UNIVERSITI SAINS MALAYSIA

# FINAL RESEARCH UNIVERSITY (RU) REPORT

**Title Project**

**BIOMATERIAL DENTAL APPLICATION OF BIPHASIC CALCIUM PHOSPHATE (BCP)**


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# TECHNICAL REPORT
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UNIVERSITI SAINS MALAYSIA

# PREPARATION OF BIPHASICS CALCIUM PHOSPHATE (BCP) DOPED WITH ZIRCONIA

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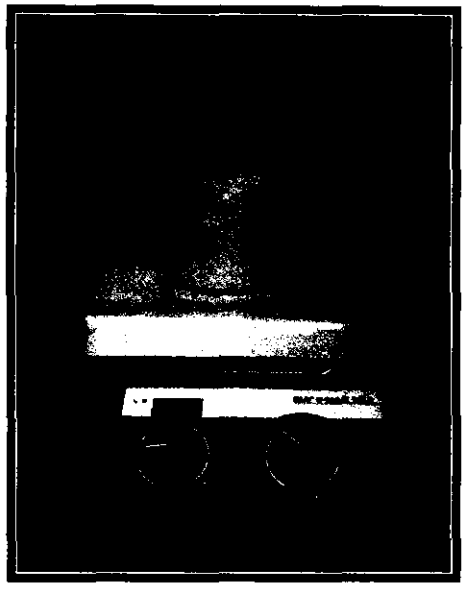
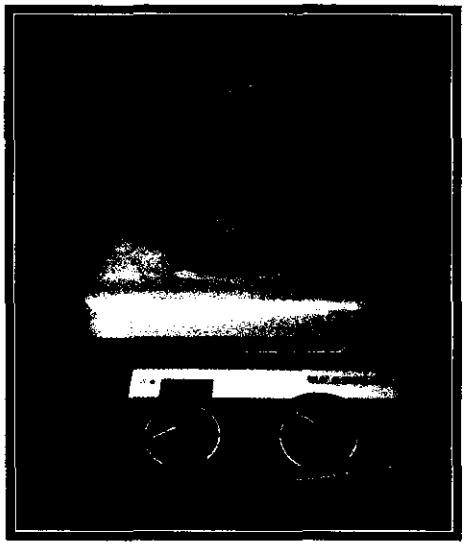
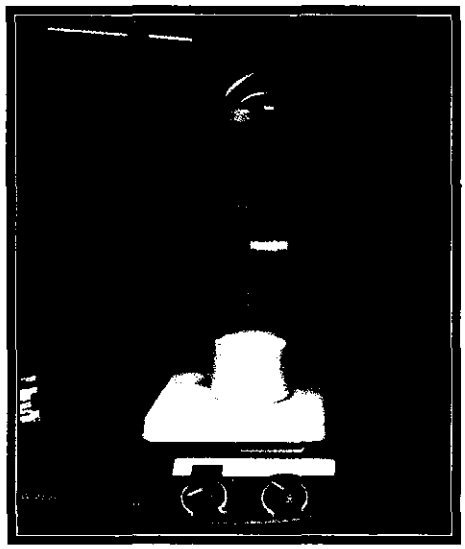


60g of calcium hydroxide, $Ca(OH)_2$ was diluted into the 300ml distilled water. The solution was stirred using a magnetic bar at 70°C for 2 hours. The solution was cooled to room temperature.

5g of zirconyl chloride octahydrate is diluted into the 150ml 4M HCl. The solution was stirred and heated at 70°C with optimum duration until zirconyl chloride octahydrate is fully dissolved.



The solution was mixed with 300ml di-(2-ethyl-hexyl)-phosphate (DEHPA) and stirred for 20 minutes



The solution was separated by using separating funnel. Two layers were formed; the bottom layer (supernatant) was discarded while the upper layer (organophosphate solution) was kept for the further step. The solution of organophosphate was dropped slowly into the $Ca(OH)_2$ and stirred continuously. The solution was left overnight.

.After leaving overnight, the precipitation was formed.

Filtering process using vacuum machine

Drying process using oven universal

Calcinations in the furnace at 1000°C for 3 hours

BCP powder

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# CHARACTERIZATION OF BIPHASIC CALCIUM PHOSPHATE (BCP) DOPED WITH ZIRCONIA AND BETA TRI-CALCIUM PHOSPHATE (β-TCP) FOR DENTAL APPLICATION

DR. SAM'AN MALIK MASUDI

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# CHARACTERIZATION OF BIPHASIC CALCIUM PHOSPHATE (BCP) DOPED WITH ZIRCONIA AND BETA-TRICALCIUM PHOSPHATE ( β-TCP) FOR DENTAL APPLICATION

## 1.0 OBJECTIVE

To determine the characterization of calcium phosphate doped with zirconia and beta-tricalcium phosphate (β-TCP) by Scanning Electron Microscope-Energy Dispersive X-ray (SEM-EDX), Transmission Electron Microscope (TEM), Fourier Transform Infrared (FTIR) and X-ray Diffraction (XRD) techniques.

