



Nanocomposites Materials Research Laboratory



College of Nanotechnology
King Mongkut's Institute of Technology Ladkrabang

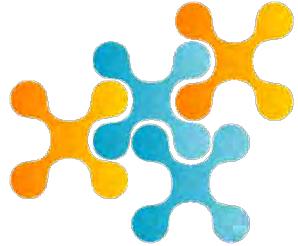


Functional Material derived from Activated Natural Ore and Its Utilizations

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Sorapong Pavasupree
Rajamangala University of Technology Thanyaburi

Keiichi N. Ishihara
Kyoto University

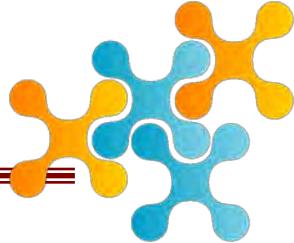


Outline

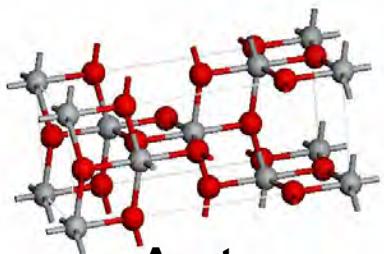
- Introduction**
- Materials and methods**
- Results**
- Conclusion**



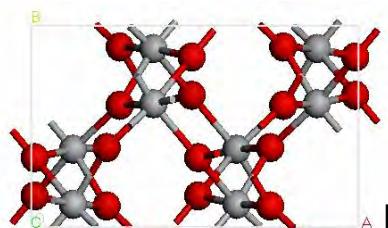
Titanium dioxide; TiO_2



Rutile



Anatase



Brookite

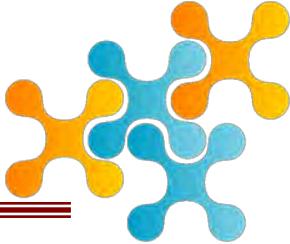
■ Prominent properties

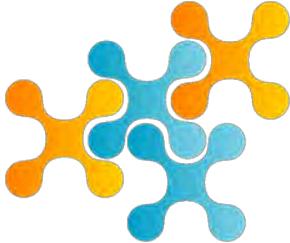
- Wide Band gap (3.0-3.2 eV)
- Strong ultraviolet absorption
- Chemical stability
- Non-toxic

Physical and structural properties of anatase and rutile TiO_2 .

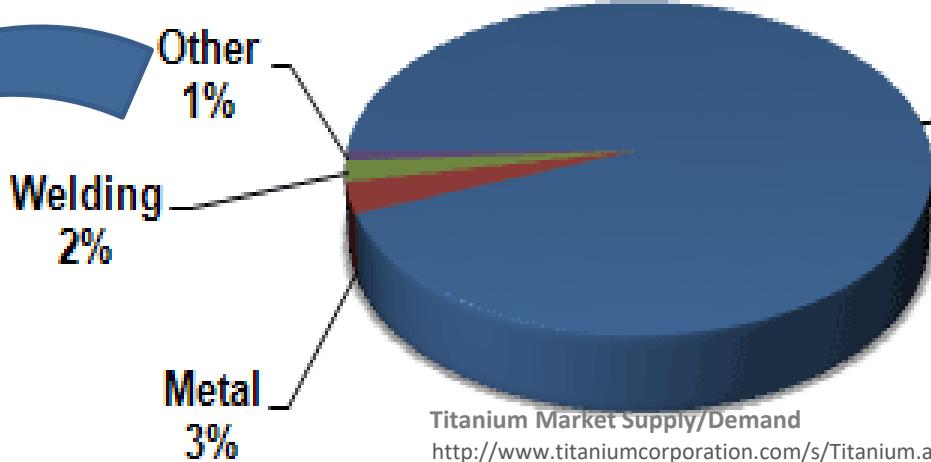
Property	Anatase	Rutile
Molecular weight (g/mol)	79.88	79.88
Melting point (°C)	1825	1825
Boiling point (°C)	2500–3000	2500–3000
Light absorption (nm)	<390	<415
Mohr's Hardness	5.5	6.5–7.0
Refractive index	2.55	2.75
Dielectric constant	31	114
Crystal structure	Tetragonal	Tetragonal
Lattice constants (Å)	$a = 3.78$ $c = 9.52$	$a = 4.59$ $c = 2.96$
Density (g/cm³)	3.79	4.13
Ti—O bond length (Å)	1.94(4) 1.97(2)	1.95(4) 1.98(2)

Applications of TiO_2

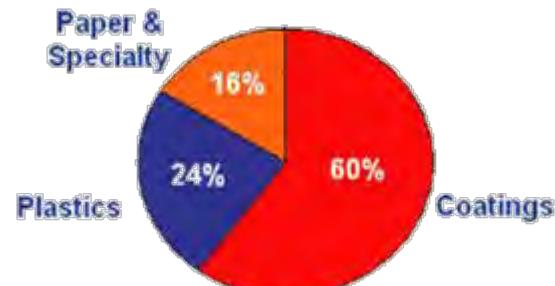




Global Consumption of TiO₂



**Pigment (paint,
paper, plastics)
94%**



food
cosmetics. sun screen lotions
Nano titanium dioxide

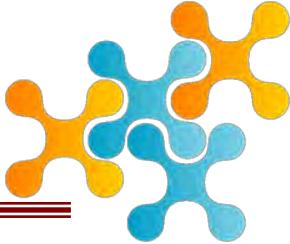
energy and environmental sciences

dye-sensitized solar cells
photovoltaic devices
photocatalysts

Energy storage
lithium/sodium storage
supercapacitors



TiO₂ in natural ores



Form of Titanium	TiO ₂ %	Magnetic Susceptibility	Electrical Conductivity	Specific Gravity
Ilmenite - Sulphate - Chloride	52 - 54 58 - 62	High	High	4.5 - 5.0
Rutile	95 - 97	Low	High	4.2 - 4.3
Synthetic Rutile	88 - 95			
Leucoxene	70 - 91	Semi	High	3.5 - 4.1

Sakorn Minerals Co., Ltd., Thailand

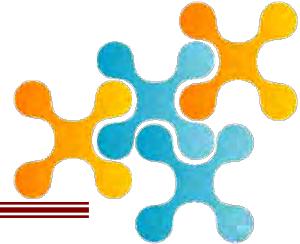
References:

<http://metalpedia.asianmetal.com/metal/titanium/resources&production.shtml>

<http://www.mindat.org/photo-95122.html>

<http://www.mine-engineer.com/mining/mineral/rutile.htm>

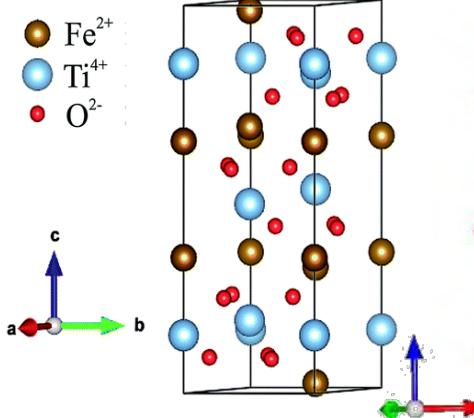
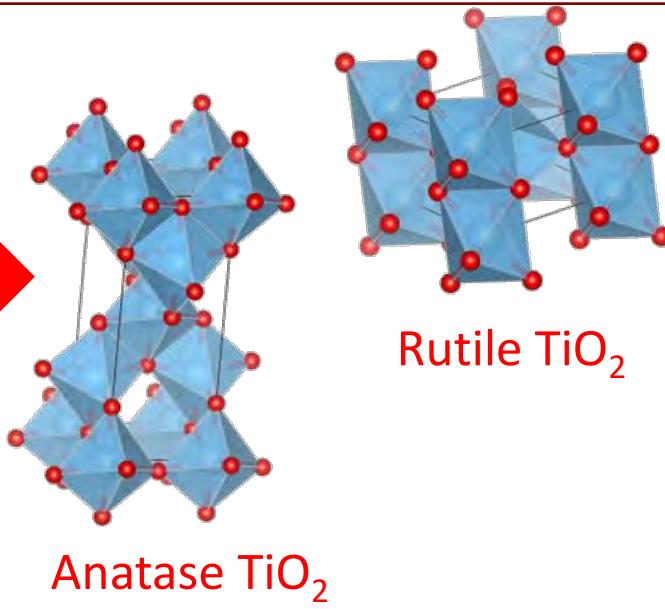
Ti-based natural minerals



