

ภาคผนวก ก คุณสมบัติการละลายน้ำของฟิล์ม

# ค่าเปอร์เซนต์คุณสมบัติการละลายน้ำของฟิล์ม

		al	น้ำหนักก่อนแช่	น้ำหนักหลังแช่	เปอร์เซ็นต์การ
สูตร	สัคส่วน	ครั้งที่			رو
			สารละลาย (g)	สารละลาย (g)	ละลายนำ (%)
		1	0.0062	0.0055	11.2903
PLA	10:0	2	0.0070	0.0062	11.4286
		3	0.0070	0.0064	8.5714
		ก่าเฉลี่ย	0.0067	0.0060	10.3960
		1	0.0146	0.0132	9.5890
PLA:ES	9:1	2	0.0142	0.0128	9.8592
		3	0.0140	0.0127	9.2857
ก่าเฉลี่ย			0.0143	0.0129	9.5794
		1	0.0117	0.0108	7.6923
PLA:ES	8:2	2	0.0119	0.0111	6.7227
		3	0.0120	0.0110	8.3333
		ก่าเฉลี่ย	0.0119	0.0110	7,5843
		1	0.0124	0.0117	5.6452
PLA:ES	7:3	2	0.0127	0.0119	6.2992
		3	0.0131	0.0123	6.1069
		ก่าเฉลี่ย	0.0127	0.0120	6.0209

ภาคผนวก ข

Reference code JCPDs

Name and formula

Reference code:

01-085-1108

ICSD name:

Calcium Carbonate

Empirical formula: CCaO3

Chemical formula: CaCO3

Crystallographic parameters

Crystal system:

Rhombohedral

Space group:

R-3c

Space group number:167

a (?):

4.9803

b (?):

4.9803

c (?):

17.0187

Beta (?): 90.0000

Alpha (?): 90.0000

Gamma (?):

120.0000

Calculated density (g/cm<sup>3</sup>):

2.73

Volume of cell (10<sup>6</sup> pm<sup>3</sup>): 365.57

Z:

6.00

RIR:

3.39

Subfiles and Quality

Subfiles: Inorganic

Pharmaceutical

ICSD Pattern

Quality: Calculated (C)

Comments

Additional pattern: See PDF 72-1214, 72-1652, 81-2027, 83-577, 83-578, 83-1762, 86-174

and 86-2334.

ICSD collection code:

037241

ICSD Space group comment: Origin translated by (1/4,1/4,1/4) prior to calculation.

Test from ICSD:

No R value given.

## At least one TF missing.

### References

Primary reference: Calculated from ICSD using POWD-12++, (1997)

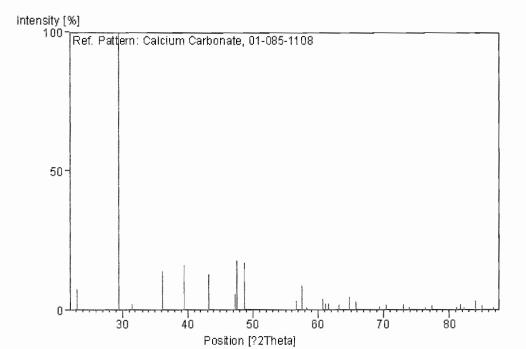
Structure: Wyckoff, R.W.G., Am. J. Sci., 50, 317, (1920)

