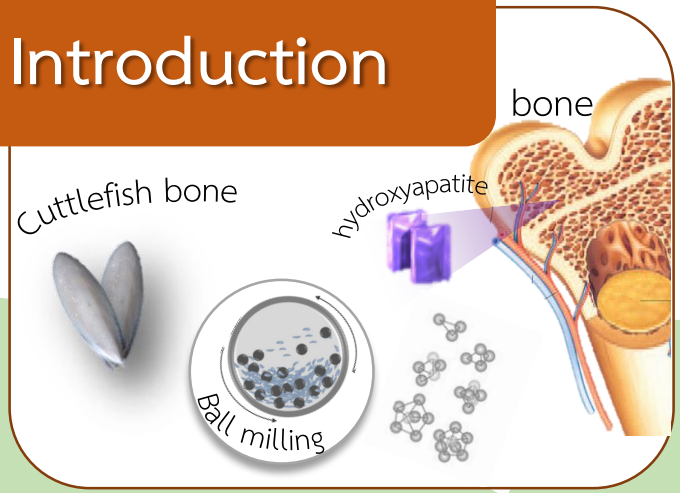
Characteristic and Formation of Hydroxyapatite Synthesized from Heat Treatment of Cuttlefish Bone

Kridsada Faksawat

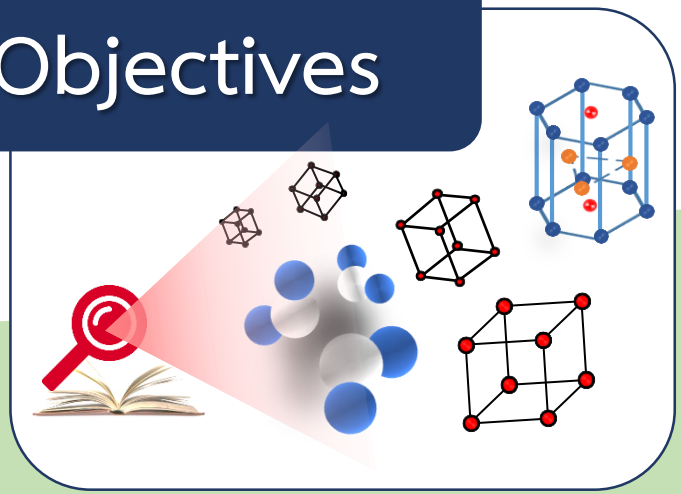
Department of Physics, Faculty of Science,
King Mongkut's University of Technology Thonburi

Outlines

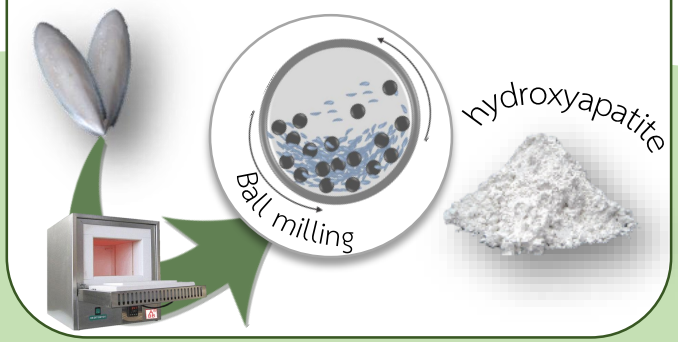
Introduction



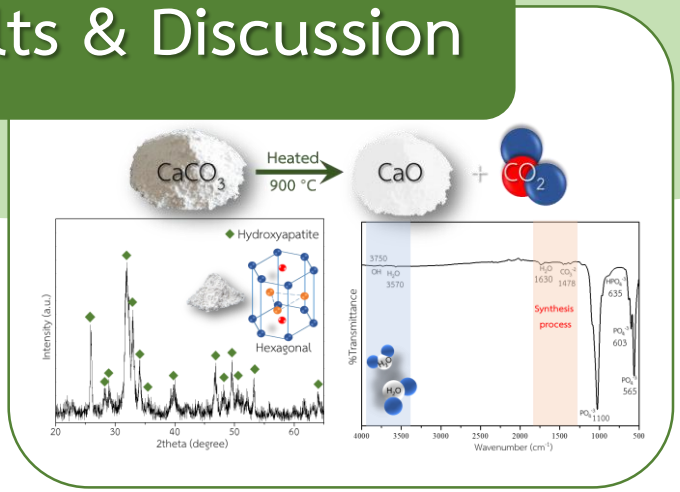
Objectives



Experiment



Results & Discussion

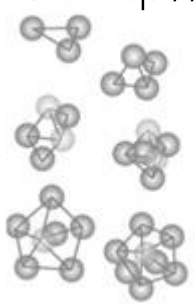


Conclusion

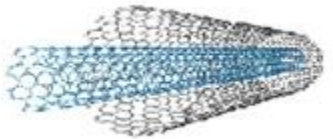
Introduction

Ref: http://commonsensecanadian.com

Ref: http://eng.thesaurus.rusnano.com



0 Dimension



1 Dimension

2 Dimension

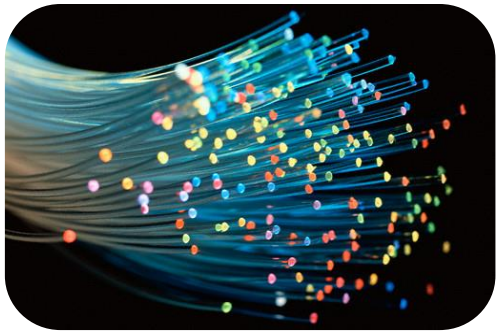
3 Dimension

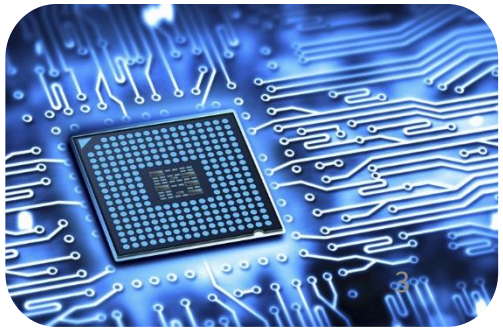
- **Nano scale**

  1 to 100 nanometers

- **Shape and Size Effect**

  Increasing of specific surface area



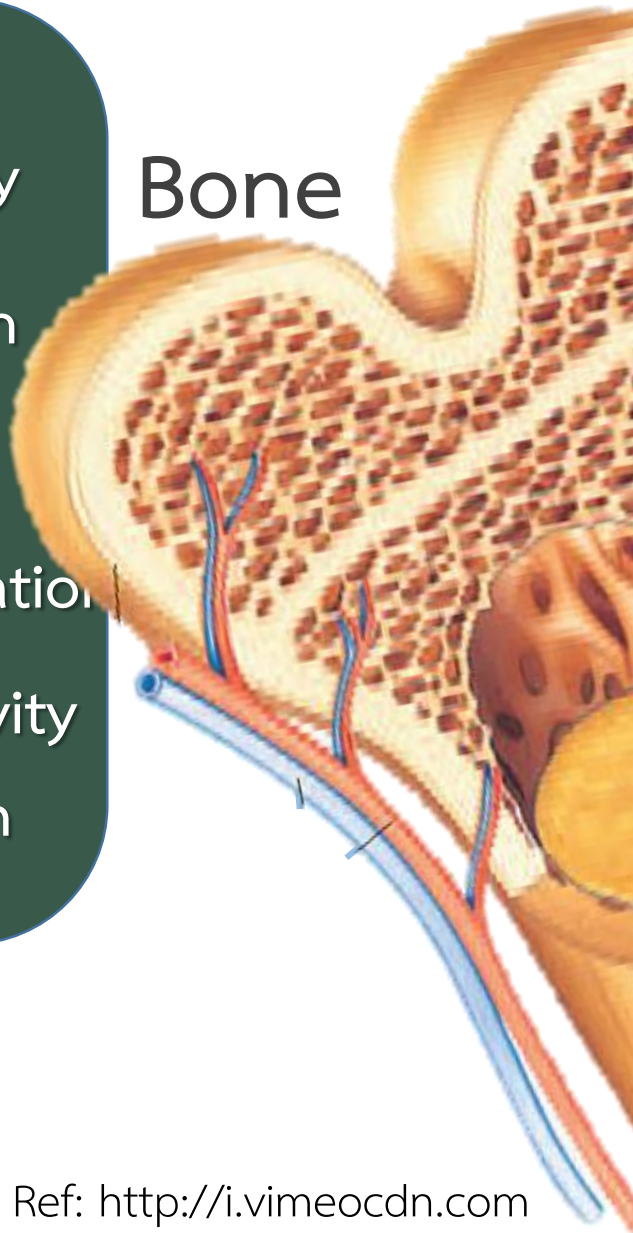


Introduction

Ref: http://onco-info.ru

- ✓ Enhanced resorbability
- ✓ Improved densification
- ✓ sinter ability
- ✓ Improved cell proliferation
- ✓ Improved cellular activity related to bone growth