Ilmenite



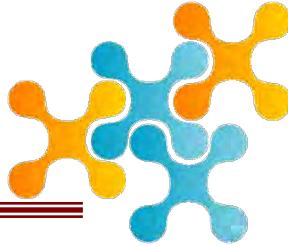
Leucoxene



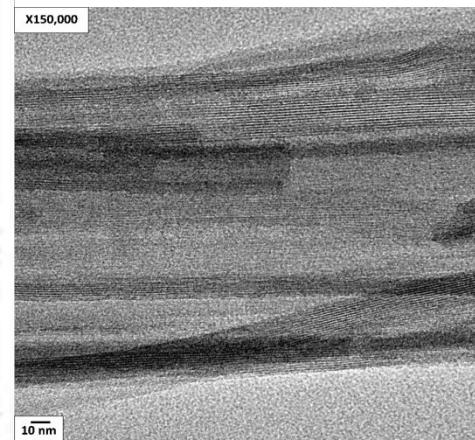
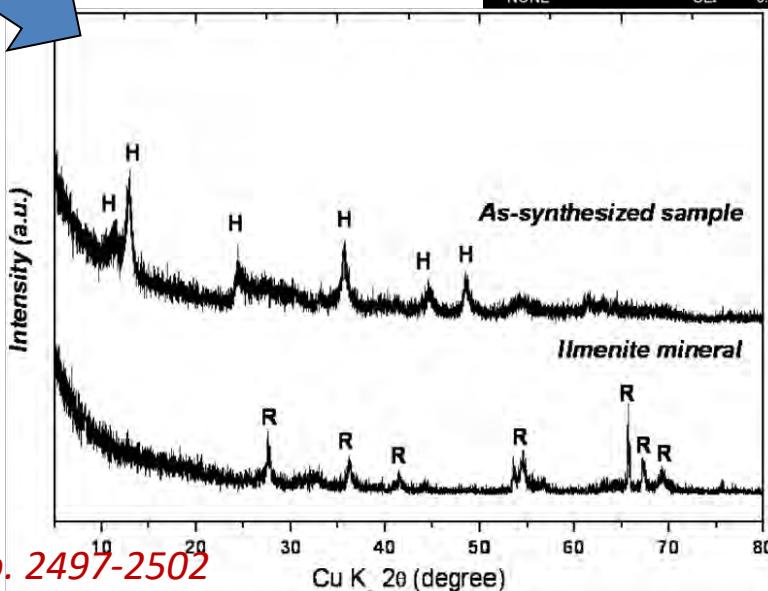
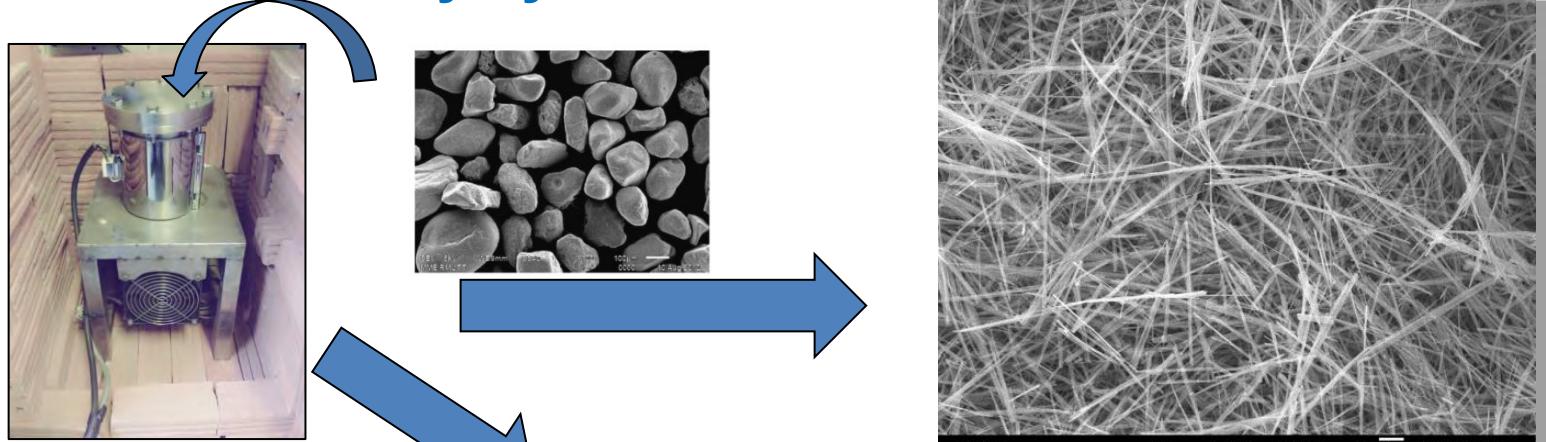
Mg, Al, Mn, Si impurities



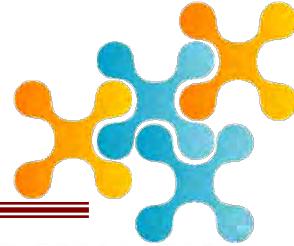
Ti-based natural minerals



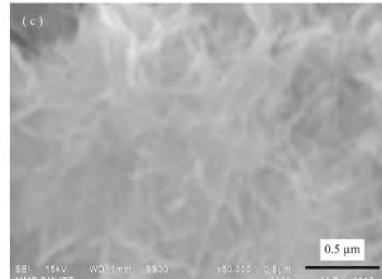
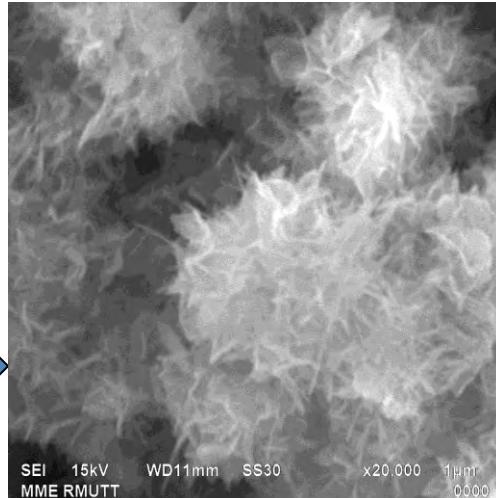
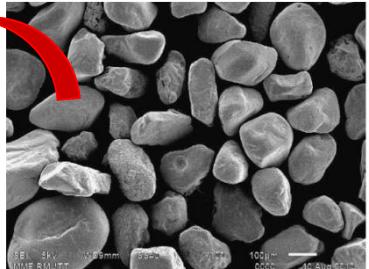
- TiO₂ nanofibers derived from ilmenite mineral by hydrothermal