## Peak list

No.	h	k	1	d [A]	2Theta[deg	g] I [%]
1	0	1	2	3.84712	23.101	7.5
2	1	0	4	3.02895	29.466	100.0
3	0	0	6	2.83645	31.516	2.0
4	1	1	0	2.49016	36.039	14.2
5	1	1	3	2.28015	39.489	16.1
6	2	0	2	2.09045	43.244	13.2
7	0	2	4	1.92356	47.213	5.7
8	0	1	8	1.90789	47.625	17.8
9	1	1	6	1.87133	48.615	17.1
10	2	1	1	1.62276	56.678	3.4
11	1	2	2	1.60107	57.517	8.8
12	1	0	10	1.58309	58.232	0.8
13	2	1	4	1.52228	60.798	4.2
14	2	0	8	1.51447	61.145	2.2
15	1	1	9	1.50597	61.527	2.3
16	1	2	5	1.47026	63.191	2.0
17	3	0	0	1.43769	64.795	4.8
18	0	0	12	1.41822	65.796	3.1
19	2	1	7	1.35399	69.349	1.3
20	0	2	10	1.33597	70.421	1.9
21	1	2	8	1.29396	73.069	2.0
22	0	3	6	1.28237	73.838	0.6
23	2	2	0	1.24508	76.439	0.9
24	1	1	12	1.23237	77.373	1.7

25	3	1	2	1.18459	81.123	0.5
26	2	1	10	1.17724	81.737	2.1
27	0	1	14	1.17004	82.349	0.2
28	1	3	4	1.15158	83.966	3.4
29	2	2	6	1.14008	85.010	1.8
30	1	2	11	1.12221	86.694	0.5

## Stick Pattern



Name and formula

Reference code:

01-086-2335

Mineral name:

Calcite magnesian

ICSD name:

Magnesium Calcium Carbonate

Empirical formula: CCa0.936Mg0.064O3

Chemical formula: (Mg.064Ca.936) (CO3)

Crystallographic parameters

Crystal system:

Rhombohedral

Space group:

R-3c

Space group number:167

a (?):

4.9673

b (?):

4.9673

c (?):

16.9631

Alpha (?): 90.0000

Beta (?): 90.0000

Gamma (?):

120.0000

Calculated density (g/cm<sup>3</sup>): 2.72

Measured density (g/cm<sup>3</sup>):

2.72

Volume of cell (10<sup>6</sup> pm<sup>3</sup>):

362.47

Z:

6.00

RIR:

3.05

Subfiles and Quality

Subfiles: Inorganic

Mineral

Corrosion

ICSD Pattern

Quality: Calculated (C)

Comments

Sample source:

Specimen from the spine of an echinoid.

ICSD collection code:

040108

Test from ICSD: Calc. density unusual but tolerable.

References

Primary reference: Calculated from ICSD using POWD-12++, (1997)

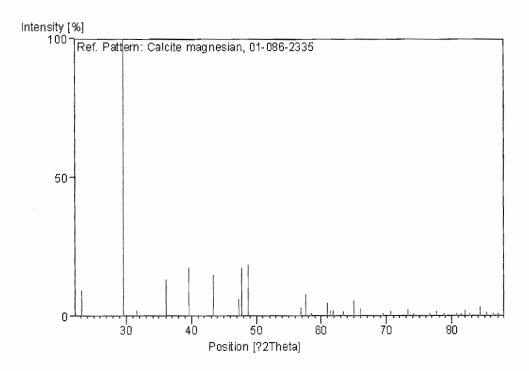
Structure: Paquette, J., Reeder, R.J., Am. Mineral., 75, 1151, (1990)

#### Peak list

No.	h	k	1	d[A]	2Theta[deg	g] I [%]
1	0	1	2	3.83655	23.165	9.4
2	1	0	4	3.02003	29.555	100.0
3	0	0	6	2.82718	31.622	2.0
4	1	1	0	2.48365	36.136	13.5
5	1	1	3	2.27395	39.601	17.6
6	2	0	2	2.08491	43.365	14.7
7	0	2	4	1.91827	47.351	6.1
8	0	1	8	1.90190	47.784	17.7
9	1	1	6	1.86591	48.765	18.6
10	2	1	1	1.61851	56.840	3.0
11	1	2	2	1.59685	57.683	8.0
12	1	0	10	1.57805	58.436	1.0
13	2	1	4	1.51817	60.980	4.9
14	2	0	8	1.51001	61.345	2.1
15	1	1	9	1.50141	61.735	2.1
16	1	2	5	1.46624	63.385	1.7
17	3	0	0	1.43394	64.985	5.3
18	0	0	12	1.41359	66.039	2.8
19	2	1	7	1.35018	69.573	0.9
20	0	2	10	1.33194	70.666	1.7
21	1	2	8	1.29026	73.312	2.5
22	3	0	6	1.27885	74.075	0.6
23	2	2	0	1.24182	76.676	1.1
24	1	1	12	1.22854	77.659	1.7