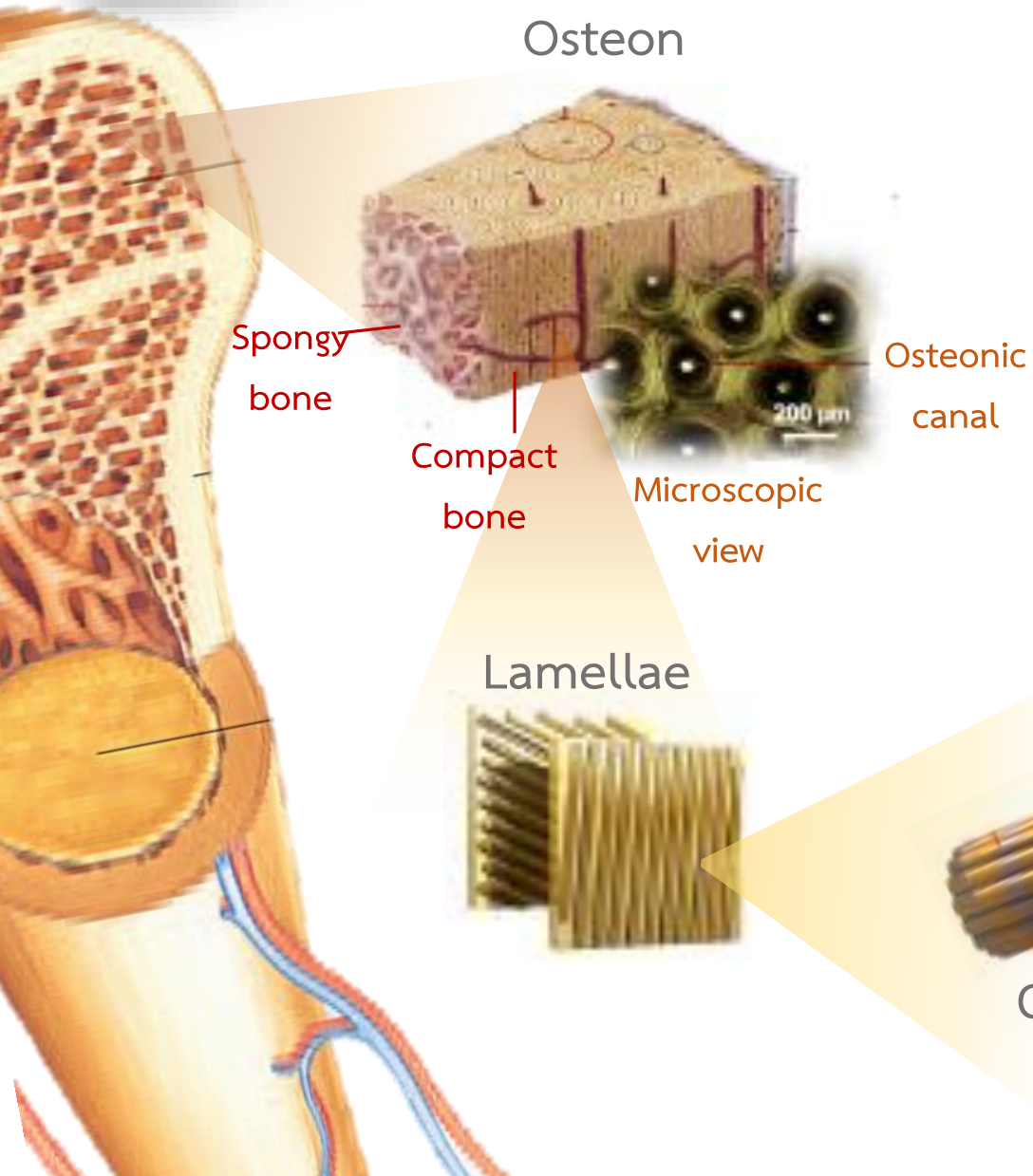
Bone



Ref: http://i.vimeocdn.com

Hydroxyapatite

Introduction



Osteon

Spongy bone

Compact bone

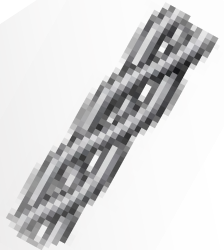
Osteonic canal

Microscopic view

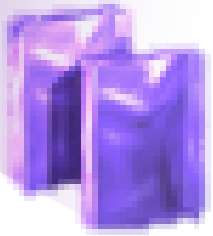
Lamellae

Collagen fiber

Mineralized fibrils



Collagen triple helix



Hydroxyapatite

$Ca_5(PO_4)_3OH$

Hexagonal

Bio ceramics

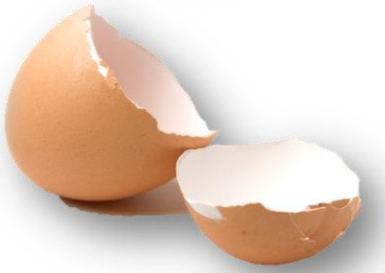
Inorganic mineral

Introduction

How do you synthesized hydroxyapatite?

- Natural material

90-95% of Ca



Hydroxyapatite

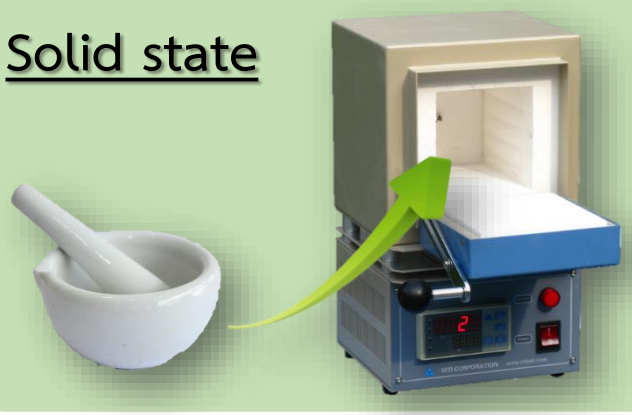
- Chemical material

K_2HPO_4	$Ca_2P_2O_7$	Na_2HPO_4	$(NH_4)_2HPO_4$
Trace of K	Found β -TCP	Trace of Na	Odor problem