Ti-based natural minerals



➤ TiO₂ nanosheets from hydrothermal process



SEI 15kV WD11mm SS30

x20.000 1pm
0000

22 Feb 2015

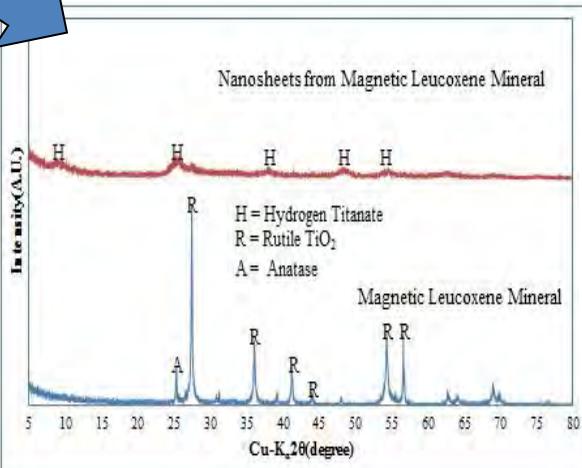
0.5 μm

Leucoxene powder

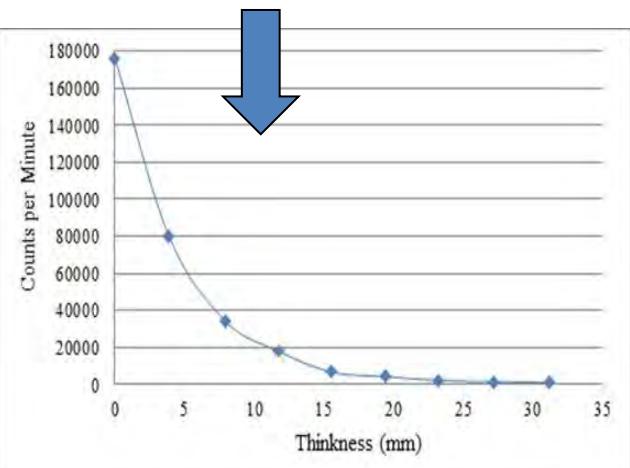
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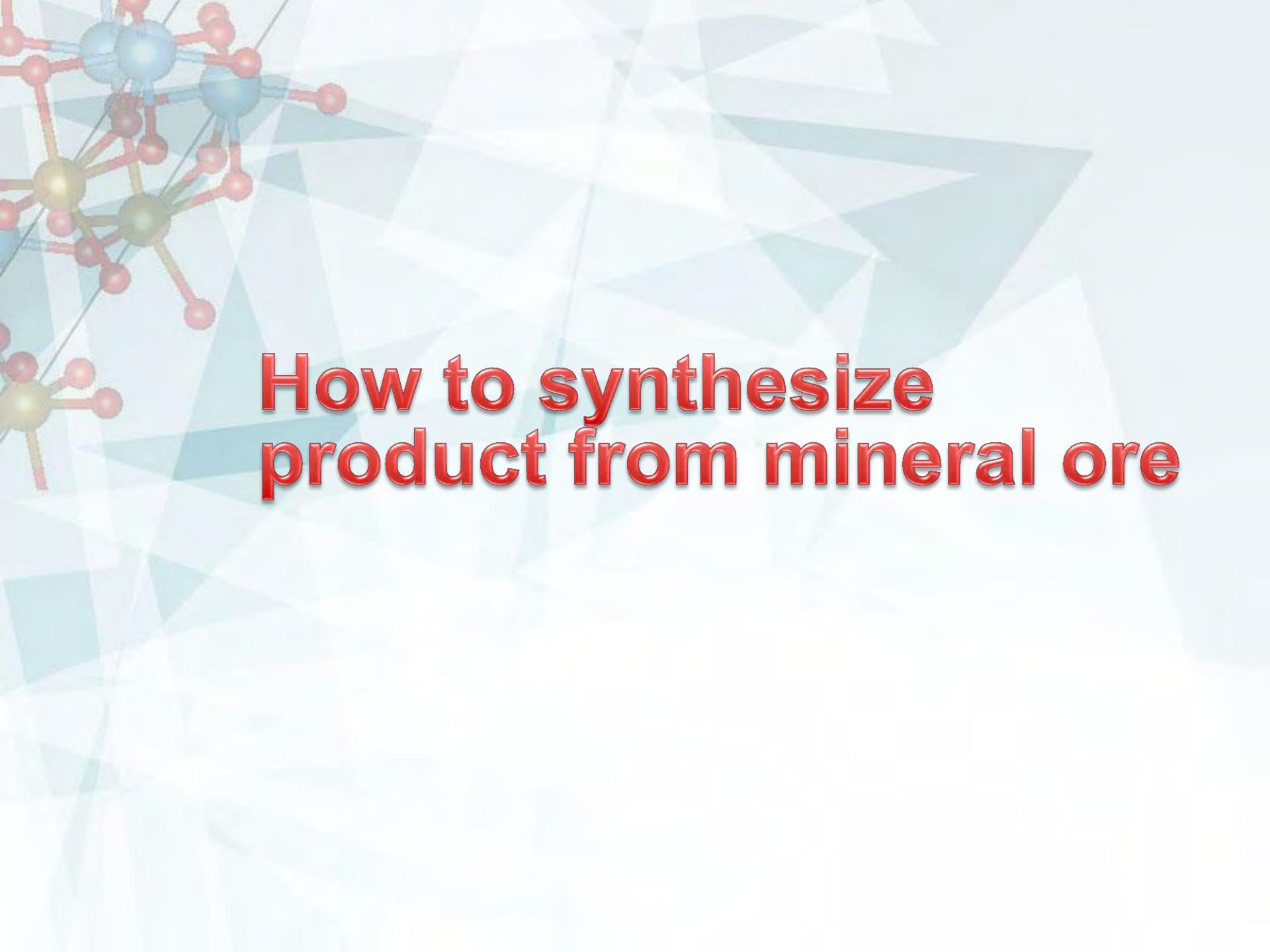
NaOH (5 M)

