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# CHARACTERIZATION OF DICALCIUM PHOSPHATE FROM WASTE CLAM SHELL FOR BONE CEMENT AND TOOTHPASTE FORMULATIONS

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## Abstract

This research was focused on the preparation of dicalcium phosphate from waste clam shells (*Mercenaria mercenaria*) and utilization of prepared dicalcium phosphate for bone cement and toothpaste formulations. Waste clam shells were collected from seafood restaurant Chanayethazan Township, Mandalay Region. The clam shell powder (calcium carbonate) was prepared from waste clam shells by washing, grinding, sieving and drying. For the assessment of the quality of clam shell powder, physicochemical properties of clam shell powder (color, odor, solubility, density, moisture and calcium carbonate content) were investigated. The clam shell powder was decomposed to calcium oxide by calcination at 1000°C for 4 hr. Dicalcium phosphate (monetite) was prepared with 1 M phosphoric acid at 75˚C for 30 min with the stirring speed 500 rpm and 24 hr aging time. The phase, functional group, elemental compositions and morphological nature were determined to identify the purity of clam shell powder and prepared dicalcium phosphate. The prepared dicalcium phosphate was used in preparation of monetite bone cement and toothpaste formulation. A low compressive strength but very simple and inexpensive orthopedic monetite bone cement had 5 min initial setting time, 15 min final setting time and 2 to 3 MPa compressive strength. In the utilization of prepared dicalcium phosphate in toothpaste, the most suitable amount of processed dicalcium phosphate as abrasive for the preparation of toothpaste was 41%.

**Keywords**: Calcium phosphate, clam shell, calcium carbonate, bone cement, toothpaste, phosphoric acid

## Introduction

Recently, interest in the recycling of waste materials is increasing. From this view point, clam shells have been used as a raw material for various applications. Generally, edible parts of clam shells are few and thus large amounts of clam shells as waste material are produced from processing of seafood (Onoda, 2012). The hard calm (*Mercenaria mercenaria*) also known

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as the quahog, northern quahog, littleneck calm, round clam, is an edible marine bivalve mollusk in the family Veneridae. The clam shell has several layers, and is typically made of calcium carbonate that is formed by the deposition of crystals of this salt in an organic matrix of the protein, conchiolin. Among the three layers of the shell, a middle prismatic layer of aragonite or calcite, a crystalline form of calcium carbonate is found (Goslin, 2004). It is secreted by a part of the molluscan body known as the mantle. The source of this kind of calcium carbonate is completely organic with 96% of $CaCO_3$, 5% biopolymers and small fraction of water. Thus, the finding suggests the possibility of using the clam shell as alternative biomaterials for production of CaO (Hoque, 2013).

Calcium phosphates have been widely investigated as implants for substituting human bone in the field of biomaterials for orthopedics. As a bioactive material, monetite (dicalcium phosphate anhydrous) is one of the stable phases of the calcium phosphates, which have attracted considerable attention (Baradaran, S., 2012). Monetite is one of the mildly acidic and soluble calcium phosphate phases and currently finding a significant place to itself in the powder components of self-hardening calcium phosphate pastes used for skeletal repair. Dicalcium phosphate is also used in powder form in some toothpaste, chewing gums and in food processing industry to act as acidity regulator, anti-caking agent, dough modifier, and emulsifier (Cuneyt, 2013).

The objective of this research was to study the feasibility of producing dicalcium phosphate from waste clam shells, and to assess the effectiveness of utilization of prepared calcium salt on the characteristics of their products.

## Materials and Methods

### Materials

The waste clam shells were collected from Shwe Gannan Seafood Restaurant, Chanayethazan Township, Mandalay Region and phosphoric acid (analar grade) was purchased from Golden Lady Chemical Store, Pabedan Township, Yangon Region.

## Methods

### Preparation and Characterization of Clam Shell Powder

The clam shells were washed thoroughly with water and then dried in air for 48 hr. After drying, they were ground and sieved using a mesh size of 200 mesh sieve. The physico-chemical characteristics of clam shell powder such as moisture content, solubility, density and calcium carbonate content were determined. The phase composition, functional groups, elemental compositions and morphological nature of clam shell powder were also investigated.