Introduction

How to synthesis of hydroxyapatite

Solid state



Ball milling

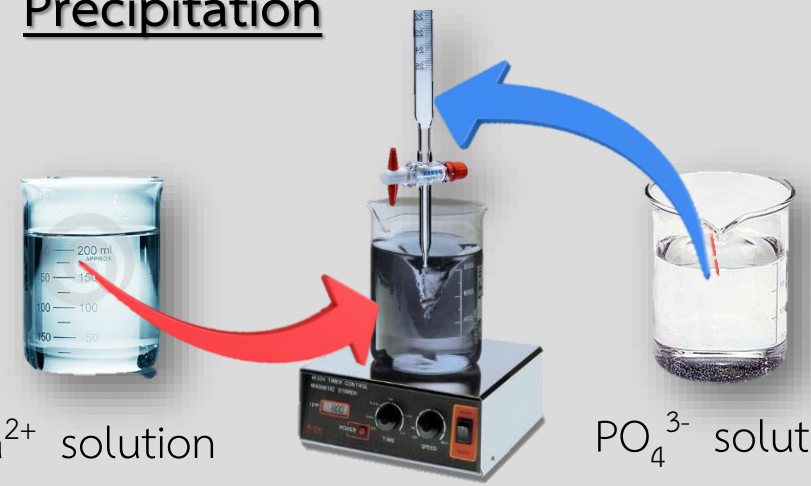
Microwave irradiation

Hydroxyapatite

Sol gel

H_2O/EtOH

Precipitation

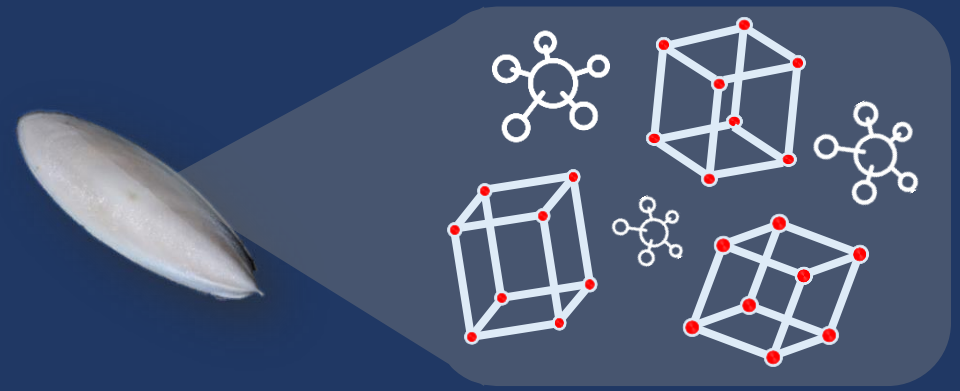


Objectives

- To study phase transformation of cuttlefish bone by various sintering temperature.
- To study characteristics of hydroxyapatite synthesized from various heated cuttlefish bone by ball milling method.
- To study crystal structure, functional group and morphology of synthesized hydroxyapatite by ball milling method.

