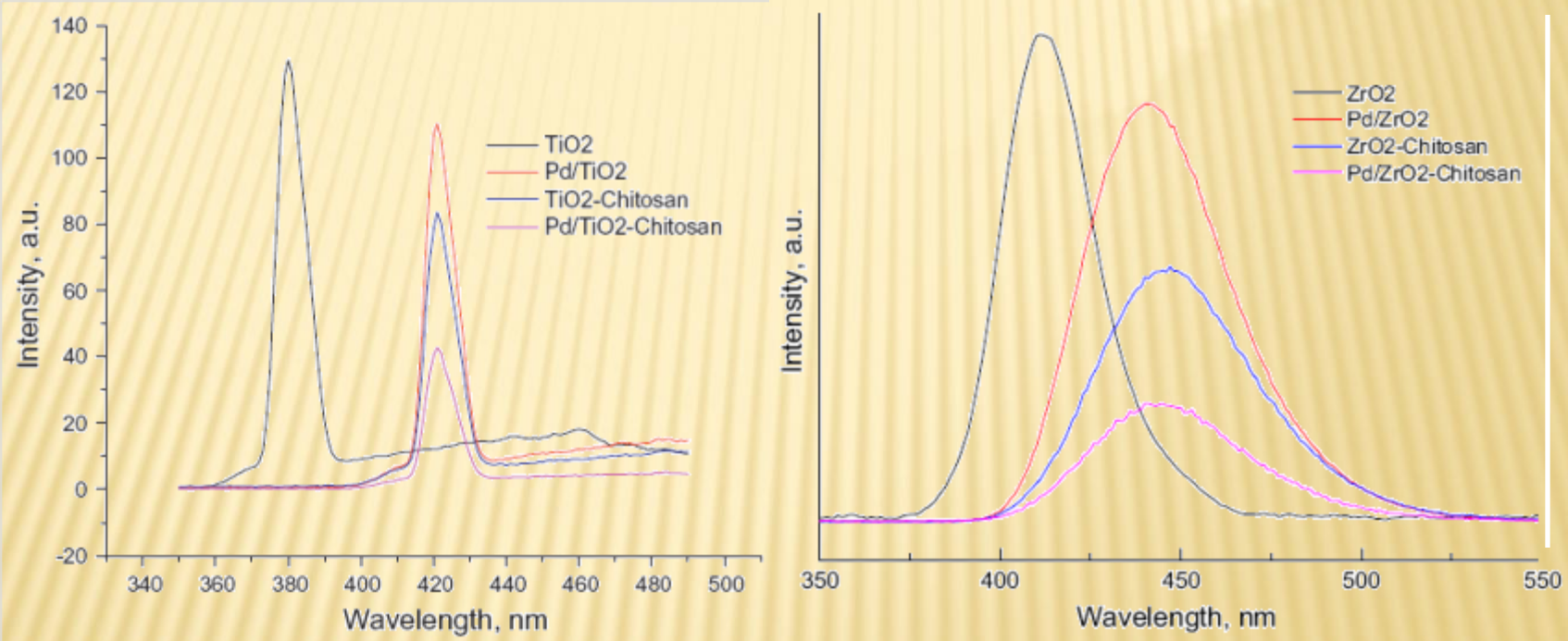


Fig. 5: Pl Spectra of MO_2 , Pd/MO_2 , MO_2-CS and Pd/MO_2-CS photocatalysts

The Intensity in both cases of Ti and Zr photocatalysts increases in the order of :

$Pd/MO_2 > MO_2$-CS > Pd/MO_2-CS

CHARACTERIZATION:

5. Surface Area

There is no significant difference between both kinds of photocatalysts in the total pore volume or pore radius, however, they increase by modification with CS rather than with Pd in the order of: MO_2-CS >Pd/MO_2-CS > MO_2 > Pd/MO_2

