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Mimicking Bone Architecture in a Metallic Structure

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Abstract. The porous metallic structure has been developed to mimic the natural bone architecture, having interconnected porosity, disposing enough room to cell migration, anchoring, vascularization, nourishing and proliferation of new bone tissue. The titanium is used as porous implants due its excellent mechanical properties and biological interaction. Research evolving porous titanium has been done with purpose to achieve desirable pore size, total porosity percentage and influence of those in the increasing of bone-implant bond strength interface. Were prepared samples of titanium by powder metallurgy adding natural polymer: corn starch, rice starch, potato starch and gelatin; at proportion of 16 wt%. In aqueous solution the hydrogenated metallic powder (TiH_2) and the polymer were mixed, homogenized and frozen in molds near net shape. The water was removed in kiln (38°C/12h) and the polymer by thermal treatment with air-oxidation (350 ºC/1h) before sintering in high-vacuum (1300 ºC/1h). Resulting from the process, the obtained pores by addition of potato and corn starches, lead to homogenous and well distributed throughout structure. Samples obtained from addition of rice starch and gelatin formed macropores and micropores randomly distributed within the structure. The apparent porosity for all samples was near 40%. The processing technique allowed the open pore formation, in which the macropores mimics the trabecular bone structure and micropores allows the bone-implant anchorage.

Introduction

Metallic biomaterials are widely used in medical areas, such as orthopedics, and dentistry, and aim to meet the needs of replacement or repair of tissues (organs) lost or injured. The main application of metallic biomaterials is as endosseous implants, among the requirements to fulfill such task these materials must have biocompatibility and biofunctionality [1].

The bone tissue is the main responsible for providing stability and support, and in this way, the receiving body of all systems of orthopedic and dental implants. Besides being a highly specialized tissue support, is able to modify their own architecture to meet the physical and metabolic factors. In the process of repair, the healing of the bone / implant interface passes through the same steps as a direct bone fracture, following an orderly sequence of events. After primary stabilization and serum protein adsorption on the implant, the initial bone healing takes place with the formation of coagulum between the bone and the implant, with subsequent clot organization allowing cells to adhere at the implant surface and forming blood vessels. Osteoprogenitor cells proliferate and differentiate in this organized environment into osteoblasts, thus promoting the deposition of mineral content to form the bone tissue throughout the implant surface [2].

The topography of the implant is one of the factors that influence the process of bone repair to osseointegrate the implant. Although implants machined have been used for many years, studies have shown that the increase in surface roughness tends to enhance, not only the surface area between the bone and the implant, but also the bond strength of the interface [3]. Among the modifications to prevailing topography, the porosity is, in implants, quite interesting because it allows the occurrence of the phenomenon of tissue invasion into the pores, known as "Bone ingrowth" [4]. The porous implants must have interconnected porosity with spaces that allow the maintenance required for vascular continuous nourishing for mineralization of bone tissue. Thus, pore channels, must have sufficient size to the infiltration of cells responsible for formation of the tissue matrix within the material, in order to meet the requirements for a lasting healing rehabilitation [5].

Titanium and its alloys are the main metals studied as porous implants by its excellent mechanical properties, biological interactions and formed passivation layer. Research on porous titanium have been carried out in order to analyze issues related to the size of pores, porosity and degree of their influence in increasing the bond strength of the bone-implant interface as well the bone ingrowth [6]. The pore size is a subject addressed by many authors due to its direct influence in the migration and maturation of osteoprogenitor cells. Some authors also determine that no vascularization occurs in pores of diameter less than 100 µm, and the reported desirable pore size fitted to proper bone and vascular reorganization, range between 100 and 500 µm [4, 5, 7].

Methods for production of porous metallic materials are based on powder metallurgy (PM). This technique allows the production of parts with complex shapes and dimensions close to the finals, near-net shape, avoiding the step of machining [6]. The manipulation of the metals in the form of particulate allows the addition of elements reaching a satisfactory structural homogeneity, and porosity [8, 9]. The production of porous structures have been proposed by various methods, such as suspension [10], space-holder [8], metal injection mold (MIM) [9], freeze-cast [11], electron beam melting (FFE) [12] and prototyping [13].

Natural polymers such as starches, are used successfully in the production of porous ceramic [14], and can be adapted to process metals for the same purpose. With sacrificial template techniques, such as suspension and space-holder, the starch is removed by thermal treatment, forming pores in the spaces once previously occupied by it. In the case of metals, oxidation during heat treatment may be a hindrance and can weaken the structure, thus to counteract these undesirable effects, hydrates can be used to mitigate this shorthand. Regarding the technique of gel-casting, or suspension, the starch has the function of gelling agent by absorbing water and expanding volumetrically, actions relevant to the production of pores in the structure [15]. Other natural polymer that can be applied as additive is gelatin, a collagen based substance, which has properties of biocompatibility, biodegradability and non-toxicity [10]. This study aims to apply technique of obtaining porous metal implants using such polymers.