Experiment

Part 1. Temperature effect on cuttlefish bone

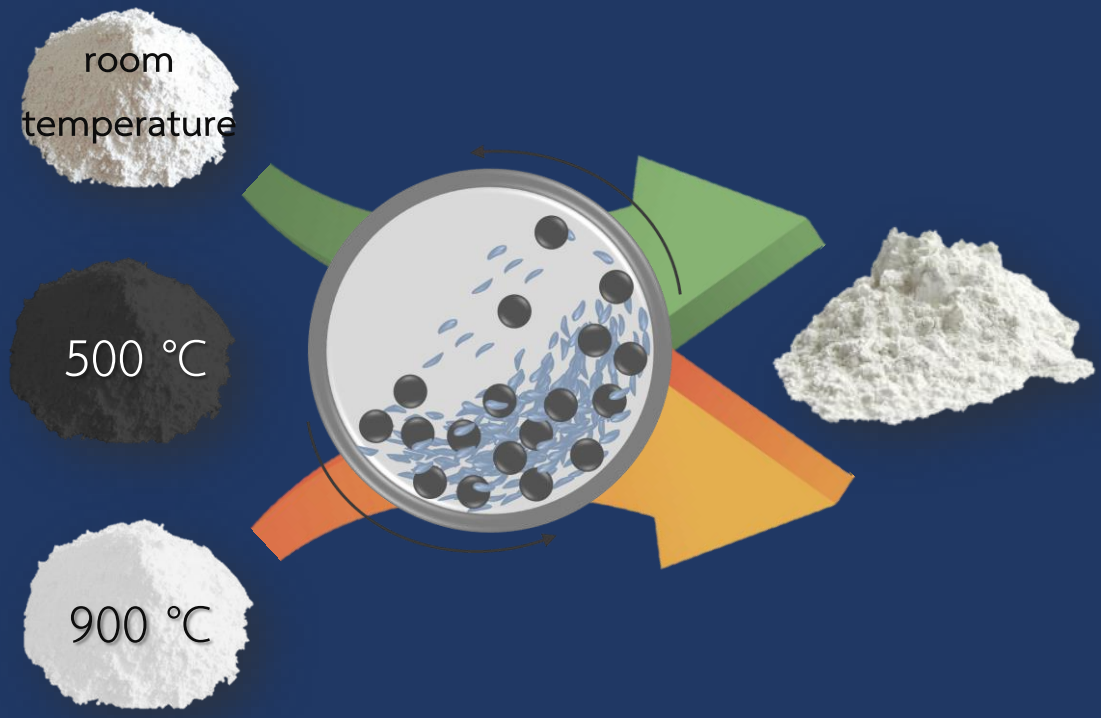


✓ 200 to 1300 ˚C

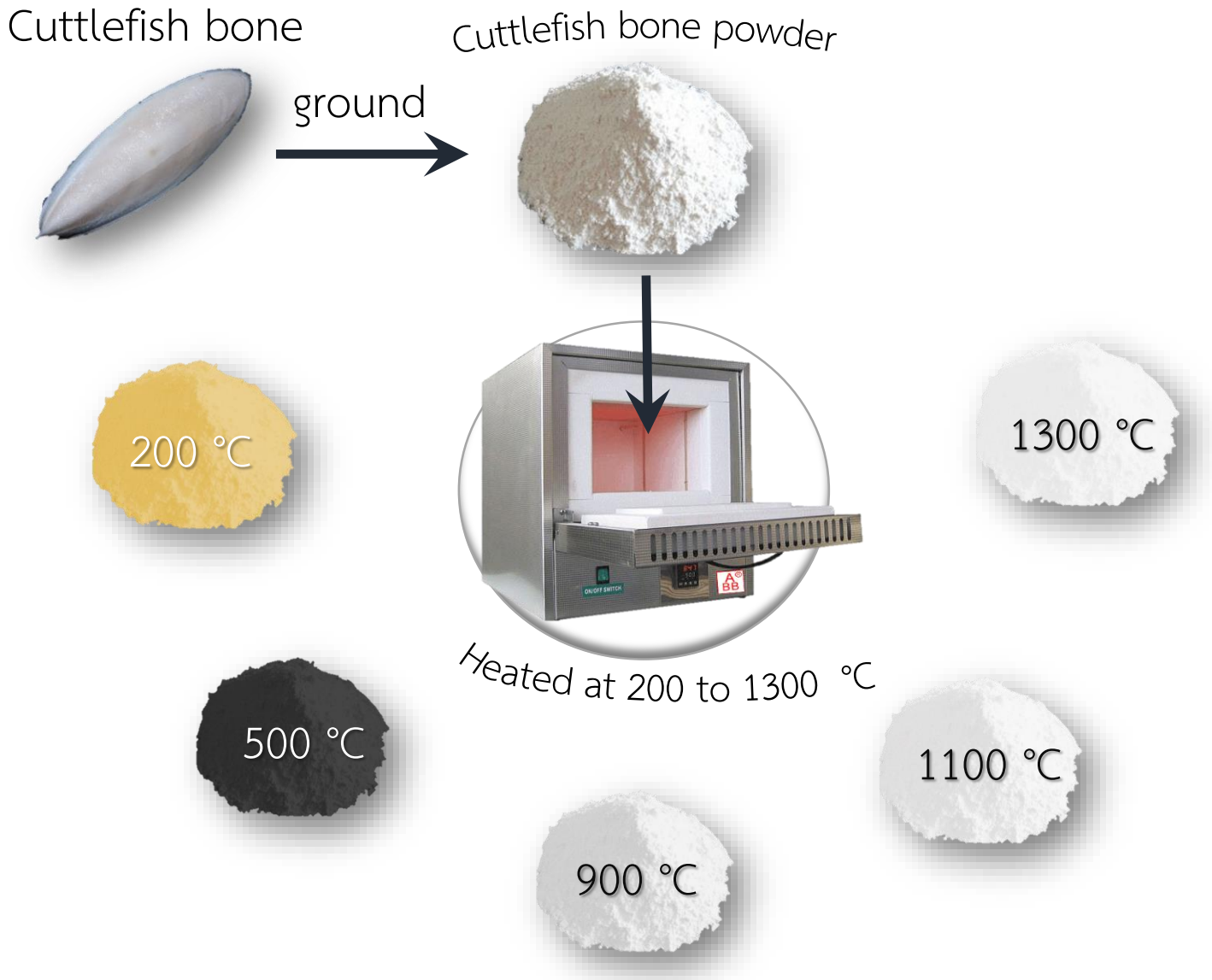
Part 2. Hydroxyapatite synthesis

- Ball milling method

✓ Vary precursor



Experiment: Part 1 Temperature effect on cuttlefish bone



Part 1

Temperature effect on cuttlefish bone

Characterization by XRD

Hydroxyapatite synthesis

Results & Discussion: Part 1 Temperature effect on cuttlefish bone

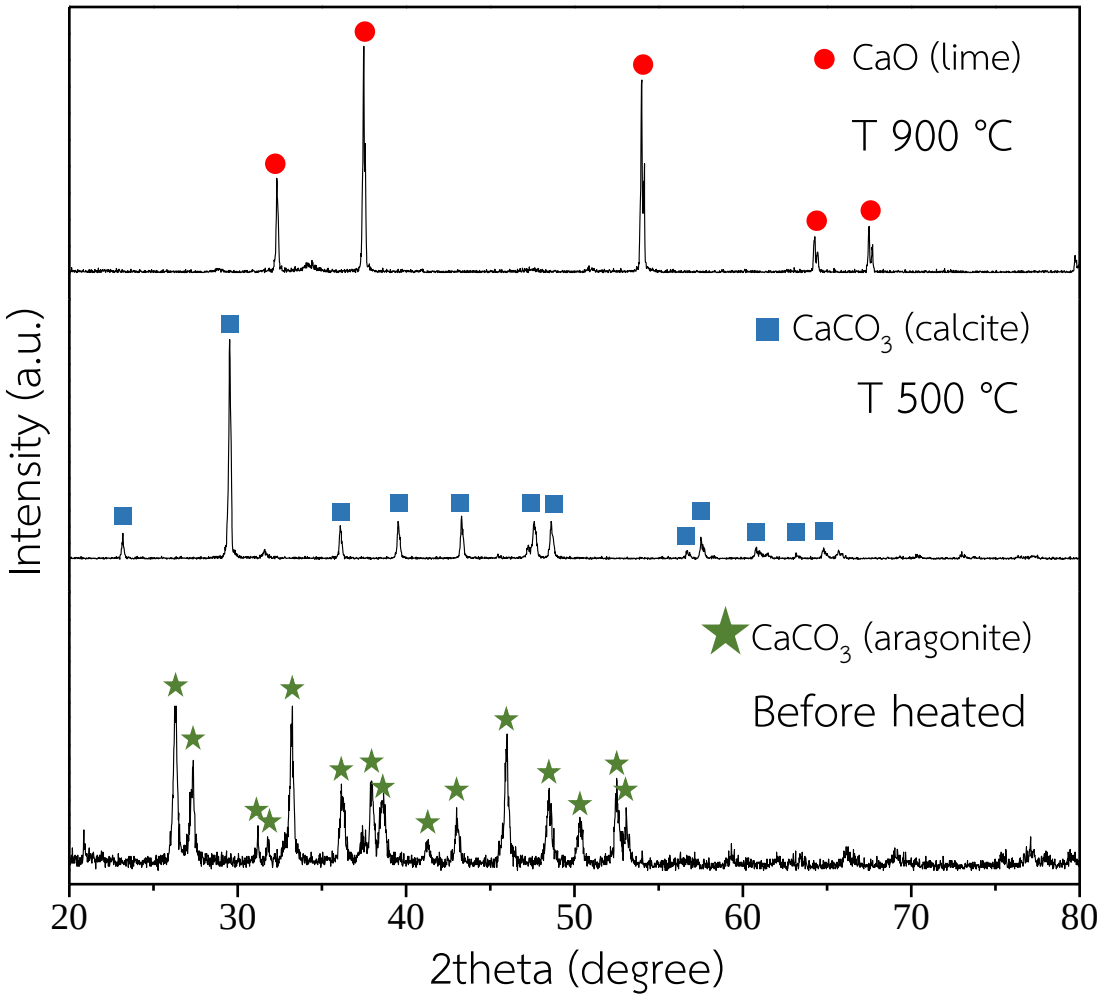
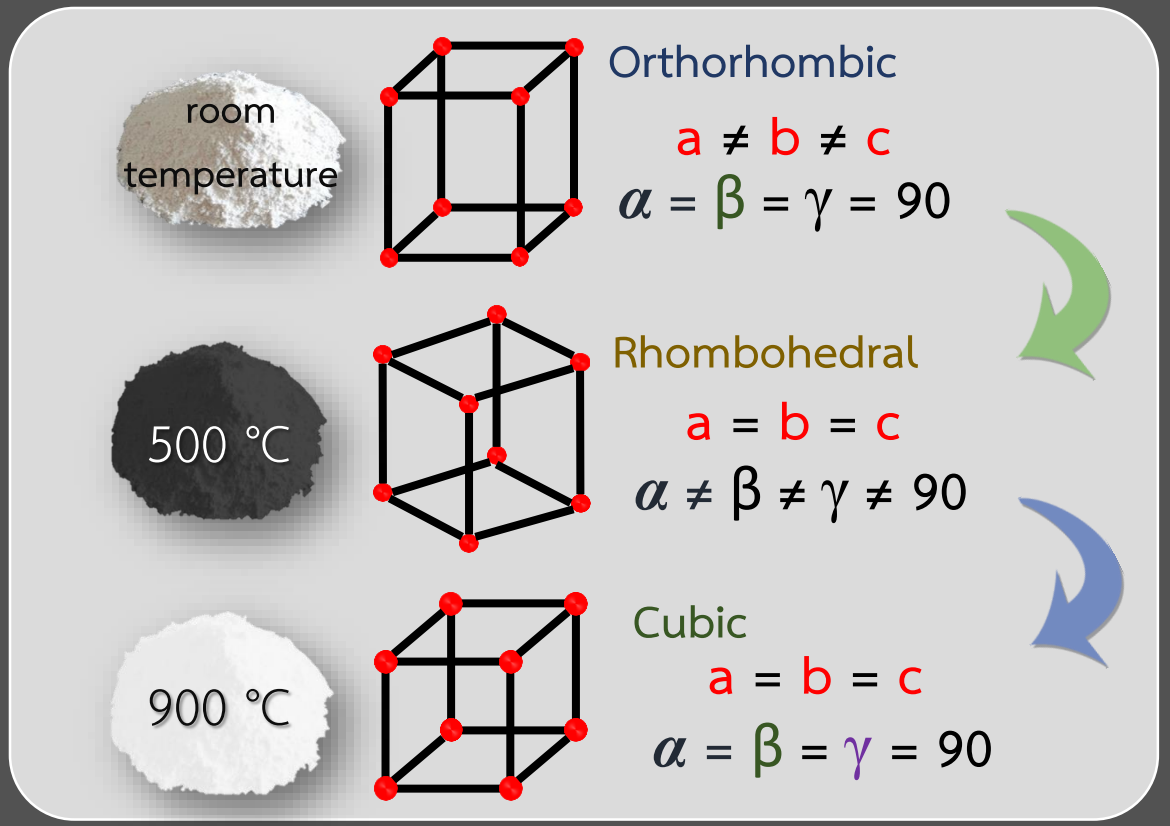
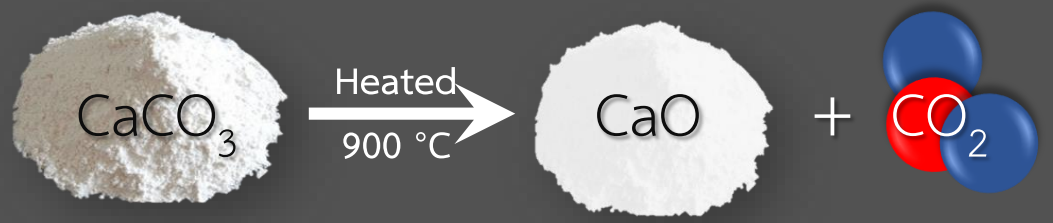