### Determination of Calcium Carbonate Content

5 g of clam shell powder was placed in a conical flask and 50 mL of 0.25M HCl was added using a pipette. The mixture was heated and stirred until all the entire sample was dissolved (no more bubbles of $CO_2$ being evolved). The unreacted acid in the flask was titrated with 0.1M NaOH.

$$\text{Number of mole of } CaCO_3 \text{ in clam shell} = \frac{(\text{used HCl mL} \times \text{N of HCl}) - (\text{used NaOH mL} \times \text{N of NaOH})}{1000 \times 2}$$

$$\%\ \text{w/w } CaCO_3 \text{ in clam shell} = \frac{(\text{no. of mole of } CaCO_3 \times \text{molecular wt. of}}{\text{weight of clam shell powder}} \times 100$$

### Processing of Dicalcium Phosphate

The clam shell powder (≈ 97 % $CaCO_3$) was calcined in a muffle furnace at 1000˚C for 4 hr to decompose the calcium carbonate and to remove organic components. Calcium oxide 5.6 (calcined clam shell powder) was finely ground in a grinder. 100 mL of 1 M phosphoric acid solution was added to the calcium oxide, heated at 75˚C, stirring with 500 rpm for 30 min. Then, the suspension was aged for 24 hr at room temperature. After aging, the mixture was filtered and the precipitate was dried at 100˚C in an oven.

### Effect of Strength of Phosphoric Acid on the Formation of Calcium Phosphate

The effect of strength of phosphoric acid was determined using 100 mL of phosphoric acid with various concentrations (0.3, 0.4, 0.5, 0.6, 0.7, 0.8, and 1M).

### Effect of Reaction Temperature on the Formation of Calcium Phosphate

The effect of reaction temperature was determined using 100 mL of 1 M phosphoric acid at various reaction temperatures (15, 45, 60, 75, and 90˚C).

### Characterization of Dicalcium Phosphate

The physico-chemical characteristics of prepared dicalcium phosphate such as moisture content, solubility, density and calcium content were determined. The phase composition, functional groups, elemental compositions and morphological nature of dicalcium phosphate were also investigated.

### Application of Prepared Dicalcium Phosphate in Monetite Bone Cement

Prepared dicalcium phosphate was utilized in the preparation of monetite bone cement. Properties of monetite bone cement such as setting time, compressive strength, expansion and bending strength were determined to identify the quality of the bone cement.

### Application of Prepared Dicalcium Phosphate in Toothpaste Formulation

Prepared dicalcium phosphate was also used in toothpaste formulation. Characteristics such as organoleptic properties, pH, foaming power, viscosity, alkalinity, density, moisture content, and stability of formulated toothpaste were investigated.

## Results and Discussion

In this research work, the physico-chemical properties of clam shell powder such as moisture content, solubility, bulk density and calcium content were determined and the results are shown in Table (1).

The analysis of mineral phase using XRD illustrated that raw clam shell was made of aragonite, $CaCO_3$. The XRD pattern of the clam shell powder is shown in Fig. (1). The surface morphology of clam shell powder was examined by SEM and the images are shown in Fig. (2). Calcite and aragonite possessed different crystal growth patterns and crystal structure.. Cube like crystals of calcite were stable as compared to rod like orthorhombic crystals of aragonite.

The FT-IR spectra of clam shell powder and analar grade calcium carbonate are presented in Fig. (3). It was found that the prominent peaks observed at 1788 $cm^{-1}$, 1479 $cm^{-1}$, 1082 $cm^{-1}$, 860 $cm^{-1}$, and 711 $cm^{-1}$ were the common characteristic features of the $CO_3^{2-}$ ions in $CaCO_3$, which were assigned to the fundamental modes of vibration of this molecule. The bands at 3454 $cm^{-1}$ were due to the presence of water content in the material of the clam shell. From the results, it can be seen that both the clam shell powder and analar grade calcium carbonate possess the same vibration frequencies as observed in the FTIR data.

The chemical analysis using EDXRF had been conducted to estimate the mineral composition in clam shell and shown in Table (2). The results indicated that calcium content in clam shell powder was over 96 % and analar grade calcium carbonate was 99 %. The clam shell powder contained negligible traces of elemental potassium, iron, manganese and strontium. Therefore the clam shells are the rich source of calcium carbonate.