Hydrothermal
100°C 5 h



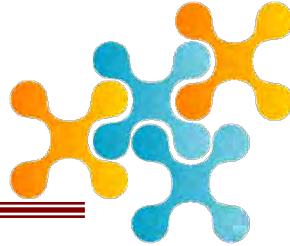
γ-ray shielding property



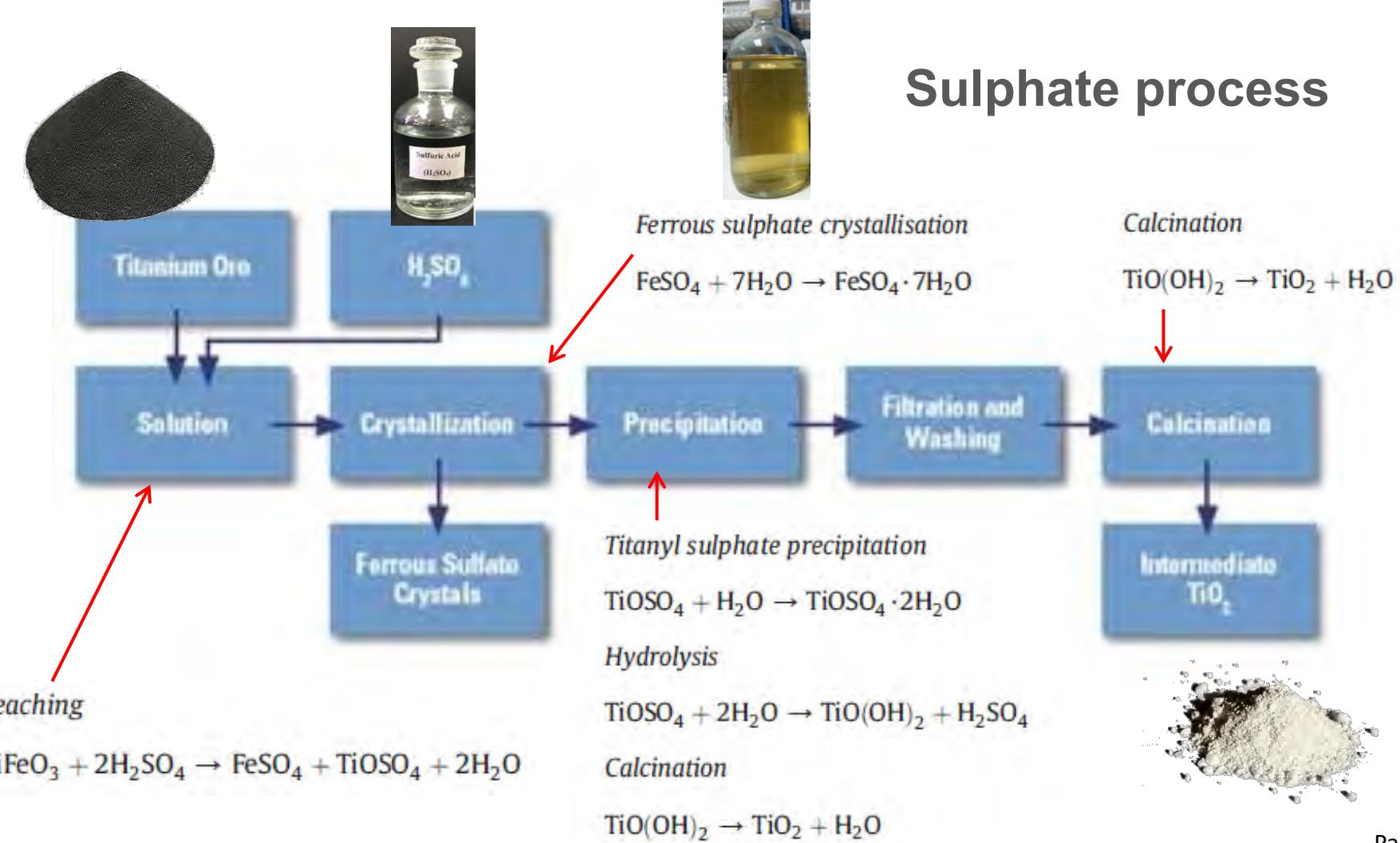


How to synthesize product from mineral ore

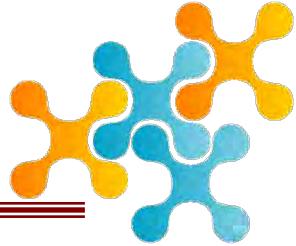
Synthesized process



➤ TiO₂ particles by chemical process

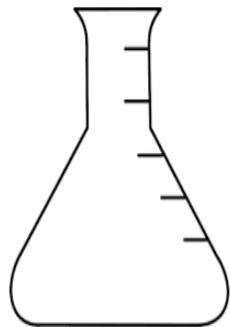


Synthesized process



- Synthetic rutile powder by milling process with ultra-sonic assisted

NSTDA

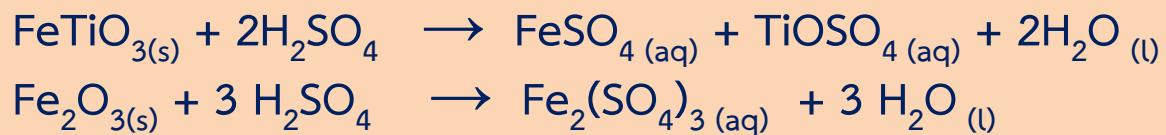


Ultrasonic assisted

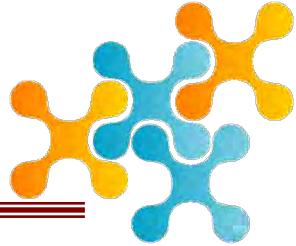


Leaching

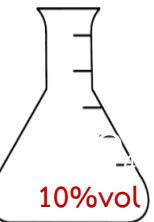
Milling



Synthesized process



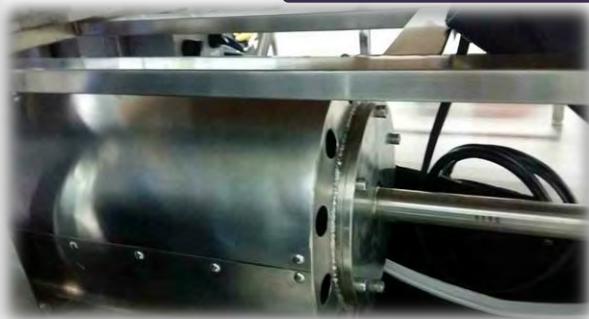
Ultrasonic-assisted process



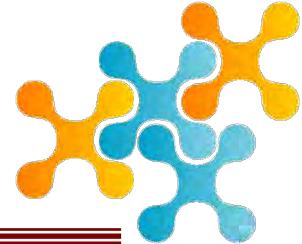
Ultrasonic Probe



Leaching



20 h with ultrasonic for 1 h and 3 h



Results

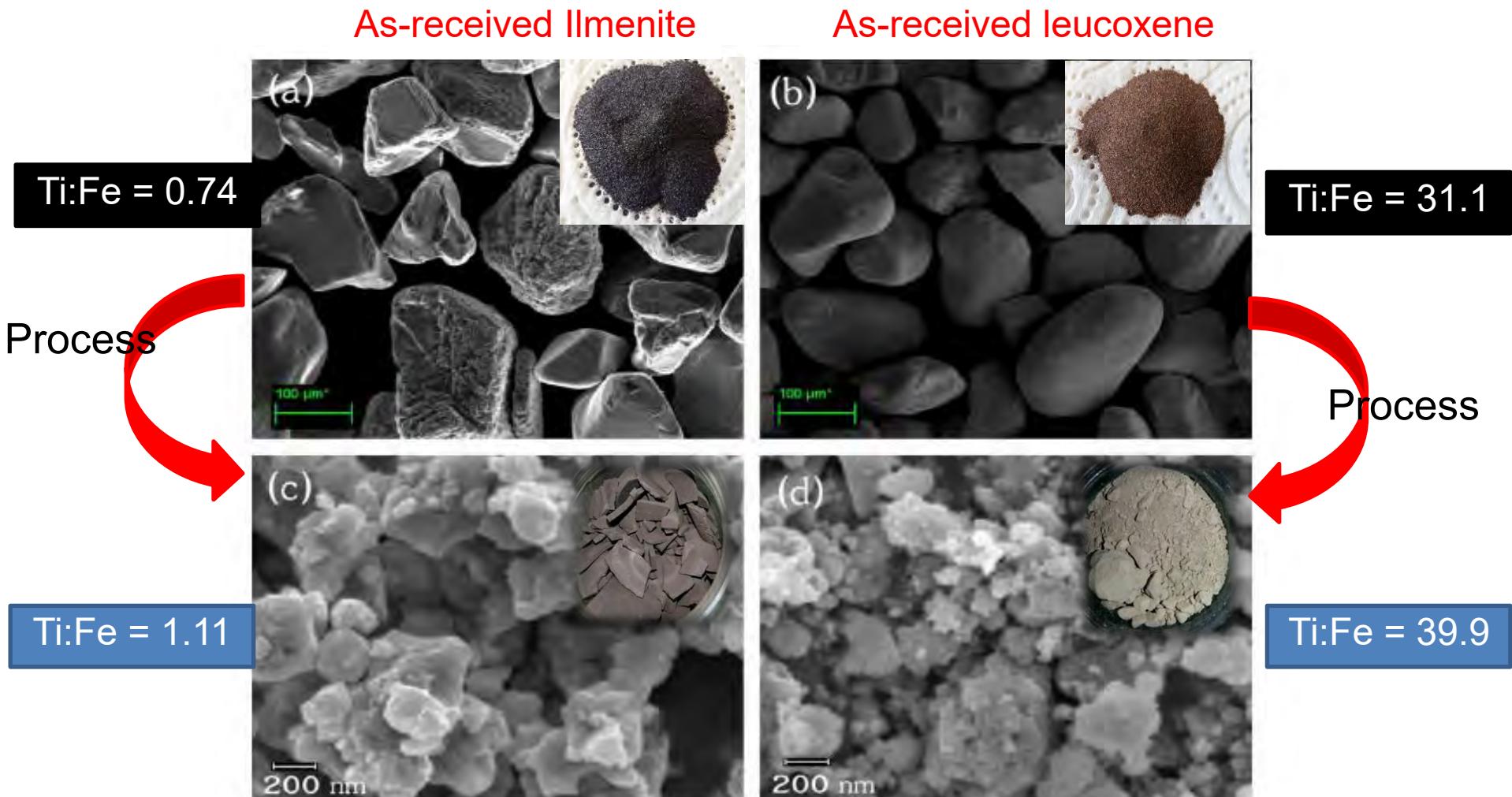
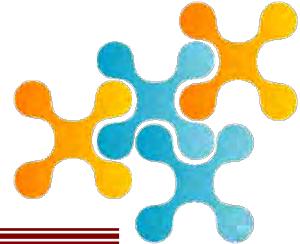


Fig. 1. SEM images of starting raw ilmenite ore (a), leucoxene ore (b), milled-ilmenite (a) and milled-leucoxene (b).



Results

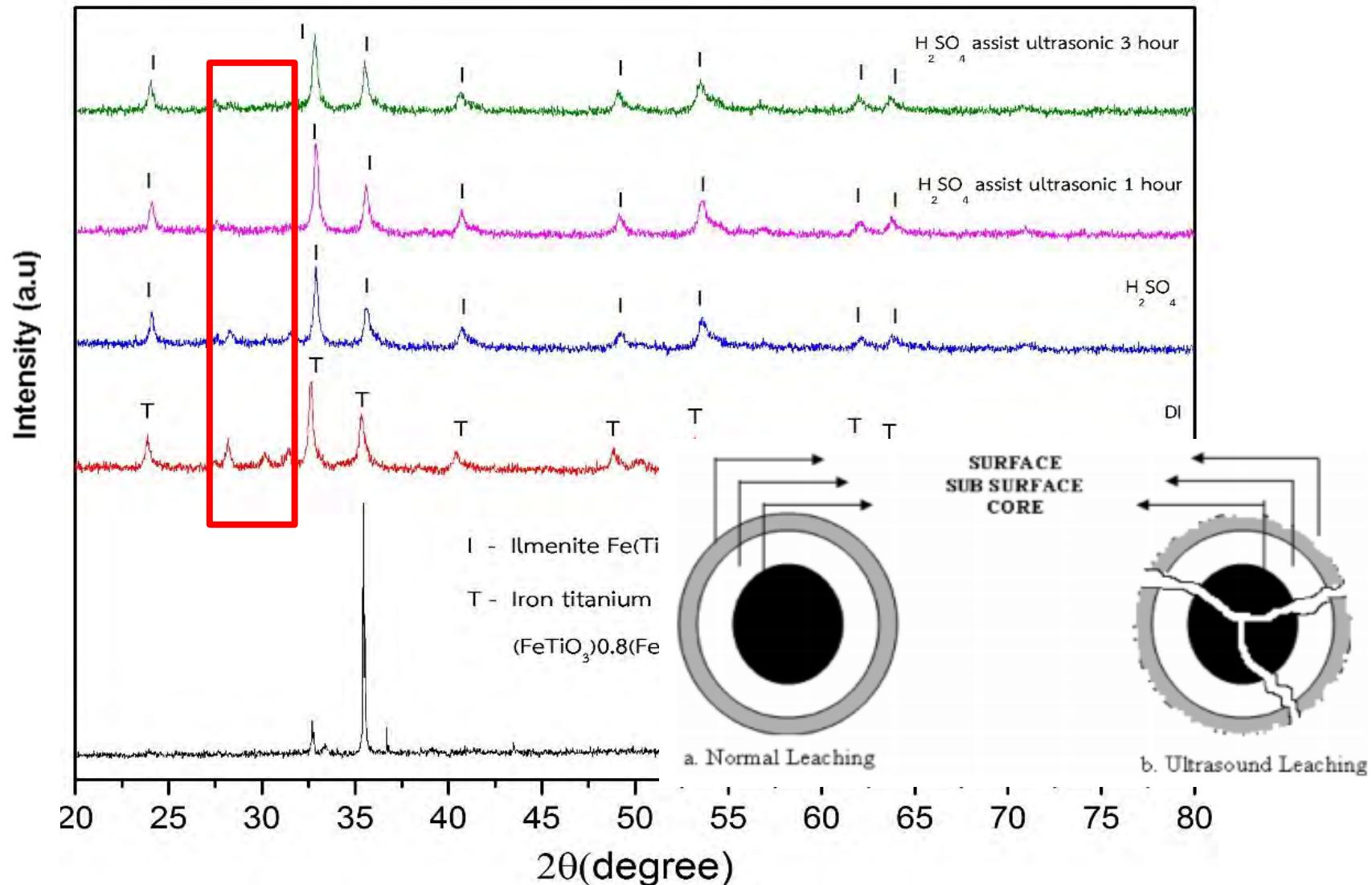
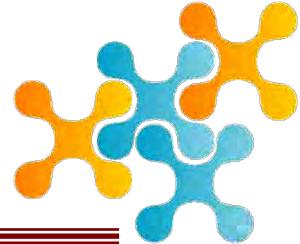


Fig. 2. XRD patterns of the products from **ilmenite ore** at different conditions.