25	2	2	3	1.21292	78.852	0.1
26	1	3	1	1.19017	80.664	0.1
27	3	1	2	1.18147	81.383	0.4
28	2	1	10	1.17380	82.028	1.9
29	0	1	14	1.16627	82.673	0.3
30	1	3	4	1.14852	84.241	3.5
31	2	2	6	1.13698	85.297	1.4
32	3	1	5	1.12553	86.375	0.1
33	1	2	11	1.11889	87.015	0.3

## Stick Pattern



ภาคผนวก ค ผลงานวิจัยที่ได้รับการตีพิมพ์เผยแพร่

# PACCON 2012-CHEMISTRY BEYOND BOUNDARIES

11-13 JANUARY 2012, THE EMPRESS CONVENTION CENTER CHIANG MAI, THAILAND

e-ABSTRACT



& THE CHEMICAL SOCIETY OF THAILAND UNDER THE PATRONAGE OF
HER ROYAL HIGHNESS PRINCESS CHULABHORN MAHIDOL

### BIP-P-017

## TRACE METAL AND MORPHOLOGY OF THE EGGSHELL: PRELIMINARY SURVEY IN CHICKEN EGGSHELL

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#### **ABSTRACT**

The present study aims to investigate physical properties and chemical properties of eggshell from local market and the Bakeries factory. The samples were evaluated for the potentiality to use as filler in biodegradable polymers production. Energy Dispersive X-Ray Fluorescence (EDXRF) spectrometer was used to determine the composition of mineral and levels of the constituent element. The results show that metals Titanium (Ti), copper (Cu), iron (Fe) and Potassium (K), have highest accumulated in the eggshell from the Bakeries factory. The crystal structure of the shells was studied by x-ray diffraction (XRD). The XRD patterns spectra reveal that the eggshells from local market made of a calcite but the eggshells from the Bakeries factory have the mixture phase of calcite and calcite magnesium phase, respectively. In this study, we also used the scanning electron microscope (SEM) to study the Morphology of the eggshells. The results were shown that the eggshells from local market have the porous scattered all over samples more than the eggshells from the Bakeries factory.

Keywords EDXRF; XRD; SEM; Chicken eggshell

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THE 6<sup>th</sup> PURE AND APPLIED CHEMISTRY INTERNATIONAL CONFERENCE 2012

## PACCON 2012-CHEMISTRY BEYOND BOUNDARIES

11-13 JANUARY 2012, THE EMPRESS CONVENTION CENTER CHIANG MAI, THAILAND

## **PROCEEDINGS**



DEPARTMENT OF CHEMISTRY, FACULTY OF SCIENCE, CHIANG MAI UNIVERSITY

& THE CHEMICAL SOCIETY OF THAILAND UNDER THE PATRONAGE OF

HER ROYAL HIGHNESS PRINCESS CHULABHORN MAHIDOL

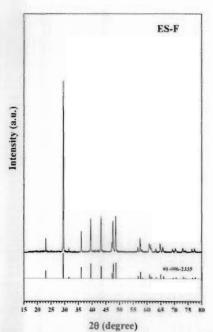


Figure 3. Powder XRD patterns of ES-F sample.

The morphologies of the eggshells are shown in the SEM micrographs (Fig. 4(a) and (b)). It is seen that the ES-M samples have the porous scattered all over samples more than the ES-F samples.