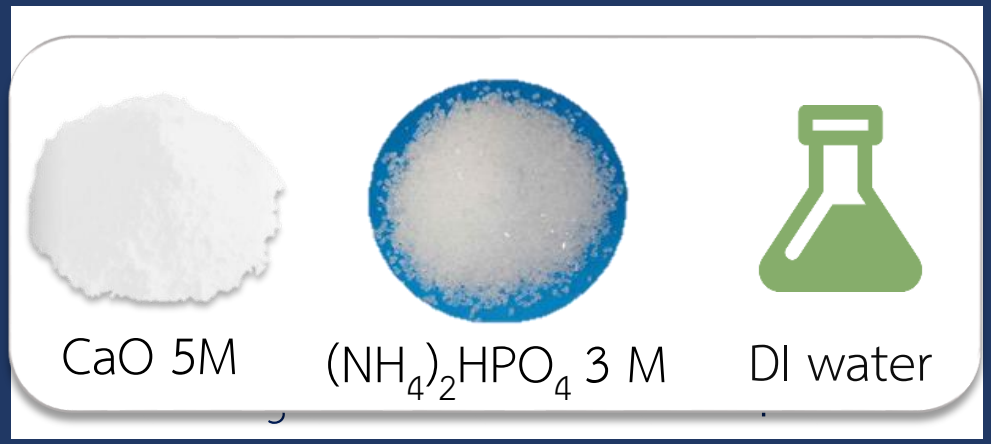
Fig 1 XRD pattern of temperature effect on cuttlefish bone at various temperature