## Effect of Concentration of $H_3PO_4$ on the Formation of Calcium Phosphate

In this work, the effects of acid concentration (0.3 to 1M) on the yield percentage and phase composition were studied and the results are shown in Tables (3) to (5).

From the XRD data, the combined phases of calcium phosphate were observed at the low concentrations of acid, 0.3 M to 0.8 M. The pure monetite (dicalcium phosphate) phase was observed with the phosphoric acid concentrations 0.9 M and 1 M.

Moreover, from the FT-IR data, the presence of dicalcium phosphate was more evident when 1M phosphoric acid was used due to the observed vibrations of $PO_4$ tetrahedral ($\upsilon_1$, 995$cm^{-1}$, $\upsilon_2$, 401$cm^{-1}$, $\upsilon_3$, 1066, 1132 $cm^{-1}$, $\upsilon_4$, 582, 563 $cm^{-1}$). With regards to the presence of carbonates, the spectra indicated by the vibrational frequencies for $CO_3^{2-}$ at 891$cm^{-1}$ for $\upsilon_2$, 1408 $cm^{-1}$ for $\upsilon_3$. The bands observed at 3454 $cm^{-1}$ was due to the presence of water in the material of the clam shell. The morphological nature of all calcium phosphate powders were of crystalline appearance.

## Effect of Reaction Temperature on the Formation of Calcium Phosphate

The effects of reaction temperature, on the phase composition are shown in Tables (6) to (8). The XRD results indicated that at low reaction temperature 30˚C, the phase was brushite $CaHPO_4 (H_2O)_2$. At 45˚C and 60˚C, the phase was a mixture of brushite $CaHPO_4 (H_2O)_2$, and monetite $CaHPO_4$. Only monetite $CaHPO_4$ phase was observed at 75˚C and 90˚C. Thus the most suitable reaction temperature was 75˚C for the preparation of dicalcium phosphate.

From the FT-IR data, the absorption band at 3450 $cm^{-1}$ assigned to the OH stretch vibration of hydrogen bonded OH groups. The peak at 582, 563 $cm^{-1}$ attributed to $\upsilon_4$, $PO_4$, 995 $cm^{-1}$ for $\upsilon_1$, $PO_4$, and the peaks at 1066, 1132 $cm^{-1}$ for $\upsilon_3$, $PO_4$. The bands at 1408 and 891 $cm^{-1}$ confirm the presence of carbonate group, respectively $\upsilon_3$, $CO_3$ and $\upsilon_4$, $CO_3$. The band at 890 $cm^{-1}$ could be attributed to $HPO_4$ groups which clearly showed monetite phase.

The SEM micrographs of the calcium phosphate powder show strongly agglomerated size. The higher the temperature, the most rounded crystals appeared. The powder prepared at 75˚C possesses circular crystals with large size.

## Preparation of Cement with Dicalcium Phosphate

The effects of amount of setting solution on the setting time are shown in Table (10). At a lower amount of setting solution containing only water of 50 to 70 mL, led to incomplete mixing and giving an inhomogeneous material with no self-setting properties. On the other hand, the higher amount of setting solution of 110 mL, led to monetite particles being suspended in an aqueous solution and hence, no cement like behavior was observed. The cement formed by using 90 mL setting solution gave cement like behavior to the pastes with self-setting properties. The compressive strength of cement were investigated and shown in Tables (10) and (11). Organic acids such as citric acid are known to be present in trace amounts in bones. Addition of a small amount of citric acid to setting solution led to significant improvement in handling of the cement. The monetite cement with 1 % citric acid into monetite improved the compressive strength of the bone cement. The cement sample did not retain their integrity and crumbled, hence the cement sample had no expansion and bending strength. Such a property of bone cement was similar to the human bones. The compressive strength of the cement was found to be 2.81 MPa. It should be noted that the human trabecular bones has a modest compressive strength over the range of 2 to 10 MPa. Clam shell is a source of biocalcium and biocalcium contains calcium to aid in the formation and maintenance to bones. So, monetite bone cement and biocalcium toothpaste were prepared from processed dicalcium phosphate.