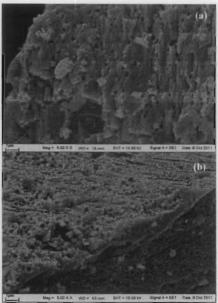
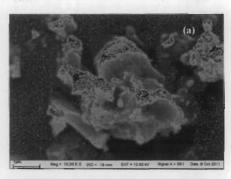


Figure 4. The figure and microstructure of eggshell samples (a) ES-F (b) ES-M.

The texture structure examination of eggshell can be observed from Fig. 5. It can be clearly seen that these two samples have different pattern. From Fig. 5(a), it appeared as rod like fracture aggregate and apparently disoriented crystals, the pattern of ES-M samples like stack of lamella foliated and disoriented foliated grains can be observed in Figure 5 (b).



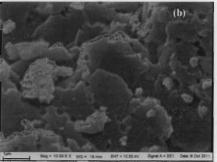


Figure 5. The figure and microstructure of eggshell samples (a) ES-F (b) ES-M

#### 4. Conclusions

The eggshell samples consist mainly of CaCO3, calcite phase. The contaminant on the eggshell sample has induced the mixture phase of calcite magnesian phase. These steps are crucial for the quality of the initial product, since the content of trace element should be as low quality as possible. Then the structural and morphological of eggshell is a key role in the formation to fabrication of biodegradable polymers.

#### Acknowledgements

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#### References

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- [5] P. Toro, R. Quijadaa, M. Yazdani-Pedramb, J. Luis Arias, Mater. Lett.. 61 (2007) 4347-4350
- [6] T. Kato, Adv. Mater.. 12 (2000) 1543-1546.

# TRACE METAL AND MORPHOLOGY OF THE EGG SHELL: PRELIMINARY SURVEY IN CHICKEN EGG SHELL

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Abstract: The present study aims to investigate physical properties and chemical properties of eggshell from local market and the Bakeries factory. The samples were evaluated for the potentiality to use as filler in biodegradable polymers production. Energy Dispersive X-Ray Fluorescence (EDXRF) spectrometer was used to determine the composition of mineral and levels of the constituent element. The result show that metals Titanium (Ti), copper (Cu), iron (Fe) and Potassium (K), have highest accumulated in the eggshell from the Bakeries factory. The XRD patterns spectra reveal that the eggshells from local market made of a calcite but the eggshells from the Bakeries factory have the mixture phase of calcite and calcite magnesian phase, respectively. In this study, we also used the scanning electron microscope (SEM) to study the Morphology of the eggshells. The results shown that the eggshells from local market have the porous scattered all over samples more than the eggshells from the Bakeries factory.

#### 1. Introduction

Several fields, including material Science give priority to the natural composite materials. Especially, the biodegradable polymers are widely attention for conventional utilization such as packaging material because of increasing environmental concerns [1,2]. According to Kasuga [3], the combination of poly (lactic acid) (PLA) and calcium carbonate (CaCO<sub>3</sub>) have been developed a composite as biodegradable polymer.

Calcium carbonate is one of large amount abundant minerals in nature. There are numerous natural calcium carbonate sources, such as mollusc shell and egg shell. To our information in Thailand, the waste shells of egg shell and mollusk shell from agricultural and food industries were produced several tons for each day.

Eggshells waste contains many precious organic and inorganic components, which can be developing into the biomaterials from this waste [4]. The eggshells consist of calcite and calcium carbonate crystals, while the eggshell membrane consisting of organic matters, such as collagen, polypeptides and amino acids [5]. Eggshell was often specific of morphology, has been the most utilization material, due to some extent of eggshell is availability and low cost. Calcium carbonate has three types of polymorphs: calcite, aragonite, and vaterite. Calcite is thermodynamically

the most stable form, while vaterite is the most unstable form [6].

This studied, is a preliminary survey, which have the purpose is to investigate the physical and chemical properties of the eggshells, also the structural and morphological of eggshells have been used to information reference of descriptions and the ability of eggshells as alternative filler for biodegradable polymers.