Table 2: Pore Volume, Vp (cm3/g) and Pore Radius, R (Å) of the nanopl

Sample	Ti-Photocatalyst		Zr-Photocatalyst	
	Vp (cm3/g)	R (Å)	Vp (cm3/g)	R (Å)
CS	0.81	34	0.81	34
MO_2	0.69	39	0.68	39
Pd/MO_2	0.66	40	0.64	42
MO_2-CS	0.77	36	0.75	35
Pd/MO_2-CS	0.71	37	0.71	38

APPLICATION:

* Photodegradation of Organic Pollutants

- **Methylene Blue (MB) and Thiophene (TH) were used as organic pollutants in case of using Ti- and Zr-photocatalysts, respectively**

- **Type and amount of the photocatalyst have been investigated as well as the catalysts reproducibility**

APPLICATION:

1. Effect of Type of the Nanophotocatalyst

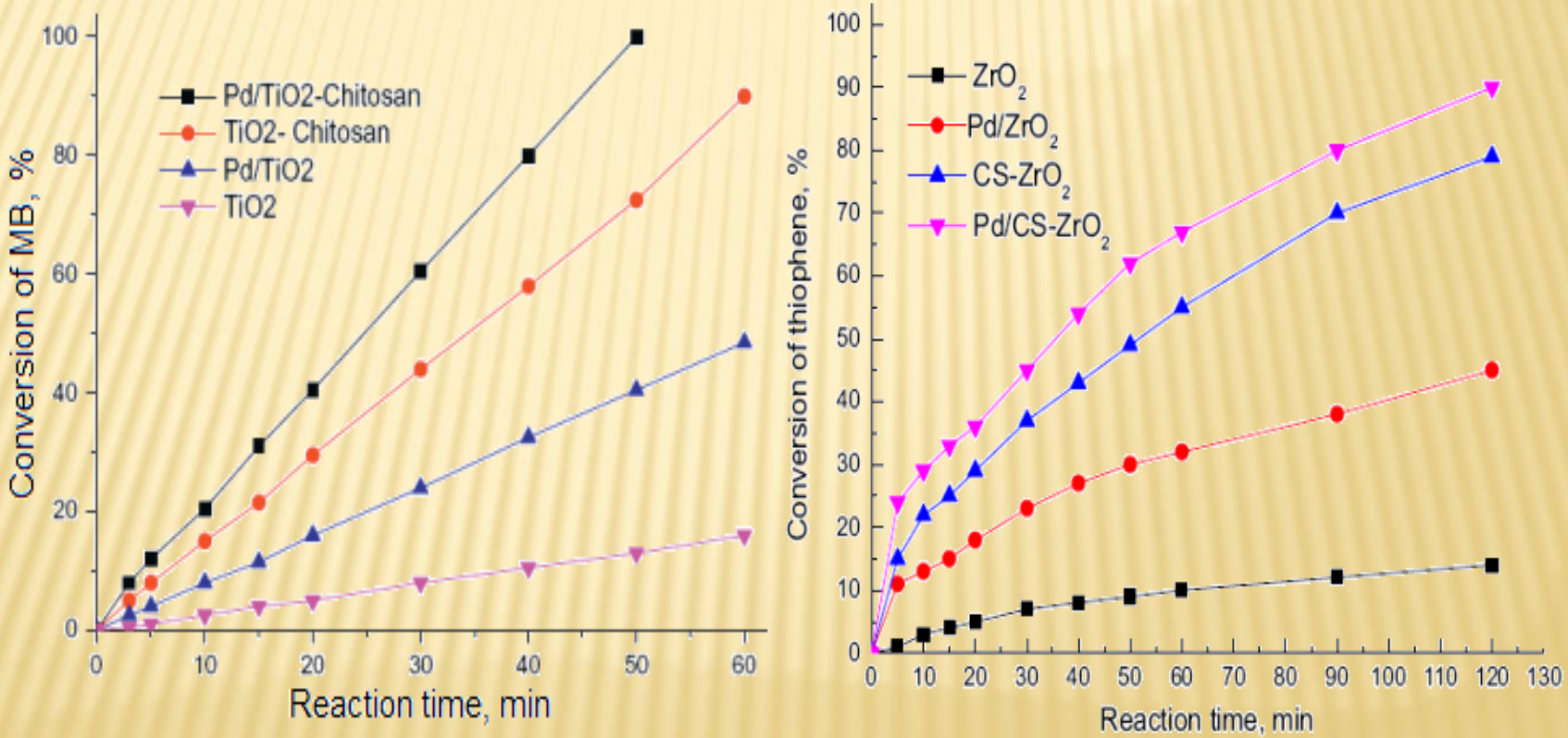


Fig. 6: Effect of catalyst type on the photocatalytic degradation of MB and TH

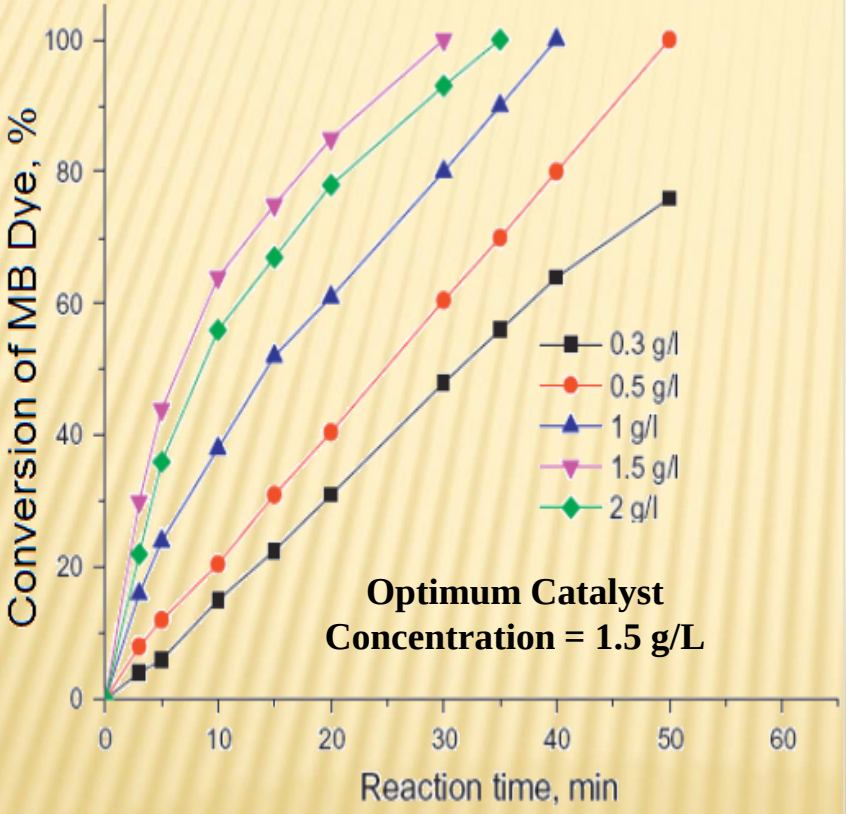
APPLICATION:

- All the investigated catalysts showed significant role as MB and TH were completely degraded into CO_2 , H_2O and SO_3 for TH only in less than 1 h in the visible light.
- The efficiency of the investigated nanophotocatalysts were in the order:

$$Pd/MO_2\text{-}CS > MO_2\text{-}CS > Pd/MO_2 > MO_2$$

APPLICATION:

