



Development of machinable hydroxyapatite-lanthanum phosphate composite for biomedical applications



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ABSTRACT

To develop a bioceramic composite that can be machined to any shape and dimension will be of great importance and demanding material as an implant. Calcium phosphate (CaP) ceramics have high hardness that restricts the machinability of these materials and limits the wide applicability due to dimensional restrictions. Development of a machinable bioceramic composite by incorporation of a weak interphase material is done in the present study. Hydroxyapatite (HAp) and lanthanum phosphate (LP) powders were prepared chemically, mixed and pressed to form composites (LP, 10–50 wt.%) followed by sintering up to a temperature of 1200 °C. The products were characterized by phase analysis, densification, mechanical behavior, bioactivity, and biocompatibility. Machinability study of the sintered composites was done by using solid carbide (SC) drills in a conventional radial drilling machine. Phase analysis showed no reaction between the reactants and confirms the composite character till 1150 °C. Increasing LP content resulted in reduced densification, flexural strength, hardness with the incorporation of machinability. Positive bioactivity and biocompatibility were observed for the composites sintered up to 1150 °C. Sintering at 1200 °C results in a reaction between HAp and LP and formation of a new compound in the system Ca-P-La-O, hampering composite nature and destroying machinability.

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1. Introduction

During the last four decades, ceramic materials and composites have created a major revolution in the medical science by improving the quality of human life through the use of artificial bone, eye, dental fixtures, etc. Artificial ceramic implants not only serve as an additional support but also as an internal prosthetic, which when integrated into the human system, fully perform the function of the skeletal materials they replace. These special classes of tailor made bioceramic materials show specific biological/functional/chemical activities in living systems. Treatment procedures for reconstructive surgery have resulted in marked improvement in the quality of life of a rehabilitated person.

Calcium phosphate (CaP) based ceramics are widely used as bone substitutes in orthopedic, maxillofacial, or spine surgery due to their chemical similarity with the inorganic part of the bone [1–4]. Several studies have shown that CaP based implants are proactive and osteoconductive. After implantation into bone defects, CaP ceramics interact with body fluids, leading to partial dissolution and precipitation

of biological carbonated apatite onto their surface [5–6]. The apatite layer formed *in vivo* contains various endogenous proteins and is colonized by osteoblastic cells producing the bone extracellular matrix [7]. As a result of this bioactivity, CaP ceramic guide bone healing on their surface using osteoconduction [8]. CaP based ceramics are osteoconductive and bio-resorbable and find applications as granules, sintered macro-porous body for a bone substitute and as a coating on metallic implants used in orthopedics [8,9]. Machinable property in these materials is beneficial, especially for large-size or geometrically complex shapes since it is possible to produce implants with a high dimensional accuracy which guarantees optimal implant integration into the bone defects.

Ceramic materials have very strong atomic bonding resulting in high strength and hardness. However, extremely high hardness causes serious problems in conventional machining. Machining is often required for quality and shape control of a component. The high cost of machining a ceramic component, involving diamond tools, is the primary impediment to the widespread use of advanced ceramics. The phenomenon is also common for CaP ceramics, which are very brittle and have a very limited ability to be machined into accurate shapes and sizes required for diverse biomedical applications. The only way to improve the flexibility of using these materials is to impart machinability.

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Imparting machinability in hard ceramic materials requires technological designs to develop a multi-phase ceramic composite where one phase will act as weak material to pass through the crack of machining whereas the main ceramic portion will retain its properties. Hence, the judicious design of a composite, selection of the components of the composite, synthesis of the components, preparation of the composite, retaining the composite nature, even after sintering the ceramic composite and their characterization are all important for such developments. And such a product development work is undertaken in the present work. Hydroxyapatite [HAp, $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$] is taken as the bioceramic component as it is most common bio-ceramic material due to its chemical similarity to the inorganic component of bone mineral.

In a composite matrix, prolonging the crack route at the weak interfaces, interfacial debonding and crack deflection between the two phases imparts machinability [10–14]. Rare earth phosphates (REP) are found to act as weak interphases in a ceramic composite matrix system and can impart machinability [15–19] by inducing interfacial debonding and crack deflection. They act as weakly layered interphase, allowing the crack generated by machining, preferentially to pass through them, thus allowing the machining process without fracturing of the ceramic materials. REP are biologically non-toxic, biocompatible and chemically [20] and thermally stable [21–23]. REPs have various biomedical applications also mainly as bio-imaging phosphor/luminescent labeling materials for bio-imaging [24,25].