#### 2. Materials and Methods

Eggshells sample was obtained from the local market in Chonburi province and Bakeries factory in Nakhon Pathom province. First of all the eggshells samples were remove impurity and interference material. After that, the eggshells sample was washed with distilled water several time and dry in an oven for 24 h. Afterward, the cleaned sample was ground into fine powder by sieved, which using a stainless laboratory test sieve with an space size of 75 µm. The powder samples were dry again and packed in phial and denoted as ES-M and ES-F for the local market and bakeries factory, respectively.

X-ray fluorescence was used for the qualitative and quantitative analyses of in organic composition in the eggshells samples by Energy Dispersive X-rays spectrometer (EDXRF) Panalytical minipal-4. The mineralogy and crystal structure was characterized by a powder X-ray diffractometer. Powder diffraction data were recorded at room temperature using a Bruker AXS D8 Advance powder diffractomter [CuK $_{\alpha}$  (Ni filtered) with scintillation detector;  $2\theta$  range,  $20\text{-}80^{\circ}$  step size  $0.02^{\circ}$ ]. The minerals in each case were indentified from the diffractograms by reference to Joint Committee on Powder Diffraction Standard File (JCPDS).

Another that, surface morphological of all eggshell samples were studied by scanning electron microscopy. The Scanning Electron Microscopy (SEM) were performed on LEO model LEO SEM 1550 with gold sputtered fractured specimens.

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#### 3. Results and Discussion

The X-Ray Fluorescence spectrometer technique is used to analysis of constituents of ES-M and ES-F sample. The chemical composition by XRF reported that all eggshells sample had different chemical contents, concerning the station variation. Both eggshells sample have the CaCO<sub>3</sub> as mainly elements and a few of other elements as shown in Table 1. The highest concentrations for metal (K, Fe, Ti) were recorded at ES-F sample.

Table 1: XRF analysis of constituents of eggshells sample (Wt%)

Station/Element	Local market	Bakeries factory	
K	Nd	0.483	
Ca	99.303	97.49	
Ti	Nd	0.010	
Fe	0.061	0.170	
Cu	0.078	0.076	
Sr	0.120	0.130	

The minor compositions of element in ES-F may be attributing to the contamination in production process of Bakeries factory.

The XRD patterns of the eggshell samples are present in Figure 1, it suggesting that the phase of eggshell sample have variation. The crystal structure of ES-M samples was made of a calcite phase, a common form of CaCO<sub>3</sub> mineral. For ES-F samples has a mixture phase of calcite and calcite magnesian phase.

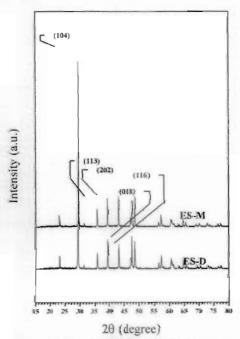


Figure 1. Powder XRD patterns of ES-M and ES-F sample.

The mineral phases of the eggshell samples were identified from the diffractograms by reference to the Joint Committee on Powder Diffraction Standard (JCPDS) numbers 00-085-1108, and 01-086-2335 correspond to calcite phase and calcite magnesian phase respectively

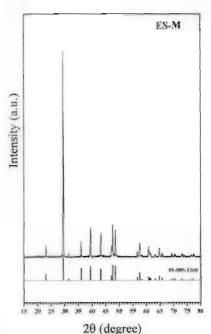


Figure 2. Powder XRD patterns of ES-M sample.

As shown in Figure 1, The diffraction peaks of the ES-M and ES-F sample can be indexed as the (104) reflection of pure calcite at  $29.555^{\circ}$  -  $2\theta$  value. All peaks in the ES-D samples were well matched with the JCPDS numbers 00-085-1108, as shown in Fig 2. From Fig. 3, The ES-F samples were matched with JCPDS numbers 00-086-2335, the diffraction peaks can be indexed as the (202), (018) and (116) reflection which have different pattern as shown in Fig 1. It indicate that the ES-F samples have the mixture phase between the calcite phase and calcite magnesian phase, then the mineral variation from ES-M samples.