Results

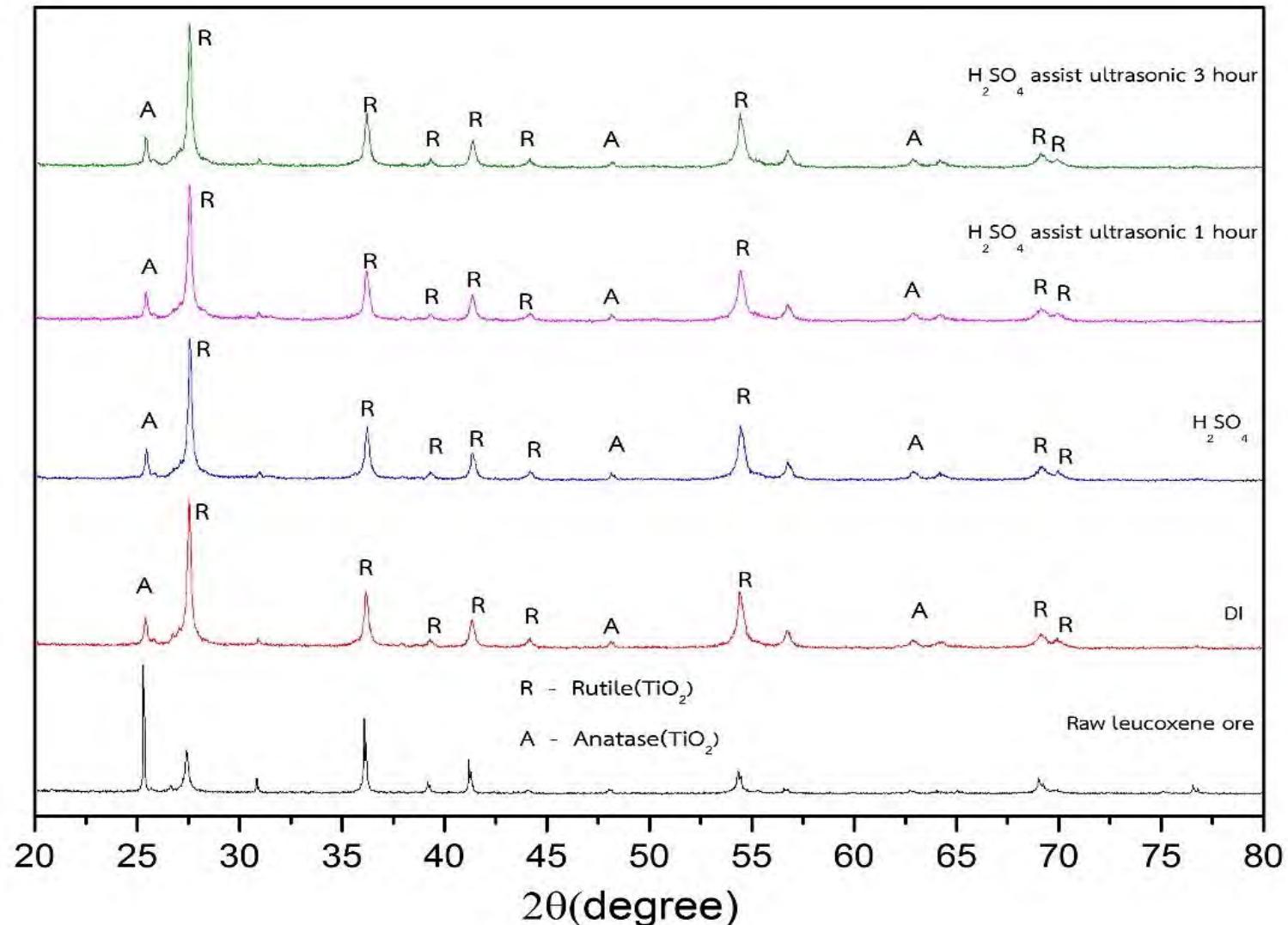
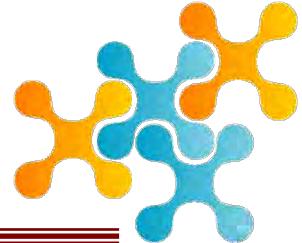


Fig. 3. XRD patterns of the products of **leucoxene ore** at different conditions.



Results

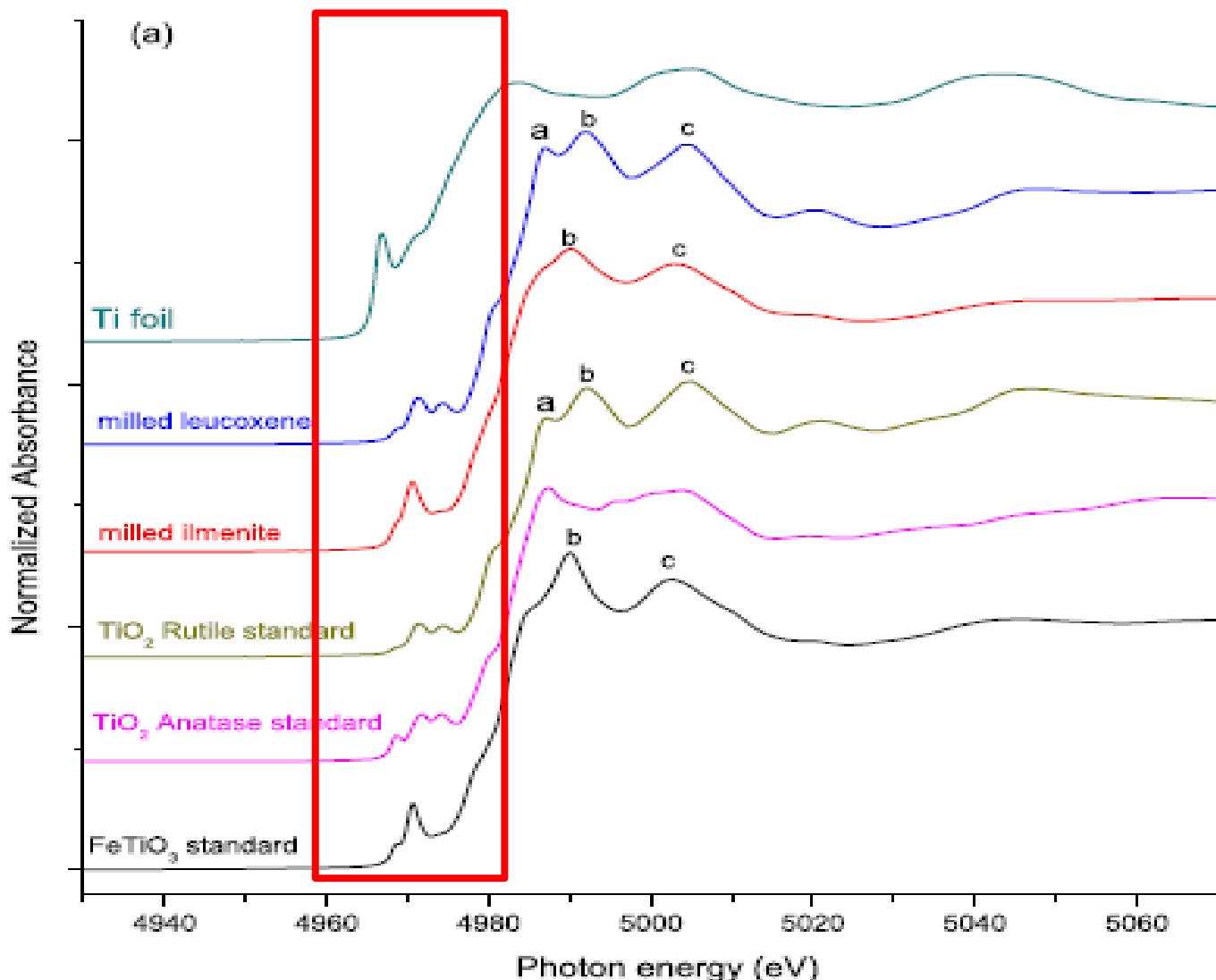


Fig. 4. X-ray absorption near edge spectra (XANES) results.