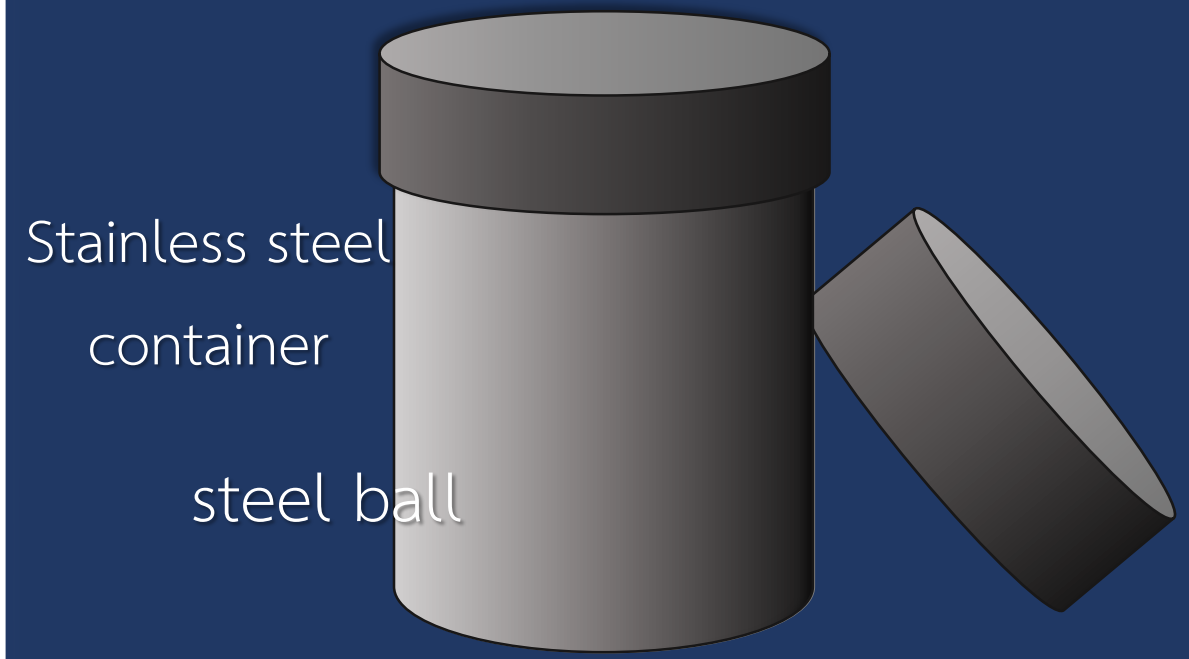


Thermal decomposition of $CaCO_3$

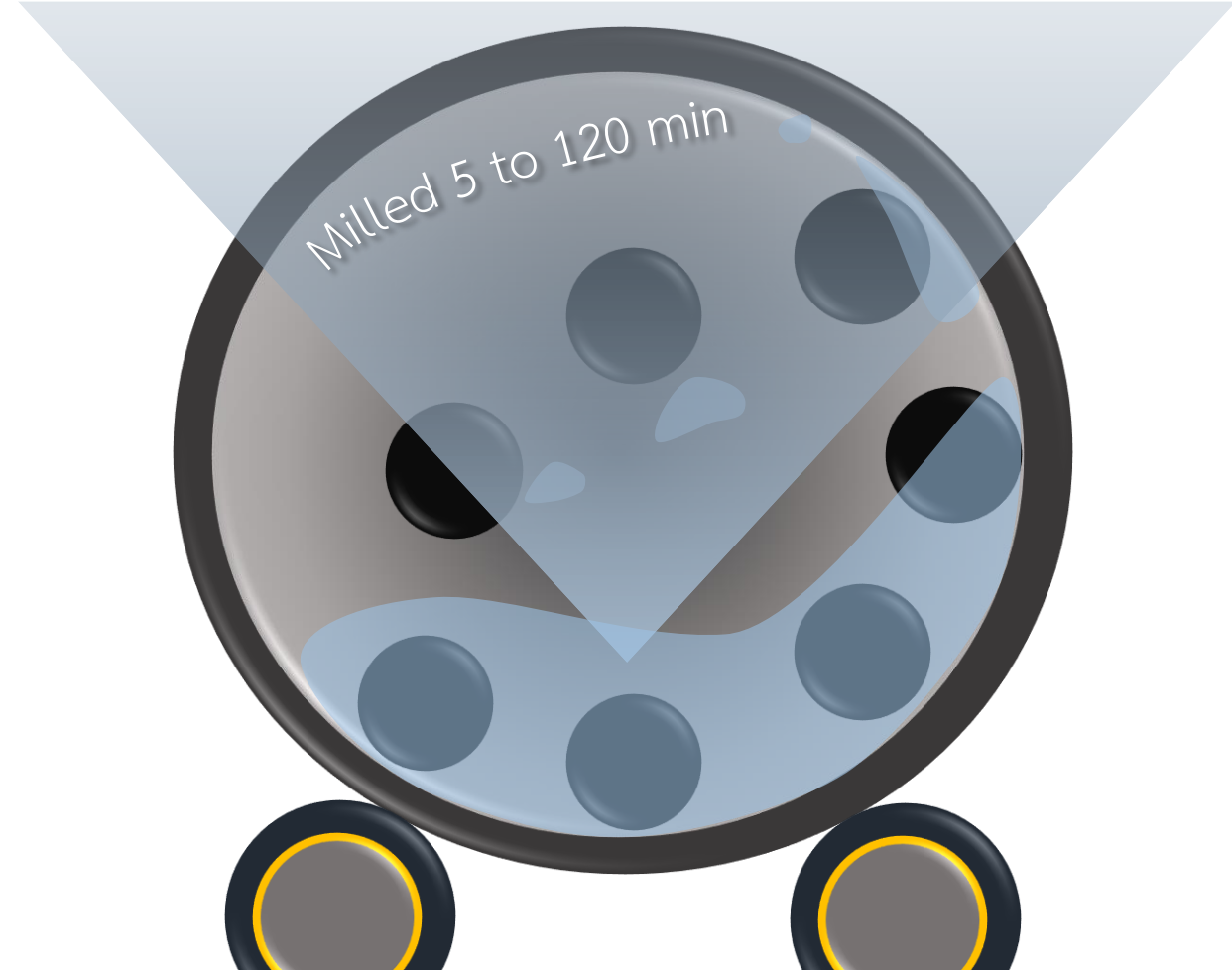


Experiment: Part 2 Ball Milling

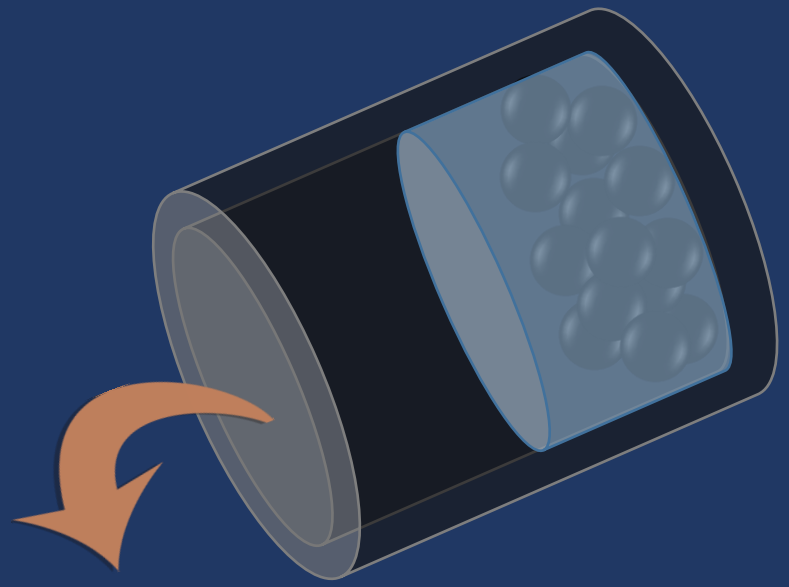


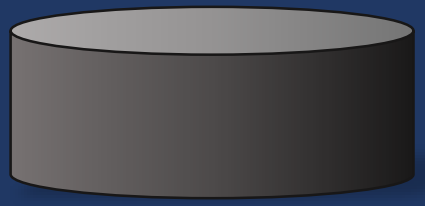


$$5CaO + 3(NH_4)_2HPO_4 + H_2O \longrightarrow Ca_5(PO_4)_3(OH) + 5H_2O + 6NH_3$$



Experiment: Part 2 Ball Milling





sample

Ground until powder

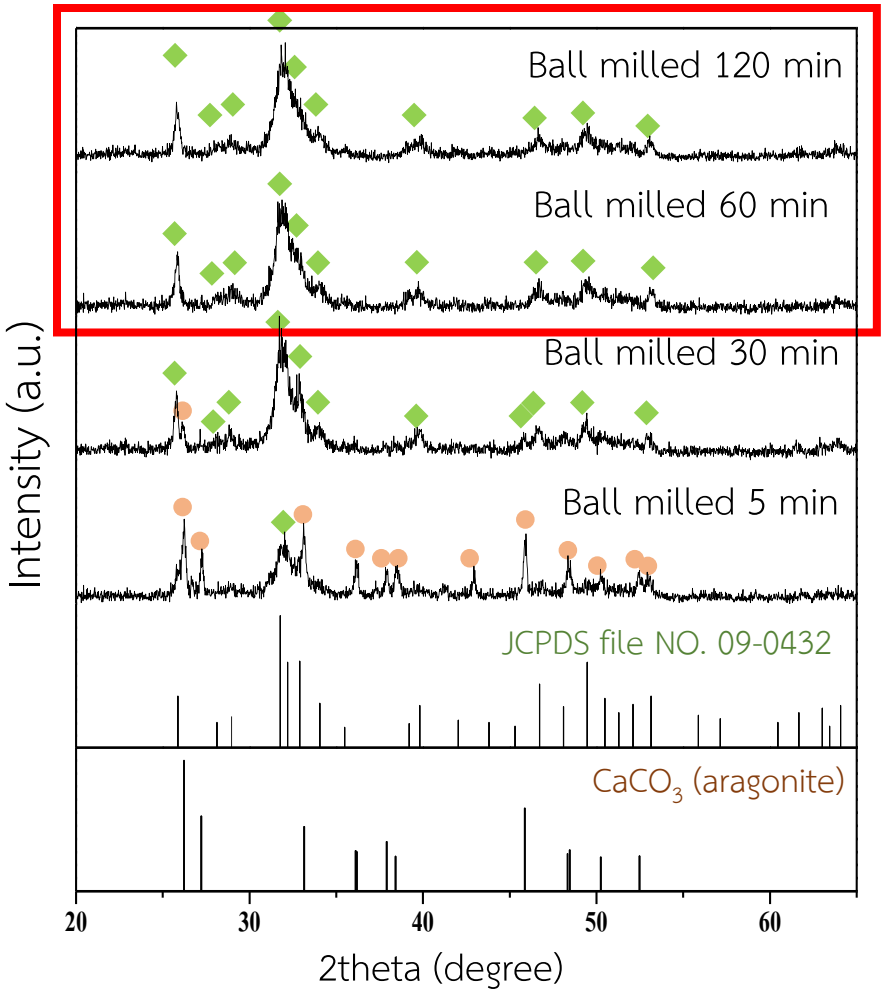
Hydroxyapatite

Characterization

- ✓ X-ray diffractometer: XRD
- ✓ Fourier transform infrared spectrometer: FTIR
- ✓ Scanning electron microscopy: SEM

Results & Discussion: Part 2 Hydroxyapatite synthesis by ball milling

hydroxyapatite $CaCO_3$ (aragonite)