**Effectiveness of Prepared Dicalcium Phosphate in Toothpaste Formulation**

The effect of amount of dicalcium phosphate (abrasive) on the pH of formulated toothpaste was determined and the results are shown in Tables (12), and (13). It was observed that the 30 g of abrasive (DCP) was the suitable amount because the pH of the formulated toothpaste was 7.0. The pH of the toothpaste with insoluble materials and low abrasiveness is generally neutral or basic (Oyewale, 2004). The physico-chemical properties of prepared toothpaste are indicated in Table (13). It was found that the viscosity of formulated toothpaste was lower than the viscosity of commercial toothpaste because of the larger particle size of prepared dicalcium phosphate that might interfere the viscosity of toothpaste. The properties of formulated toothpaste were found to be within the respective limits of toothpaste specifications. (Oyewale, 2004). So, the prepared dicalcium phosphate was suitable as abrasive in toothpaste formulation.

**Table 1: Physico-chemical Properties of Clam Shell Powder**

| Sr. No | Properties | *Clam Shell Powder | $CaCO_3$ (Analar grade) |
|---|---|---|---|
| 1 | Colour | Gray | white |
| 2 | Odour | Odourless | odourless |
| 3 | Moisture content (%w/w) | 0.5 | 1.0 |
| 4 | Solubility in water | 0.005 | 0.001 |
| 5 | Density ($g/cm^3$) | 2.79 | 2.83 |
| 6 | Calcium (%w/w) | 35.9 | 37.67 |

*Clam shell powder contained ≈ 97% $CaCO_3$

**Table 2: Elemental Composition of Clam Shell Powder**

| Sr. No | Elements | Clam Shell Powder (% w/w) | $CaCO_3$ (Analar grade, BDH) (% w/w) |
|---|---|---|---|
| 1 | Calcium (Ca) | 96.195 | 99.710 |
| 2 | Iron (Fe) | 1.304 | - |
| 3 | Potassium (K) | 1.144 | - |
| 4 | Manganese (Mn) | 0.707 | - |
| 5 | Strontium (Sr) | 0.650 | 0.290 |