Possible applications and Proposal project



Possible applications

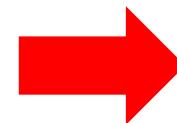
Topic 1: Solid state degradation of plastic by synthetic-rutile/anatase TiO₂ nanocomposite



2 g of Low-density Polyethylene (LPDE)



Dissolving in 50 ml cyclohexane and heated at 70 °C for 1.30 h



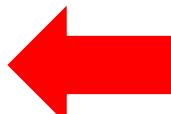
loading 20wt.% synthetic-rutile/anatase TiO₂ composite at 0, 0.5, 1 and 2 wt.%

Mixed by high energy ball milling machine operated at 400 rpm for 30 min

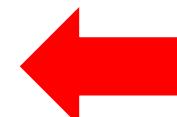
Stirred for 30 min at 50 °C for homogenous solution

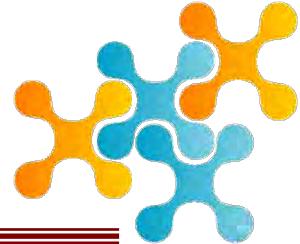


LPDE composite film



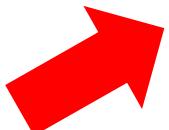
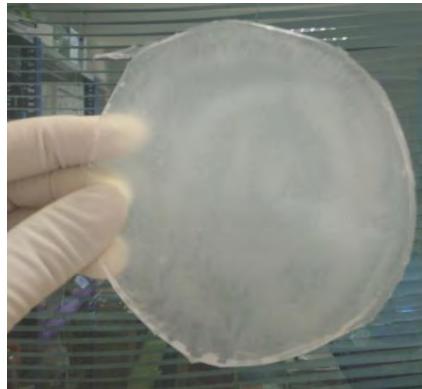
Poured 10 ml of the solution on petri dish and dry at 40 °C





Possible applications

- Topic 1: Solid state degradation of plastic by synthetic-rutile/anatase TiO_2 nanocomposite



Light simulator
Xenon lamp
irradiation for 50 h
(Japan)

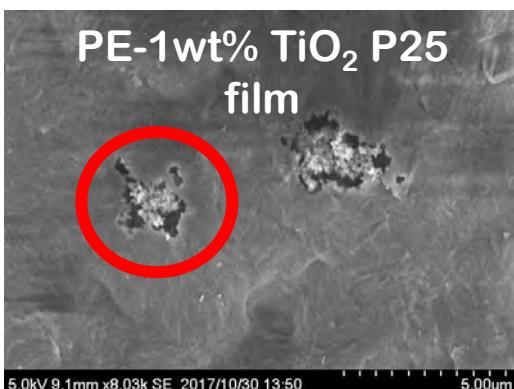
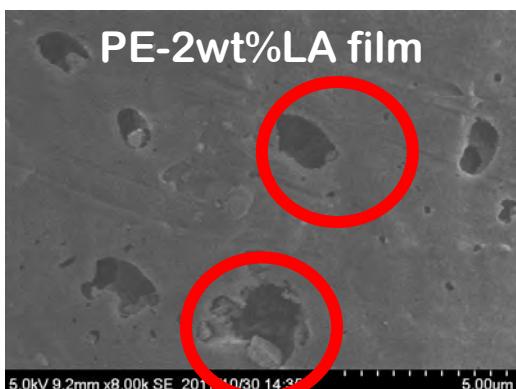
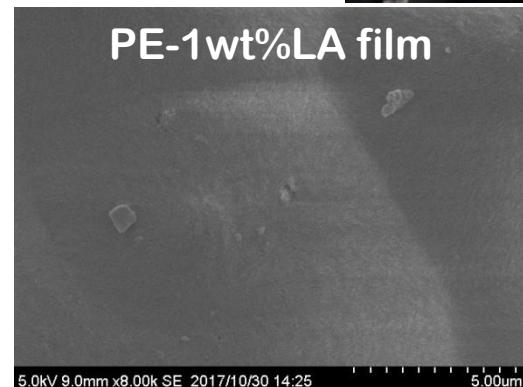
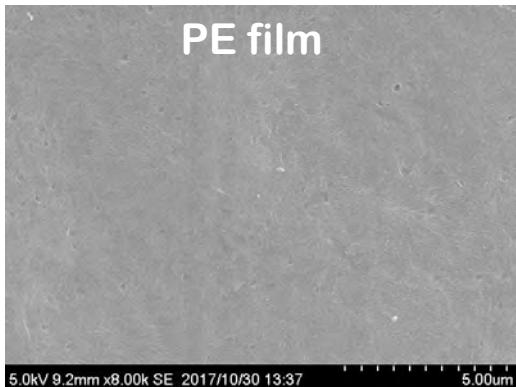
Plastic composite films



Solar light
irradiation for 40 days
(Thailand)

Possible applications

- Topic 1: Solid state degradation of plastic by synthetic-rutile/anatase TiO_2 nanocomposite



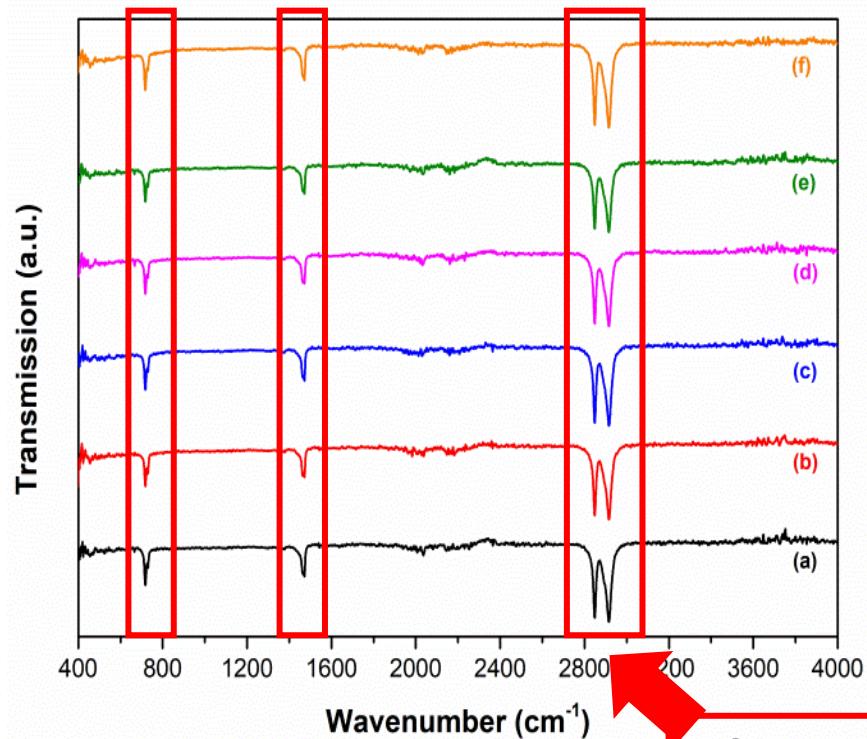
SEM images of PE films with synthetic-rutile/anatase TiO_2 nanocomposite loading after Xe lamp irradiation for 50 h

Possible applications

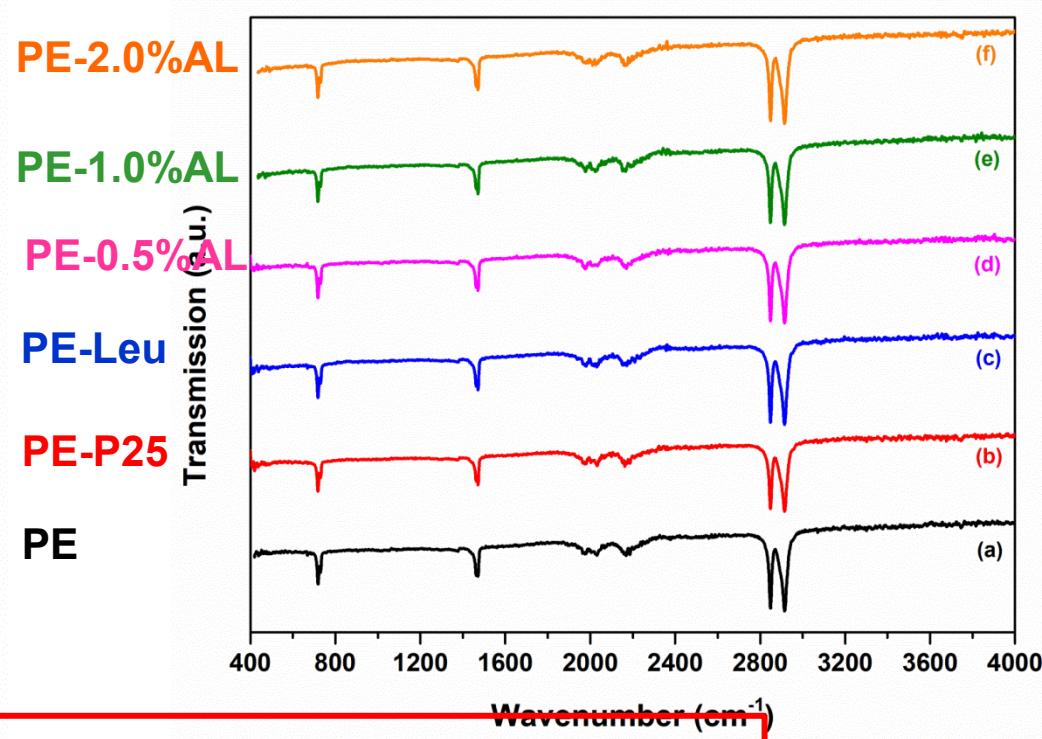
- Topic 1: Solid state degradation of plastic by synthetic-rutile/anatase TiO_2 nanocomposite



Before light irradiation



After light irradiation (50 hr)