2. Effect of Amount of the Nanophotocatalyst



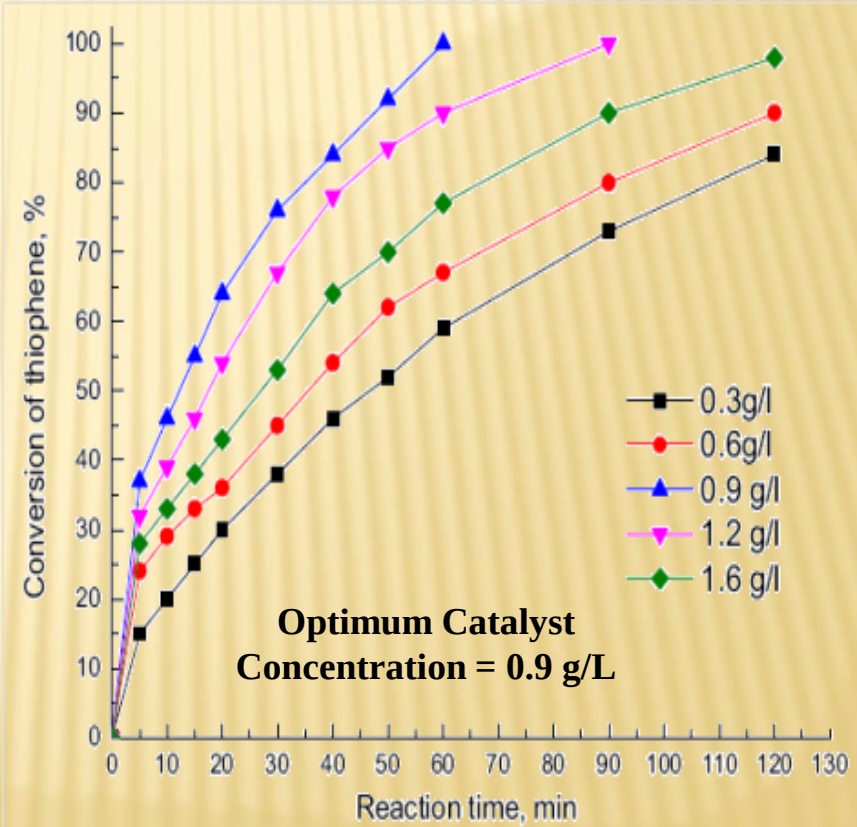


Fig. 7: Effect of amount of catalyst on the photocatalytic degradation of MB and TH

APPLICATION:

3. Reproducibility of the nanophotocatalyst

Both kinds of the nanophotocatalysts showed good reproducibility up to FIVE cycles with no loss in their activities