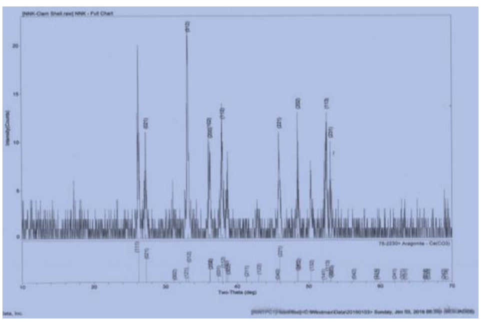

**Fig1:** XRD Pattern of Clam Shell Powder

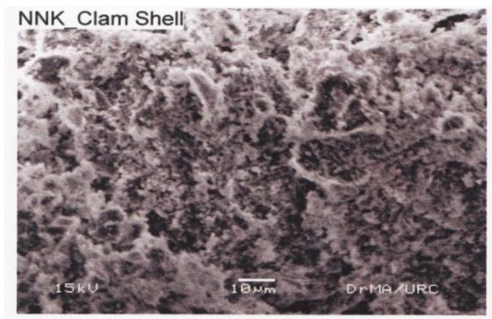


**Fig2:** SEM Micrograph of Clam Shell Powder

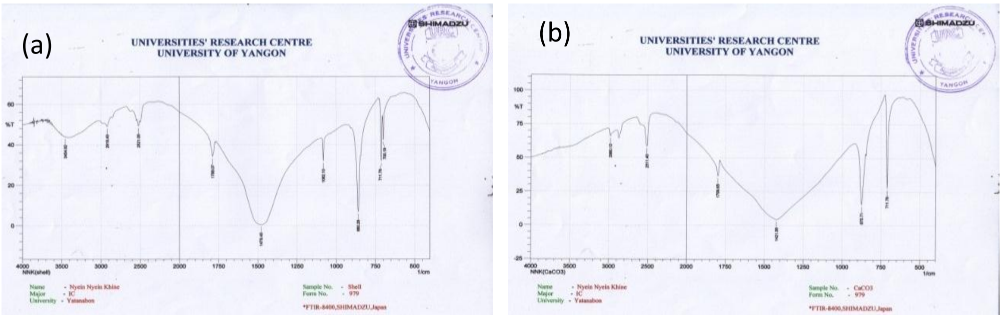


**Fig 3**: FTIR Spectrum of (a) Clam Shell Powder
(b) Calcium Carbonate (Analar grade)

**Table 3: Effect of the Concentration of Phosphoric Acid on the Yield Percent and Phase Formation of Calcium Phosphate**

Clam shell powder = 10 g (5.6 g of CaO)
Volume of $H_3PO_4$ = 100 mL
Reaction temperature = 75˚C
Reaction time = 30 min
Stirring speed = 500 rpm
Aging time = 24 hr

| Sr. No | Strength of $H_3PO_4$ (M) | Yield (% w/w) | Phase |
|---|---|---|---|
| 1 | 0.3 | 67.69 | $Ca_{10}(PO_4)_6O$, $Ca(OH)_2$, $Ca_3(PO_4)_2$ |
| 2 | 0.4 | 67.19 | $Ca_{10}(PO_4)_6O$, $Ca(OH)_2$, $Ca_3(PO_4)_2$ |
| 3 | 0.5 | 67.32 | $Ca_{10}(PO_4)_6O$, $Ca(OH)_2$, $Ca_3(PO_4)_2$ |
| 4 | 0.6 | 66.37 | $Ca(OH)_2$, $CaHPO_4$ |
| 5 | 0.7 | 66.02 | $Ca(OH)_2$, $CaHPO_4$ |
| 6 | 0.8 | 65.67 | $Ca(OH)_2$, $CaHPO_4$ |
| 7 | 0.9 | 64.17 | $CaHPO_4$ |
| 8 | 1* | 63.55 | $CaHPO_4$ |

***** most suitable acid concentration

**Table 4: Elemental Composition of Calcium Phosphate Prepared with Different Concentrations of Phosphoric Acid**

| Sr. No | Elements | Calcium Phosphate Prepared with Different Concentrations of $H_3PO_4$ (M) | | | | | | | | $CaHPO_4$ (Analar grade, BDH) | * Literature |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 0.3 | 0.4 | 0.5 | 0.6 | 0.7 | 0.8 | 0.9 | 1 | | |
| 1 | Calcium (Ca) (% w/w) | 88.864 | 84.839 | 82.427 | 77.566 | 77.971 | 73.202 | 73.279 | 77.683 | 78.851 | 77.23 |
| 2 | Phosphorus(P) (% w/w) | 10.097 | 14.316 | 16.012 | 21.570 | 20.352 | 25.722 | 25.723 | 19.860 | 20.312. | 22.77 |
| 3 | Potassium (K) (% w/w) | - | - | 0.650 | - | 0.690 | - | - | - | 0.595 | - |
| 4 | Iron (Fe) (% w/w) | 0.437 | 0.284 | 0.340 | 0.340 | 0.432 | 0.550 | 0.421 | 1.357 | 0.179 | - |
| 5 | Strontium (Sr) (% w/w) | 0.602 | 0.561 | 0.569 | 0.524 | 0.555 | 0.527 | 0.577 | 1.063 | 0.063 | - |

*Pradyot (2002)

**Table 5: Various Functional Groups of Prepared Dicalcium Phosphate by FTIR Spectrum**

Clam shell powder = 10 g (5.6 g of CaO)
Volume of $H_3PO_4$ = 100 mL
Reaction temperature = 75°C
Reaction time = 30 min
Stirring speed = 500 rpm
Aging time = 24 hr