## 2.0 INTRODUCTION

### 2.1 Calcium phosphate with zirconia

Calcium phosphate, $Ca_3(PO_4)_2$ is the name given to a family of minerals containing calcium ions ($Ca^{2+}$) together with orthophosphates ($PO_4^{3-}$), Calcium phosphate ceramics have gained wide-spread attention due to their compositional and structural similarity to the mineralized constituent in hard tissues ( Hench, 1991 ). Hydroxyapatite is the main mineral constituent of natural bone, and thus synthetic calcium phosphate ceramics may create an excellent bond with natural tissue and can even stimulate new bone growth. These ceramics have been used extensively for dental applications, bone grafts or as bioactive coatings. However, due to their relatively low strength and toughness, susceptibility to physiological attack, and poor fatigue properties, the biomedical uses of calcium phosphate ceramics are limited to non-load bearing applications (Mansur et. al.,1996)

One of the most promising approaches to increase the strength in ceramics is through transformation toughening based on the tetragonal-monoclinic transformation of zirconia (Christel, 1989). A ceramic with dispersed tetragonal zirconia can be used to enhance the strength and toughness. In this case study, zirconia and calcium phosphate powders were used to produce a biocomposite.

## 2.2 Beta-tricalcium phosphate ( β-TCP)

β-tricalcium phosphate (β-TCP) is a calcium phosphate ceramic used as an alternative bone substitute (to autograft) in bone grafting. It has been reported that β-TCP has excellent osteoconduction and resorbability when filling the bone defect. (Hirokazu Komaki et al.)

β-TCP containing ceramics displays affinity for high speed biological degradation. However in TCP, the biodegradation is much higher than the growth rate of natural bones (Spivak and Hasharoni,2001 ; Kivrak and Cuneyt Tas,1998)

## 3.0 METHODOLOGY

### 3.1 Scanning Electron Microscope-Energy Dispersive X-ray (SEM-EDX)

Energy Dispersive X-ray (EDX) spectrum imaging has been performed in a scanning electron microscope (SEM) on the sample to characterize the elemental distribution near the interface.

Spectrum imaging, where a complete x-ray spectrum is collected at every pixel in a scanned image, is a powerful new tool for materials characterization. Spectrum imaging differs from mapping, where only windows around pre-selected energy ranges are acquired, in that the entire spectrum is collected at each pixel, as the beam is rastered across the specimen (Paul et al., 2000).

The analysis was performed in a Leo Supra 50 VP Field Emission SEM equipped with Oxford INCA 400 energy dispersive x-ray microanalysis system.

Figure 1 : Leo Supra 50 VP Field Emission SEM

## 3.2 Transmission Electron Microscope (TEM)

### 3.2.1 TEM procedure for sample

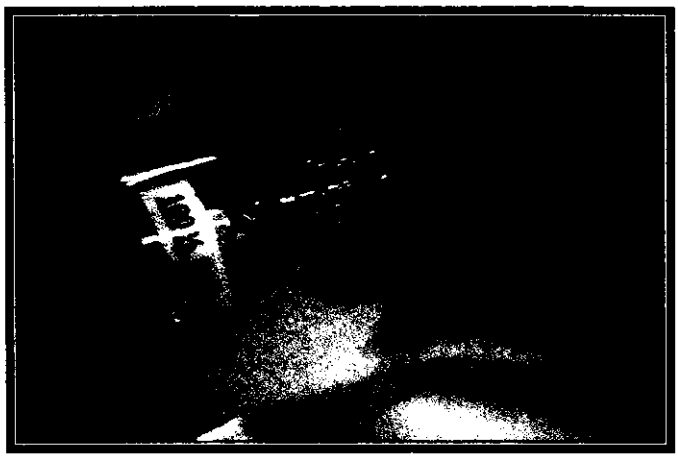

Figure 2 : Suspend sample in ethanol

Figure 3 : Place a droplet* of the suspension on a carbon film coated 400 mesh copper grid for 1-3 minutes**

* The drop should be pipetted out after the larger particles have settled to the bottom of the sample tube

** Time varies and is dependent on the thickness of the suspension

Figure 4 : The droplet is then wicked to dryness using pieces of filter paper

Figure 5 : Place the grid in a filter paper lined petri dish until it can be examined ( Label : user, date, sample code )

Figure 6 : Clean the fine forceps used with ethanol / filter paper before storing it away