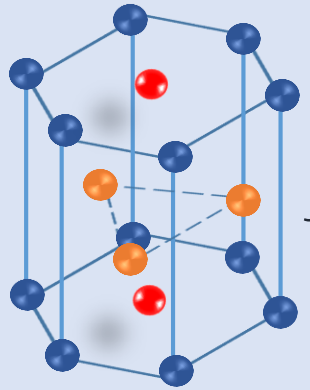


a) Aragonite phase precursor

$$5CaCO_3 + 3(NH_4)_2HPO_4 + H_2O \longrightarrow$$

$$HAp = Ca_5(PO_4)_3OH + 4H_2O + 5CO_2 + 3NH_3$$

Pure hydroxyapatite phase at milling time 60 min



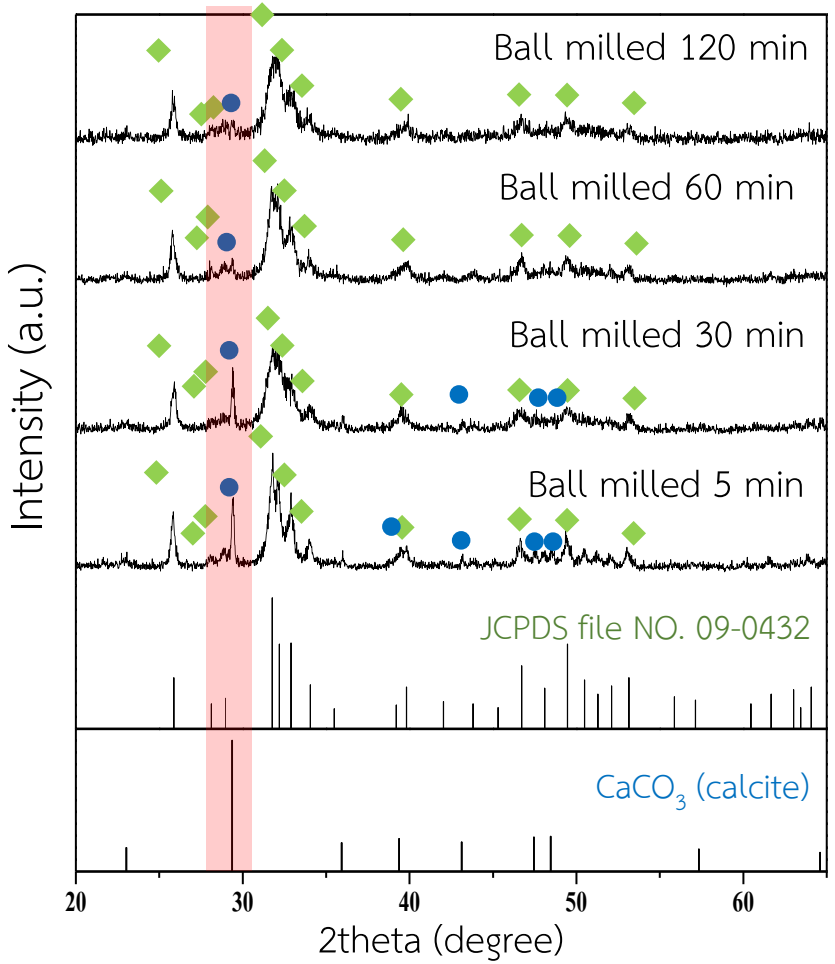
Hexagonal

According to JCPDS file NO. 09-0432 Hydroxyapatite phase

Fig 2 XRD pattern of synthesized hydroxyapatite from cuttlefish bone at **different phase precursor** by ball milling method

Results & Discussion: Part 2 Hydroxyapatite synthesis by ball milling

hydroxyapatite $CaCO_3$ (calcite)

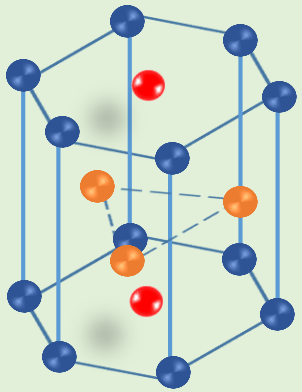


b) Calcite precursor

$$5CaCO_3 + 3(NH_4)_2HPO_4 + H_2O \longrightarrow$$

$$HAp = Ca_5(PO_4)_3OH + 4H_2O + 5CO_2 + 3NH_3$$

Hydroxyapatite and $CaCO_3$ phase appeared at milling time 5 to120 min



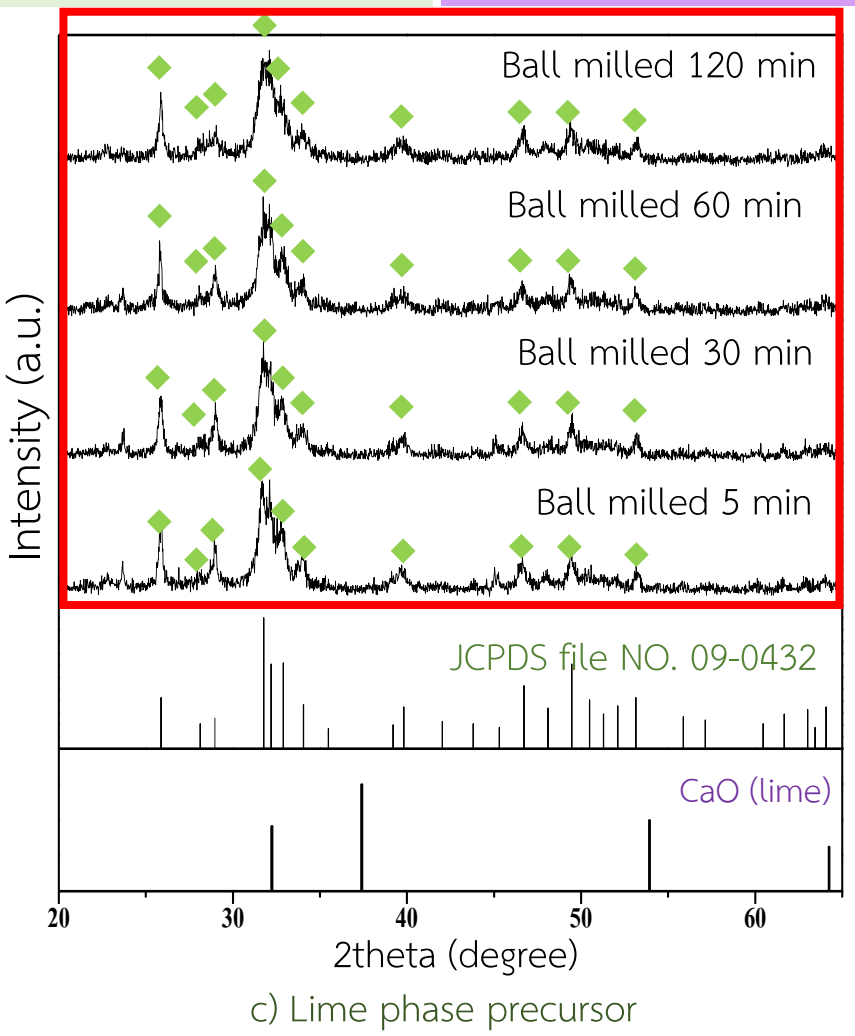
Hexagonal

According to JCPDS file NO. 09-0432 Hydroxyapatite phase

Fig 2 XRD pattern of synthesized hydroxyapatite from cuttlefish bone at **different phase precursor** by ball milling method

Results & Discussion: Part 2 Hydroxyapatite synthesis by ball milling

hydroxyapatite CaO (lime)

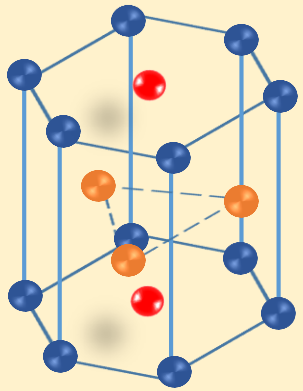


c) Lime phase precursor

$$5CaO + 3(NH_4)_2HPO_4 + H_2O \longrightarrow$$

$$HAp = Ca_5(PO_4)_3(OH) + 5H_2O + 6NH_3$$

Pure hydroxyapatite phase at milling time 5 min



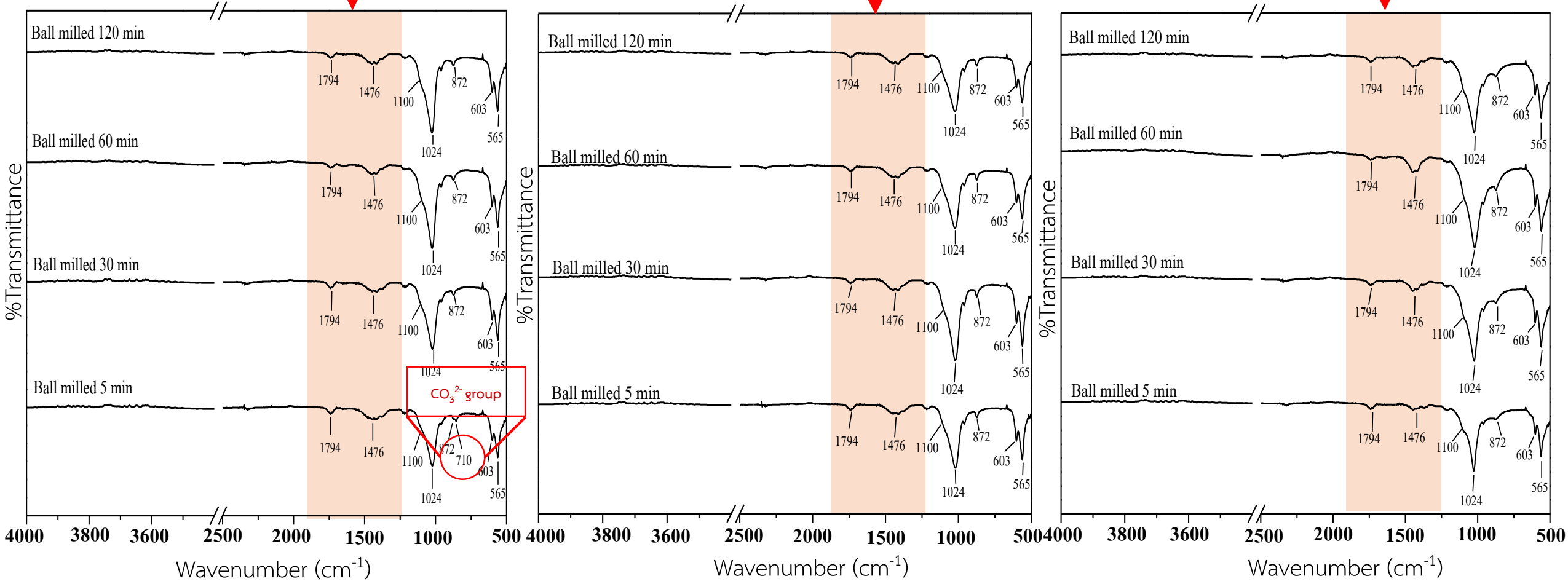
According to JCPDS file NO. 09-0432 Hydroxyapatite phase