| Absorption Frequencies of $CaHPO_4$ ($cm^{-1}$) | | | Assignment | |
|---|---|---|---|---|
| Prepared with 1 M $H_3PO_4$ | Analar grade (BDH) | * Literature | | |
| 3450 | 3456 | 3461 | υ O-H | O-H stretching of residual water |
| 2827 | - | 2852 | υ CH | C-H stretching mode |
| 2391 | 2515 | - | $\upsilon_1+\upsilon_3$ | Symmetric stretching and asymmetric stretching vibration of $CO_3^{2-}$ |
| - | 1799 | 1637 | υ O-H | O-H bending and rotation of residual water |
| 1408 | 1423 | 1400 | $\upsilon_3$ | P-O-H in plane bending mode |
| 1132 | - | 1130 | $\upsilon_3$ | P-O stretching mode |
| 1066 | - | 1064 | $\upsilon_3$ | P-O stretching mode |
| 995 | - | 902 | $\upsilon_1$ | P-O (H) stretching mode |
| 891 | 875 | - | $\upsilon_2$ | Symmetric stretching mode of $CO_3^{2-}$ |
| 582 | 709 | 583 | $\upsilon_4$ | O-P-O (H) bending mode |
| 563 | 574 | - | $\upsilon_4$ | O-P-O (H) bending mode |
| 528 | - | 530 | $\upsilon_4$ | O-P-O (H) bending mode |

*Javadpour, (2012)

**Table 6: Effect of Reaction Temperature on the Yield Percent and Phase Formation of Calcium Phosphate**

Clam shell powder = 10 g (5.6 g of CaO)
Volume of $H_3PO_4$ = 100 mL
Strength of $H_3PO_4$ = 1 M
Reaction time = 30 min
Stirring speed = 500 rpm
Aging time = 24 hr

| Sr No | Reaction Temp (°C) | Yield (% w/w) | Phase |
|---|---|---|---|
| 1 | 30 | 67.10 | $CaHPO_4$, $(H_2O)_2$ |
| 2 | 45 | 64.69 | $CaHPO_4$, $CaHPO_4$,$(H_2O)_2$ |
| 3 | 60 | 61.7 | $CaHPO_4$, $CaHPO_4(H_2O)_2$ |
| 4 | 75* | 63.55 | $CaHPO_4$ |
| 5 | 90 | 63.60 | $CaHPO_4$ |

* most suitable reaction temperature

**Table 7: Elemental Composition of Calcium Phosphate Prepared at Different Reaction Temperatures**

Clam shell powder = 10 g (5.6 g of CaO)
Volume of $H_3PO_4$ = 100 mL
Strength of $H_3PO_4$ = 1 M
Reaction time = 30 min
Stirring speed = 500 rpm
Aging time = 24 hr

| Sr. No | Elements | Calcium Phosphate Prepared at Different Temperatures (°C) | | | | | $CaHPO_4$ (Analar grade, BDH) | * Literature |
|---|---|---|---|---|---|---|---|---|
| | | 30 | 45 | 60 | 75 | 90 | | |
| 1 | Calcium (Ca)(% w/w) | 75.939 | 72.312 | 76.271 | 77.683 | 77.485 | 78.851 | 77.23 |
| 2 | Phosphorous (P) (% w/w) | 22.552 | 26.367 | 22.222 | 19.860 | 21.074 | 20.312 | 22.77 |
| 3 | Potassium (K) (% w/w) | 0.684 | 0.614 | 0.691 | - | 0.656 | 0.595 | - |
| 4 | Iron (Fe)(% w/w) | 0.311 | 0.274 | 0.275 | 1.357 | 0.249 | 0.179 | - |
| 5 | Strontium (Sr)(% w/w) | 0.513 | 0.433 | 0.541 | 1.063 | 0.535 | 0.063 | - |

*Pradyot (2002)

**Table 8: Various Functional Groups of Prepared Dicalcium Phosphate by FTIR Spectrum**

Clam shell powder = 10 g (5.6 g of CaO)
Volume of $H_3PO_4$ = 100 mL
Strength of $H_3PO_4$ = 1 M
Reaction time = 30 min
Stirring speed = 500 rpm
Aging time = 24 hr
Drying temp for $CaHPO_4$ = 100 °C