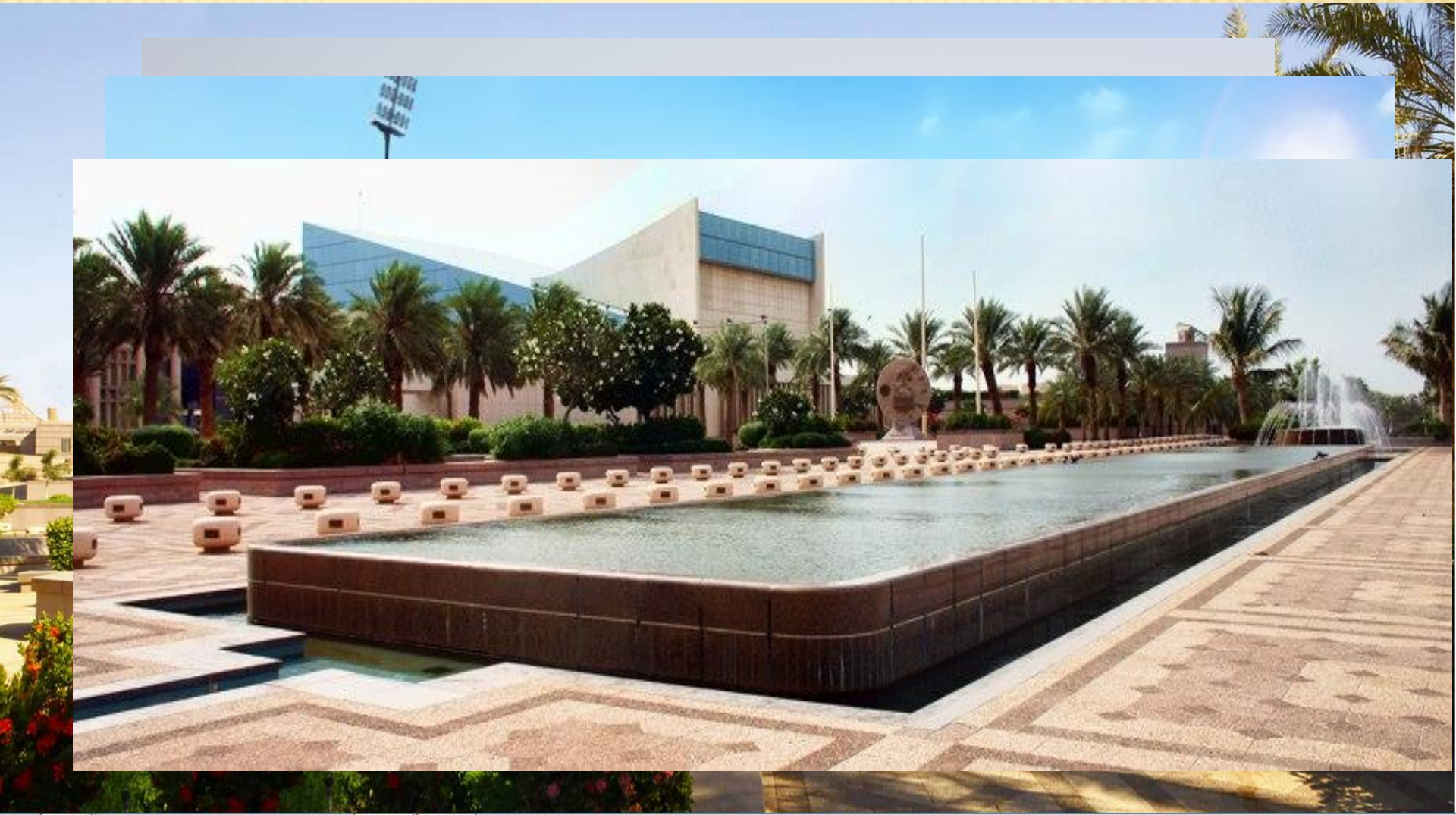
CONCLUSION AND REMARKS:

- **Chitosan helps successfully to prepare nano-sized photocatalysts of relatively uniform and of dispersed homogeneity.**
- **Chitosan plays a promoting role in the crystallization process and prevents agglomeration in the calcination process of the nanocatalyst.**
- **The Photocatalytic degradation of the nanocatalyst proved activity reaches up to 100 % conversion of the investigated pollutant within relatively short time.**
- **Band gab energy of the photocatalyst was reduced in presence of Chitosan for both kinds of catalysts and the reduction of the band gap energy for Ti-photocatalysts was more than that for Zr-photocatalysts.**

My presentation is coming to end. I'd like to use the chance and show you few slides about the places where I'm working in Saudi Arabia (as Sabbatical) and also where I live and work permanently in Mansoura University in my country, Egypt

PLACES IN HEART:

A. King Abdulaziz University, Jeddah, Saudi Arabia




بيئة نظيفة حياة افضل
عزيزى الطالب
على جميع الطلبة والطالبات
عدم القاء أو ترك مخلفات
المأكولات والمشروبات فى شوارع
الحرم الجامعى والمنشآت
الخضراء ووضعها فى صناديق
القمامة حفاظا على
المظهر الحضارى لجامعتكم
وذلك منعا للمسائلة
شارك معنا فى نظافة جامعتك

ACKNOWLEDGEMENTS

- Firstly, I'd like … e co-author, Prof. …
- Also, I'd like t… …anship of Scientific Res… …dah, Saudi Arabia for tec… …pport.
- Finally, many … …ng Committee fo… … since submission o… …ng sure about accomm…
- At last but no… …ank all audience for p…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