CH_2 stretching and bending vibration

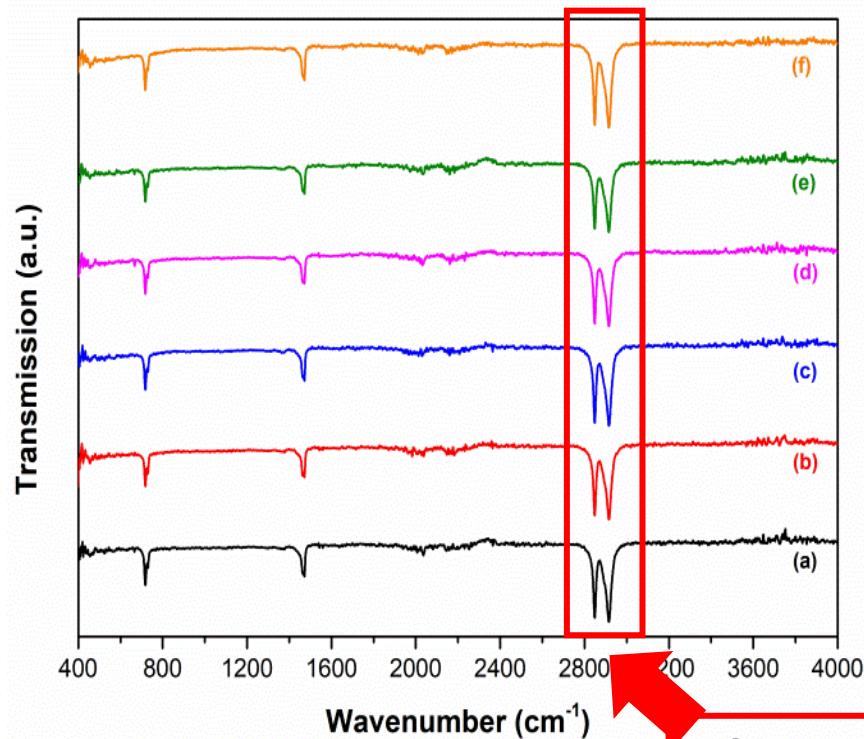
FTIR Spectra: PE- TiO_2 nanocomposite films before-after Xe lamp illumination

Possible applications

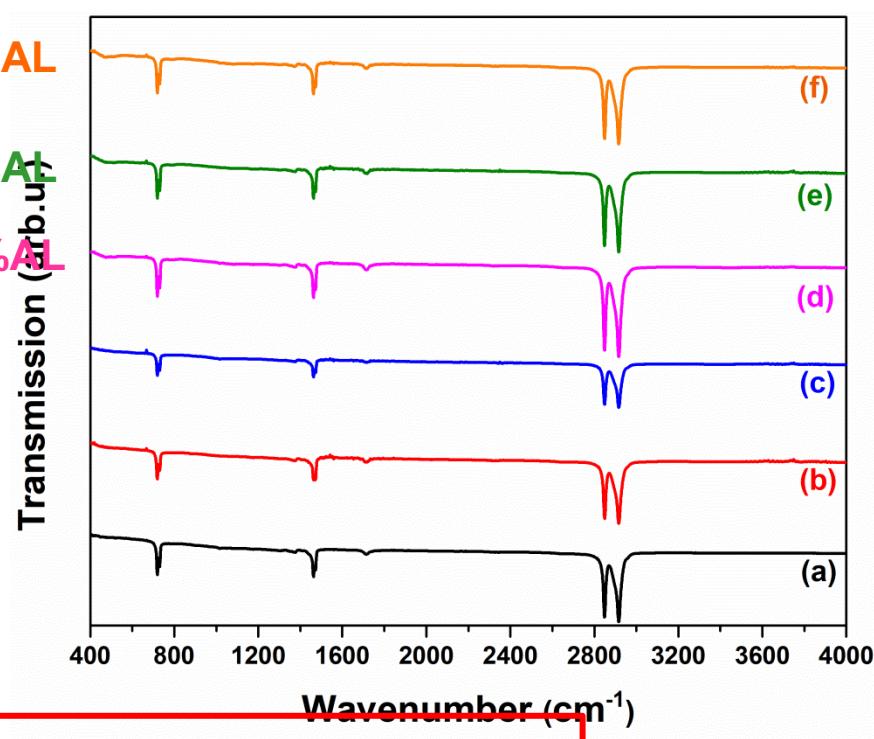


- Topic 1: Solid state degradation of plastic by synthetic-rutile/anatase TiO_2 nanocomposite

Before light irradiation

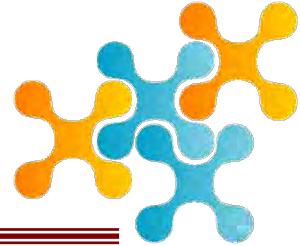


After light irradiation (1 month)



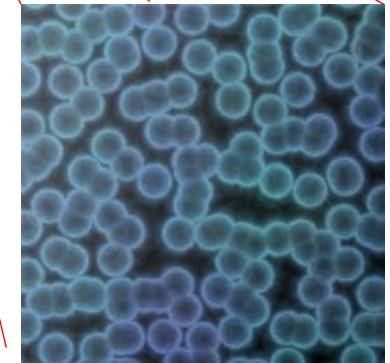
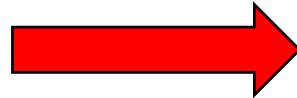
CH_2 stretching and bending vibration

FTIR Spectra: PE- TiO_2 nanocomposite films before-after solar light illumination



Possible applications

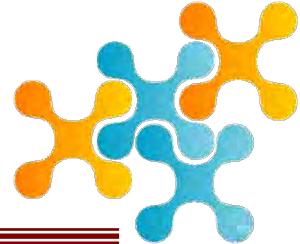
- Topic 2: Enhancement efficiency of natural-mineral-derived TiO₂ nanoparticles for earthenware-roof tile coating material



<http://www.wisegeek.com/what-are-ceramic-tiles.htm>

<http://www.gettyimages.co.jp/detail>

https://www.zazzle.co.uk/blue_bacteria_background_small_square_tile-227996258974984605



Possible applications

Topic 2: Enhancement efficiency of natural-mineral-derived TiO₂ nanoparticles for earthenware-roof tile coating material

Leached residue



Binder solution

- Polyethylene glycol (PEG)
- Monoaluminum phosphate (MAP)

Dip coating

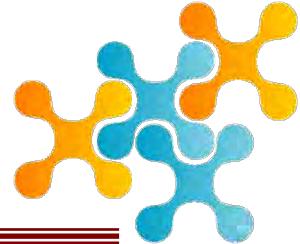
Coating process



Characterizations

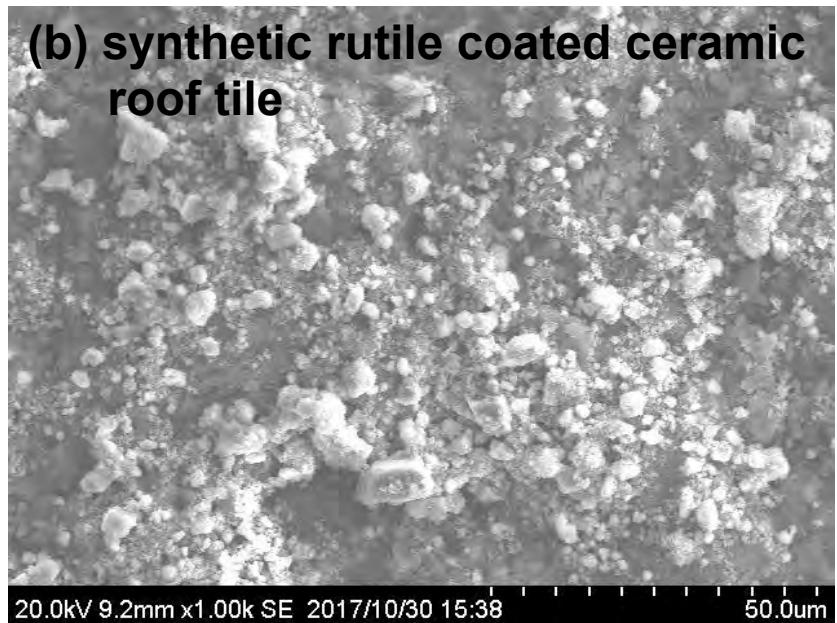
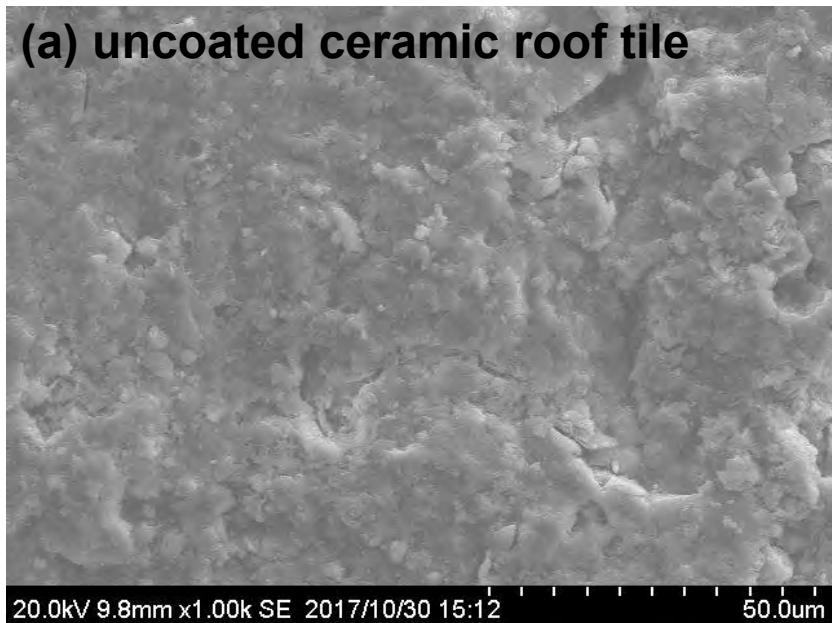