| Absorption Frequencies of $CaHPO_4$ ($cm^{-1}$) | | | Assignment | |
|---|---|---|---|---|
| Prepared at 75˚C | Analar grade (BDH) | * Literature | | |
| 3450 | 3456 | 3461 | υ O-H | O-H stretching of residual water |
| 2827 | - | 2852 | υ CH | C-H stretching mode |
| 2391 | 2515 | - | $\upsilon_1+\upsilon_3$ | Symmetric stretching and asymmetric stretching vibration of $CO_3^{2-}$ |
| - | 1799 | 1637 | υ O-H | O-H bending and rotation of residual water |
| 1408 | 1423 | 1400 | $\upsilon_3$ | P-O-H in plane bending mode |
| 1132 | - | 1130 | $\upsilon_3$ | P-O stretching mode |
| 1066 | - | 1064 | $\upsilon_3$ | P-O stretching mode |
| 995 | - | 902 | $\upsilon_1$ | P-O (H) stretching mode |
| 891 | 875 | - | $\upsilon_2$ | Symmetric stretching mode of $CO_3^{2-}$ |
| 582 | 709 | 583 | $\upsilon_4$ | O-P-O (H) bending mode |
| 563 | 574 | - | $\upsilon_4$ | O-P-O (H) bending mode |
| 528 | - | 530 | $\upsilon_4$ | O-P-O (H) bending mode |

*Javadpour (2012)

**Table 9: Physico-chemical Properties of Dicalcium Phosphate**

| Sr. No | Properties | Prepared $CaHPO_4$ | Analar Grade $CaHPO_4$ | *Literature |
|---|---|---|---|---|
| 1 | Appearance | powder, white, odorless | powder, white, odorless | powder, white, odorless |
| 2 | Moisture content (%) | 1.5 | 3 | 2.92 |
| 3 | pH (10% solution) | 3.6 | 3.1 | 3.4 |
| 4 | Solubility | 0.5 | 0.35 | 0.31 |
| 5 | Specific gravity | 2.3 | 2.4 | 2.3 |

*MSDS Code 16 (www.potashcorp.com)

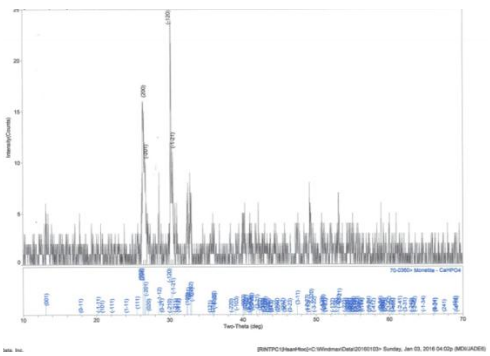


**Fig 4:** XRD pattern of Prepared $CaHPO_4$

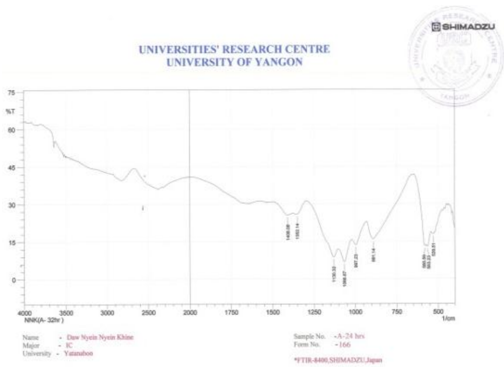


**Fig5:** FTIR pattern of Prepared $CaHPO_4$

**Table 10: Effect of Setting Solution on Properties of Monetite Cement**

Weight of dicalcium phosphate = 100 g

| Sr. No | Setting Solution (Deionized water) (mL) | Initial Setting Time (min) | Final Setting Time (min) | Compressive Strength (MPa) | Expansion | Bending Strength (MPa) |
|---|---|---|---|---|---|---|
| 1 | 50 | 7 | 15 | 1.11 | ND | ND |
| 2 | 70 | 7 | 18 | 2.51 | ND | ND |
| 3 | *90 | 10 | 21 | 2.53 | ND | ND |
| 4 | 110 | 22 | 38 | 2.43 | ND | ND |
| 5 | 130 | 30 | 45 | 2.24 | ND | ND |

* most suitable setting solution
Monetite cement = Dicalcium Phosphate Cement, ND = not detected
These experiments were conducted at Cement Factory, NCDC, Naypyitaw.

