

Chapter 1

Introduction

1.1 Motivation, Objective and Scope

Motivation:

Bioceramic is a material that is important for medical application especially calcium phosphate compounds. Calcium phosphate compounds have long been investigated as bone and tooth implant materials because they closely resemble vertebrate bone and tooth materials. They have been proven to be biocompatible materials. Dicalcium phosphate dihydrate is one of calcium phosphate compounds that suitable for dental application, for example, as composition of self-setting cement, dentifrice polishing agent, mouthrinse, gum, medicine and etc. At present, pure dicalcium phosphate dihydrate suitable for dental application is expensive because pure chemicals used as raw materials are expensive too.

In Thailand, cattle are not only used for an agrarian but also used for food. In the past, cattle bone was useless

but now it is used as raw material to prepare fertilizer. It is known that cattle bone consists of calcium phosphate. A successful preparation of hydroxyapatite (OHAp, $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) from cattle bone was reported by Lorprayoon (1989) but there has been no report on dicalcium phosphate dihydrate (DCPD, $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$). Therefore it is very interesting to prepare this material from cattle bone. As a result, a research project with the title "Preparation of dicalcium phosphate dihydrate from cattle bone" is proposed.

Cattle bone is available in Thailand at very low cost and it is expected to be a cheap source for calcium phosphate. Among the important calcium phosphate compounds, dicalcium phosphate dihydrate is a common one. Besides the mentioned applications, dicalcium phosphate dihydrate is also a starting material for preparing other phosphate compounds, especially hydraulic calcium phosphates.

Objective:

The objective of this study is to prepare dicalcium phosphate dihydrate from cattle bone, characterize the prepared dicalcium phosphate dihydrate and use it as a starting material to prepare α -tricalcium phosphate (α -TCP),

a hydraulic calcium phosphate.

Scope:

The preparation of dicalcium phosphate dihydrate from cattle bone was based on the dissolution of the bone ash in acid followed by precipitation of dicalcium phosphate dihydrate under various conditions. α -Tricalcium phosphate was prepared by heating the calcium phosphate dihydrate with calcium carbonate.

The sequence of the experiment was: preparation of bone ash, characterization, dissolution in acid, precipitation dicalcium phosphate dihydrate from the bone ash solution under various conditions, characterization of the dicalcium phosphate dihydrate obtained and preparation of α -tricalcium phosphate from the dicalcium phosphate.

1.2 Literature Survey

Most of the highly pure dicalcium phosphate dihydrate had been manufactured by a procedure which was represented by the step of first conducting a reaction between a refined calcium salt and expensive dry process phosphoric acid (Cremer et al. 1966, and Brachtel et al. 1988), filtering and washing the precipitates of dicalcium phosphate dihydrate formed from the said reaction. The dicalcium phosphate dihydrate which was obtained from the reaction between the relatively cheaper wet process phosphoric acid and a calcium salt had a colour hue and contained a number of impurities. Therefore it was impossible to obtain the desired highly pure dicalcium phosphate dihydrate.

Enomoto et al. (1977) and Hayakawa et al. (1969) tried to prepare a highly pure dicalcium phosphate dihydrate. The experiment had been developed after an extensive research to find a method for producing at low cost, highly pure dicalcium phosphate dihydrate containing no substances detrimental to the health of humans and animals, by adding ammonia and a basic sodium salt to impure wet process phosphoric acid to precipitate and separate crystals of sodium ammonium hydrogen phosphate ($\text{NaNH}_4\text{HPO}_4 \cdot 4\text{H}_2\text{O}$) or so-called

microcosmic salt, and by mixing a calcium salt such as calcium chloride to a solution of the said microcosmic salt to precipitate dicalcium phosphate dihydrate ($\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$) while maintaining pH and temperature of the mixed liquid at 3.5 to 5.0 the temperature thereof at 45°C, or lower and the calcium to phosphorus mole ratio (Ca/P) thereof at 1.0 to 1.3.

In the case of tricalcium phosphate, β -tricalcium phosphate was prepared from cattle bone ash by dissolution in nitric acid, consecutive precipitation with ammonium hydroxide at pH 8.0-8.5 and heating to 1230°C and 1280°C within 2 to 3 hours in an electric furnace (Lorprayoon, 1989). In addition, Pauchiu and Wang, 1993, β -tricalcium phosphate was observed from sintering at 1100°C for 4 hours in air of hydroxyapatite. Upon sintering at 1300°C tetracalcium phosphate appeared. During sintering at 1350°C some of the β -tricalcium phosphate converted to α -tricalcium phosphate.