But much work is not available in imparting machinability in bioceramics. Lanthanum phosphate [LP, LaPO_4] has been reported to impart machinability in some ceramic matrix such as alumina, zirconia due to its layered structure and weak interface [14,15,17]. It acts as a weak interphase material in a ceramic matrix composite system, causes debonding and preferentially allows the cracks to pass through it and make the ceramic machinable. This effect of weak interphase, causing debonding in the ceramic matrix composites continues till the material remains as composite and LP remains as separate phase [17,20,22]. Any reaction of the LP with the ceramic system ceases the debonding mechanism and also the machinability. Lanthanum phosphate also showed good thermal stability, bioactivity and biocompatibility results when introduced in alumina matrix for machinability [26–28]. Recently the machinability of tri-calcium phosphate (TCP) and LP [29,30] composite has been reported, and also the osteoblast adhesion properties of this material have been studied [31]. HAp - yttrium phosphate (YP) and TCP-YP composites were also found to be machinable and biocompatible [32,33]. However, many areas are remaining unexplored, and many questions unanswered. So a detailed investigation is very much required to develop machinable bioceramics based on HAp-LP composite.

In the present work, sintered composite of HAp, with a weak inter-phase bonding material LP is studied up to an LP content of 50 wt.%. Sintered composites were characterized for various properties including drillability (machinability) by solid carbide drill bits in a conventional radial drilling machine, bioactivity, and biocompatibility study.

2. Experimental

2.1. Synthesis of HAp and LP powder

HAp is synthesized by chemical coprecipitation route, as proposed by Hayek et al. [34] and practiced in our earlier work [32]. Analytical reagent (A. R.) grade calcium nitrate tetrahydrate [$\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$], di-ammonium hydrogen phosphate [$(\text{NH}_4)_2\text{HPO}_4$] and ammonia solution (NH_4OH) (all from Loba Chemie, India) are used as per the following reaction:

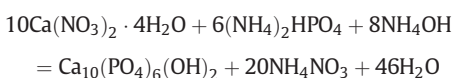
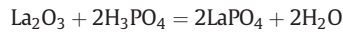


Table 1
Batch composition.

| HAp-LP composite (abbreviation) | HAp (wt.%) | LP (wt.%) |
|---------------------------------|------------|-----------|
| H0 | 100 | 0 |
| HL1 | 90 | 10 |
| HL2 | 80 | 20 |
| HL3 | 70 | 30 |
| HL4 | 60 | 40 |
| HL5 | 50 | 50 |

LP was prepared by the method proposed by Wang et al. [35], using A. R grade lanthanum oxide [La_2O_3] and orthophosphoric acid [H_3PO_4] (all from Loba Chemie, India), as per the following chemical reaction:



Both the powders were calcined to get their respective forms and then phase purity was confirmed by powder X-ray diffraction (XRD) technique (Rigaku, Japan make). Phase stability of HAp and LP powders were also studied by firing the powder up to 1200 °C and then by phase analysis.

2.2. Preparation of HAp-LP composite

HAp-LP composites were prepared by powder mixing method using isopropyl alcohol as the liquid medium, varying the LP content between 10 to 50 wt.%. The batch compositions are given in Table 1. Mixed batches were shaped in pellet form by uniaxial pressing in a tool-steel die of 15 mm diameter at 100 MPa specific pressures in a uniaxial hydraulic press (Carver, USA, make) with 6% PVA solution as a green binder. Shaped pellets were then sintered at 1100°, 1150° and 1200 °C with 2 h soaking time in a programmable electrical resistance furnace (Bysaskh & Co, India make).

2.3. Characterization of the composites

2.3.1. Phase analysis

The sintered pellets were characterized by phase analysis in X-Ray powder diffractometer (Rigaku, Japan) fitted with Ni filter. The diffraction patterns were measured using $\text{Cu}_{\text{K}\alpha}$ ($\lambda = 1.5418 \text{ \AA}$) radiation generated at a voltage of 40 kV and a current of 40 mA. The samples were scanned in the interval $20^\circ < \theta < 50^\circ$ at a scanning rate $20^\circ/\text{min}$ with step size 0.05° in a continuous mode. XRD analysis of the powder samples was performed using X'pert High Score software.

2.3.2. Densification study

Densification studies of the composites sintered at all the temperatures were done by the conventional liquid displacement technique according to the modified Archimedes principle for porous samples using

Table 2
Chemical composition of SBF solutions.

| Order | Reagent | Amount (g/l) |
|-------|---|--------------|
| 1 | NaCl | 6.547 |
| 2 | NaHCO ₃ | 2.268 |
| 3 | KCl | 0.373 |
| 4 | N ₂ HPO ₄ · 2H ₂ O | 0.178 |
| 5 | MgCl ₂ · 6H ₂ O | 0.305 |
| 6 | CaCl ₂ · 2H ₂ O | 0.368 |
| 7 | Na ₂ SO ₄ | 0.071 |
| 8 | (CH ₂ OH) ₃ CNH ₂ | 6.057 |

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