Hexagonal

Fig 2 XRD pattern of synthesized hydroxyapatite from cuttlefish bone at **different phase precursor** by ball milling method

Results & Discussion: Part 2 Hydroxyapatite synthesis by ball milling

Synthesis process



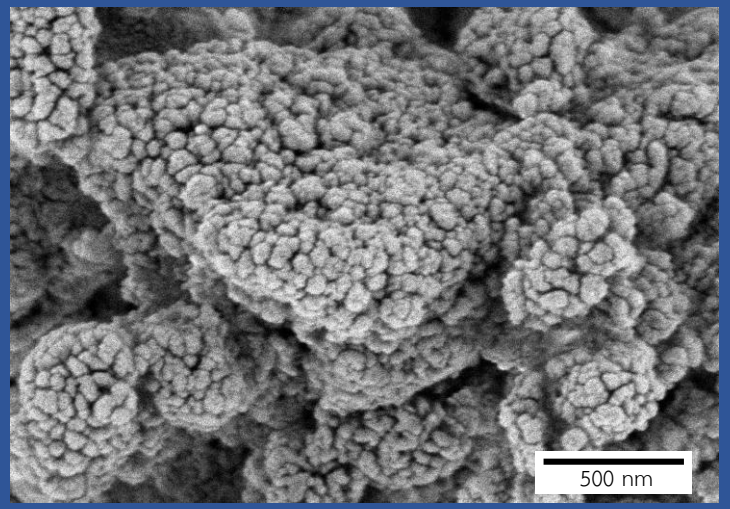
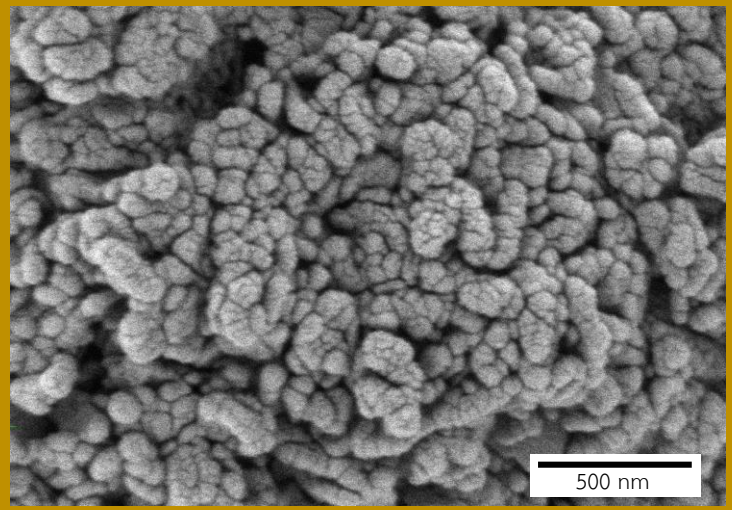
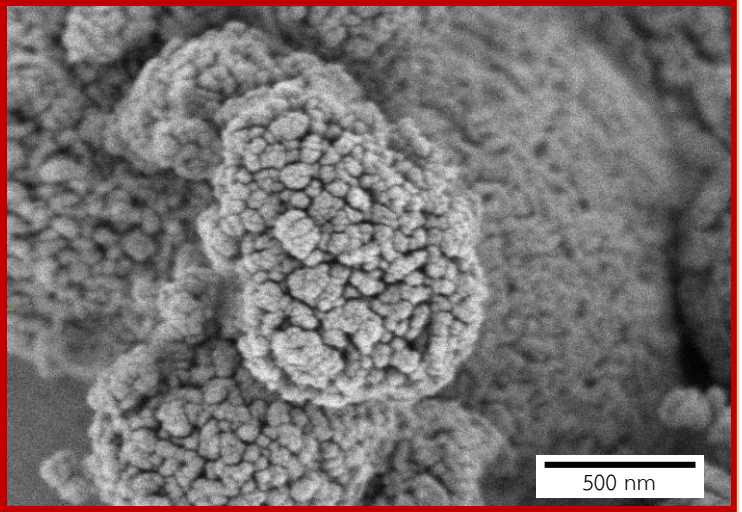
a) Aragonite phase precursor

b) Calcite precursor

c) Lime phase precursor

Fig 3 FTIR spectra of synthesized hydroxyapatite from cuttlefish bone at various different **phase precursor** by ball milling method

Results & Discussion: Part 2 Hydroxyapatite synthesis by ball milling

Ball milling time	a) Aragonite phase precursor	b) Calcite precursor	c) Lime phase precursor
120 min			

Average particle size 60-70 nm

Fig. 4 SEM image of synthesized hydroxyapatite from cuttlefish bone at various different **phase precursor** by ball milling method

Conclusions

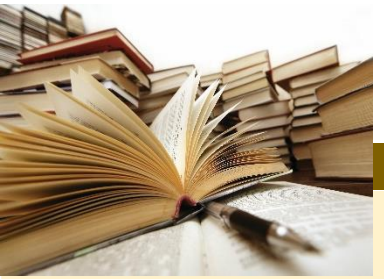
From results of temperature effect on cuttlefish bone

Aragonite phase change to calcite phase completely and calcite phase transform to lime phase completely at temperature 500 °C and 900 °C respectively.

From results of hydroxyapatite synthesis

Hydroxyapatite phase appear at milling 5 minutes

- Hydroxyapatite phase appear completely at 60 minutes in $CaCO_3$ (aragonite phase) precursor.
- Hydroxyapatite phase appear completely at more than 120 minutes in $CaCO_3$ (calcite phase) precursor
- Hydroxyapatite phase appear completely at 5 minutes in CaO (lime) precursor



References

- ✓ Amin, S., Bekhit, A.E., Azam, A. and Zhifa, S., 2015, “Synthesis of Nano- Hydroxyapatite (nHA) from Waste Mussel Shells Using a Rapid Microwave Method”, **Materials Chemistry and Physics**, Vol. 149-150, pp. 607-616.
- ✓ Mehdi, S.S., Khorasani, M.T., Ehsan, D.K. and Ahmad, J., 2013,“Synthesis Methods for Nanosized Hydroxyapatite Indiverse Structures ”, **Acta Biomaterialia**, Vol. 4, pp. 281-312.
- ✓ Liu, J., Li ,K., Wang, H., Zhu, M. and Yan, H., 2014, “Rapid Formation of Hydroxyapatite Nanostructures by Microwave Irradiation”, **Chemical Physical**, Vol. 396, pp. 429-432..

Thank you for your attention