**Table 11: Effect of Different Amounts of Citric Acid (Setting Solution) on Properties of Monetite Cement**

Weight of dicalcium phosphate = 100 g
Volume of citric acid = 90 mL

| Sr. No. | Setting Solution (Strength of Citric Acid, (%) | Initial Setting Time (min) | Final Setting Time (min) | Compressive Strength (MPa) | Expansion | Bending Strength (MPa) |
|---|---|---|---|---|---|---|
| 1 | 0.5 | 6 | 10 | 1.24 | ND | ND |
| 2 | *1 | 5 | 11 | 2.81 | ND | ND |
| 3 | 1.5 | 8 | 20 | 2.83 | ND | ND |
| 4 | 2 | 15 | 30 | 2.62 | ND | ND |
| 5 | 2.5 | 20 | 35 | 2.43 | ND | ND |

* most suitable setting solution
Monetite cement = Dicalcium Phosphate Cement, ND = Not Detected
These experiments were conducted at Cement Factory, NCDC, Naypyitaw.

**Table 12: Effect of Dicalcium Phosphate (Abrasive) on pH of Toothpaste**

| Sample No. | Ingredients | | | | | | | | | | | pH |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Dicalcium Phosphate | | Sodium Fluoride | Glycerine | Sodium Lauryl Sulphate | Sorbitol | Titanium Dioxide | Xanthan Gum | Methyl Paraban | Pepermint Oil | Distilled Water | |
| 1 | processed DCP | 20 | 0.5 | 33 | 2 | 0.2 | 5 | 2 | 0.05 | 1 | 30 | 6.7 |
| 2 | | 25 | | | | | | | | | | 6.8 |
| 3 | | 30* | | | | | | | | | | 7.0 |
| 4 | | 35 | | | | | | | | | | 7.6 |
| 5 | | 40 | | | | | | | | | | 7.9 |
| 6 | analar grade DCP (BDH) | 20 | | | | | | | | | | 6.9 |
| 7 | | 25 | | | | | | | | | | 7.1 |
| 8 | | 30 | | | | | | | | | | 7.5 |
| 9 | | 35 | | | | | | | | | | 7.9 |
| 10 | | 40 | | | | | | | | | | 8.1 |

* most suitable amount of dicalcium phosphate

**Table 13: Physico-chemical Properties of Formulated Toothpaste**

| Sr. No | Characteristics | Formulated Toothpaste | Commercial Toothpaste (Laser) | *Literature |
|---|---|---|---|---|
| 1 | Colour | white opaque | white opaque | white opaque |
| 2 | Texture | smooth | smooth | smooth |
| 3 | Taste | slightly chalky | slightly chalky | slightly chalky |
| 4 | pH | 7.0 | 8.2 | 7 - 8 |
| 5 | Viscosity (cP) | 210000 | 230000 | 170000 - 200000 |
| 7 | Density ($gcm^{-3}$) | 1.3 | 1.26 | 1.3 |
| 8 | Akalinity (ppm) | 19.6 | 20 | 20 - 40 |
| 9 | Moisture content (%) | 27 | 30 | 30 |
| 10 | Foaming power (mL) | 160 | 170 | > 150 |

* Oyewale.A.O (2004)

## Conclusion

This research work confirmed the successful production of dicalcium phosphate from waste clam shells. The using of waste materials can cut off the cost of synthesis material since the quality and purities of the product almost same as the analar grade calcium salts. This work made to generate economic return from wastes and to meet the local requirement of calcium salts for the appropriate purposes. The parameters such as acid concentration, reaction temperature and time, stirring speed and drying temperature had considerable effect on the formation of dicalcium phosphate and the compositions of the products. During the processing of dicalcium phosphate, the concentrations of acid effect on the phases formation of the product whether pure phase or combined phases. Correlation of temperature, time and stirring speed undoubtedly defined the composition and morphological nature of the individual calcium salts. As clam shell is a source of biocalcium, biocalcium rely to strong the bones throughout the life. So, in this research, the prepared dicalcium phosphate was applicable in the monetite bone cement and biocalcium toothpaste. Hence the novelty of this study was actually the noteworthy conversion of waste clam shell to dicalcium phosphate for use in producing health and wellness products toward a healthy and fulfilling life.

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