- Optical property (DRS)
- Morphology (SEM)
- Chemical elements (EDX)
- Hydrophilic and hydrophobic
- Photocatalytic property

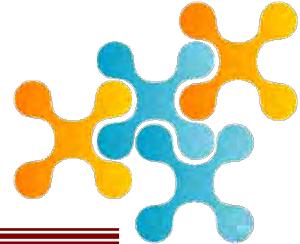


Possible applications

- Topic 2: Enhancement efficiency of natural-mineral-derived TiO₂ nanoparticles for earthenware-roof tile coating material



Surface morphologies by SEM images of (a) uncoated ceramic roof tile and (b) synthetic rutile coated ceramic roof tile.

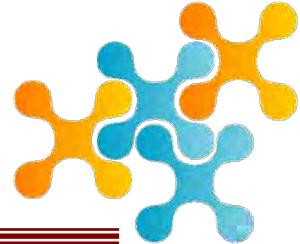


Possible applications

- Topic 2: Enhancement efficiency of natural-mineral-derived TiO₂ nanoparticles for earthenware-roof tile coating material

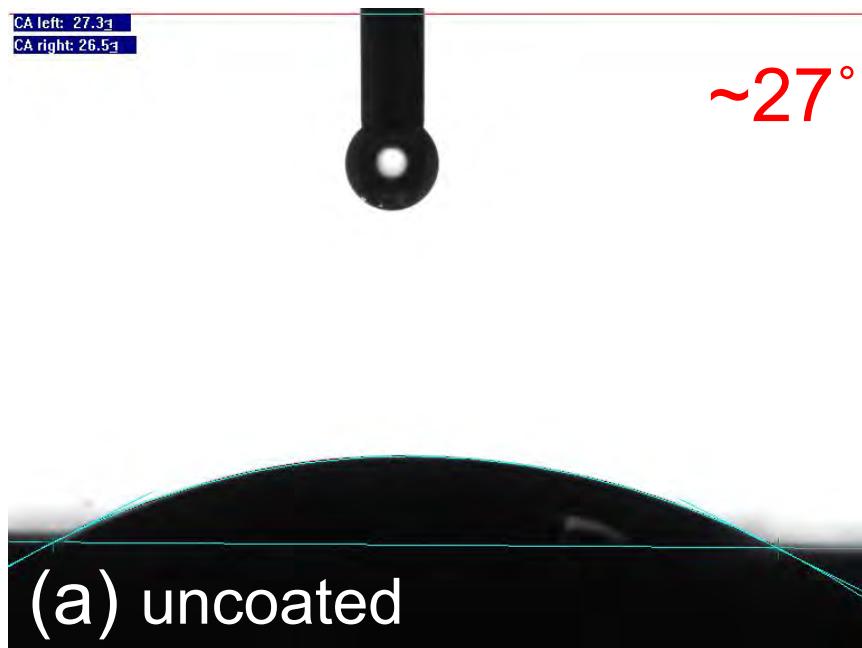
Table 1: Chemical elements of TiO₂ derived from leucoxene ore coated on ceramic roof tile

Element (%wt)	Specimens	
	Uncoated	Coated
Silicon	24.96	16.84
Aluminum	12.99	11.79
Oxygen	44.93	39.09
Iron	5.63	5.13
Titanium	0.48	17.85
Gold	5.50	5.72
Magnesium	1.33	0.63
Calcium	1.91	0.49
Potassium	2.22	2.42



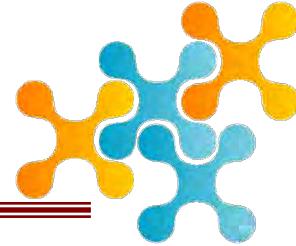
Possible applications

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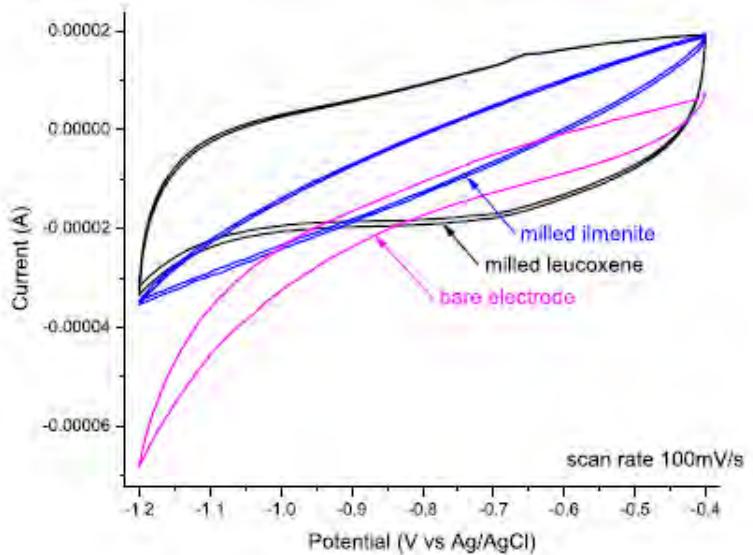
Contact angle measurement for hydrophilic and hydrophobic testing on (a) uncoated and (b) coated ceramic tile under non-irradiation.

Possible applications

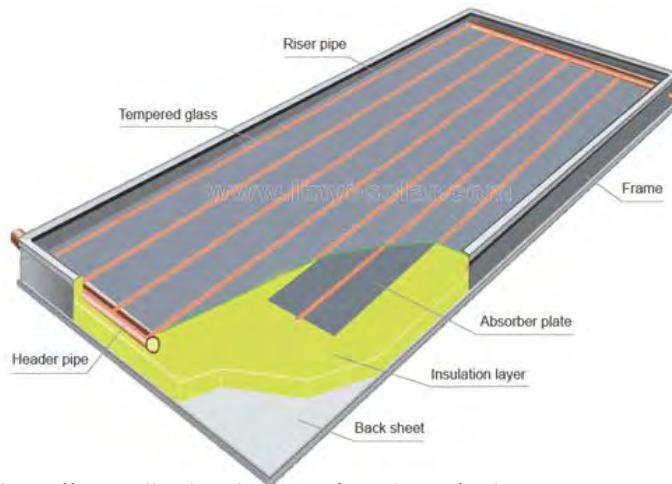
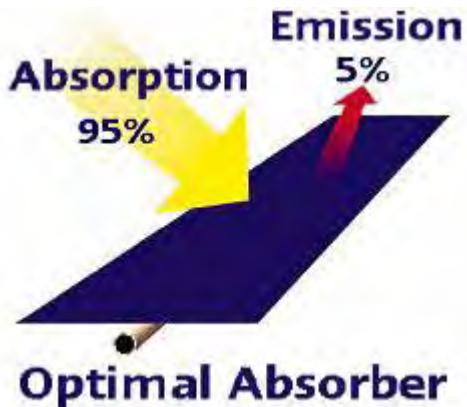


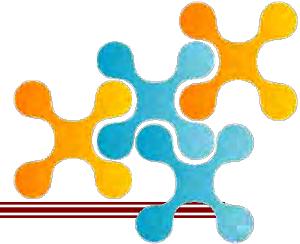
⊕ Research proposal for energy storage

- Material for supercapacitor



- Material for solar absorber

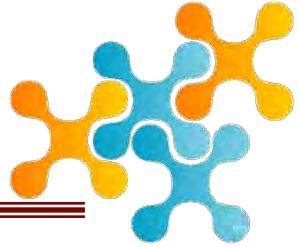




Conclusion

- This work was carried out to synthesize activated ilmenite and leucoxene from natural ores by sulfuric acid leaching-assisted ultrasonic ball-milling process.
- Impurity phases in as-received ores were significantly leached after increasing ultrasonic time and milling in acid solution corresponding with the increase of absorbance spectra from separated solution.
- The optimized condition of synthetic rutile/anatase nanocomposite loading in PE films performed the improvement of plastic degradation efficiency.
- The possibility of self-cleaning surface can be proposed by coated synthetic rutile on earthenware tile.

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- Nanomaterial and applications
- Energy and environment materials
- Sensors, sensing materials and related devices
- Materials for health science
- Optical and electronic materials
- Metal alloys and composite materials
- Material process and manufacturing

Abstract due: Feb 23, 2018



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