Experimental Procedure

Hydrogenated commercially pure titanium (cpTi) powder was used in this study, the morphology was characterized by scanning electron microscopy (SEM) (Philips XL 30); crystalline phases by X-ray diffraction (XRD) (DMAX 2000, Rigaku) with Cu-Kα radiation; particle size distribution by granulometry (CILAS, granulomer – 1064); mass deviation by thermogravimetry (SHIMADZU 60WS), the results of hydride cpTi powder was compared with values obtained with metallic cpTi powder.

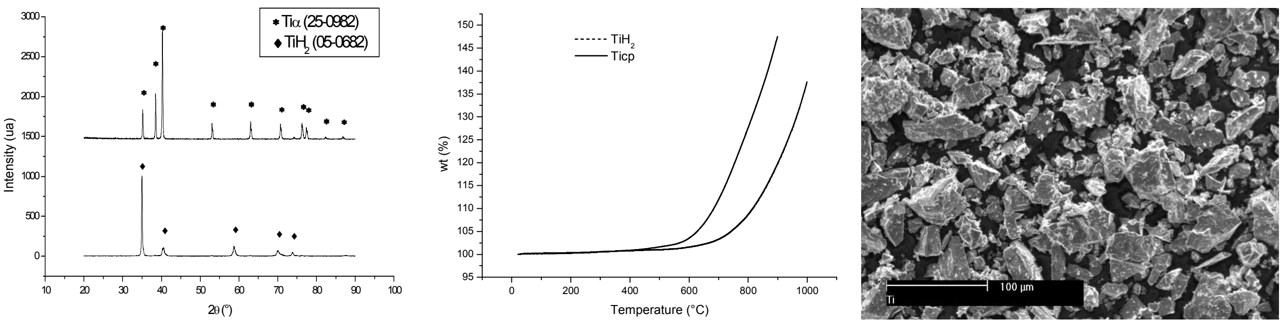
One of the used technique to obtain porosity involves pores formation by suspension added of metal, using the metal powder in a suspension of water and natural polymers (starch from corn, potato, rice and gelatin). The ratio used was 16% by weight of polymer from the total solids in hot water. Cylindrical molds were filled with the formed paste with aid of a syringe to remove bubbles. Immediately after filling the mold, it was frozen in liquid nitrogen and the samples removed, they were placed in a freezer (-10°C). After 12 h, samples were placed in a kiln (38°C) prior to thermal treatment and sintering. All samples were heat treated in an oxidizing atmosphere at 350°C for 1 hour with heating rate of 1°C/min. This heat treatment was necessary for the decomposition of organic material and removing carbon from the same. The samples in the crucible of alumina (Al2O3) were sintered in a furnace of tungsten heating element with high vacuum (10-5 mBar), 1300°C for 1 hour.

Sintered samples were characterized for density by Archimede's method (ρ), morphology by SEM; crystalline phases by XRD with Cu-Kα radiation; and roughness (SJ-201, Mitutoyo). The apparent porosity was calculated according to the standards of ASTM C20-00 [16].

Results and Discussion

To confirm the crystalline phase of the TiH2 and cpTi raw material, XRD analysis was performed and only TiH2 phase and α-phase were found respectively (Fig.1A). The use of hydrogenated powder facilitates the processing of porous material, because metallic powder can suffer oxidation when in contact with the aqueous solution and during the heat treatment step. As the mechanism of pore formation is the result of degradation of organic material by raising the temperature, it is necessary to perform the heat treatment in an oxidizing atmosphere for the removal of natural polymers added before sintering in vacuum. The behavior of both cpTi metallic and the hydride powders was analyzed by thermogravimetric analysis, which promoted the air-oxidation of metals by the rising temperature, interfering directly on the structure of material during sintering. The oxidation of metallic cpTi begins at 500°C, as temperature rises there is significant mass increase (Fig. 1B). On the other hand the oxidation of particulate TiH2 begins at 700°C, hence it has greater stability than metallic cpTi. This happens because prior to the oxidation the particulate TiH2 needs to dehydrogenate, process which took place at ~600°C (Fig. 1B). Based on these findings, the chosen material to perform the samples were the hydrides, due its greater stability to oxidation when compared to cpTi.

In powder metallurgy, morphology and size of the powder used is very important to the sintering process. The morphology analysis performed by SEM of TiH_2 showed particles of irregular shapes (Fig.1 C). By the particle size distribution, the average particle diameter was 43 µm.



A B C

Figure 1. A) TiH_2 powder XRD. B) TiH_2 powder and metallic cpTi powder thermogravimetry. C) TiH_2 powder morphology by SEM.

The values of apparent porosity of the groups were calculated after sintering. There is no significant difference among porosity values when using different natural polymer additives, the mean porosity was ~ 40% (Table 1). The choice of these additive natural polymers came not only by the percentage of porosity, although they had a heterogeneous and homogenous distribution, depending on the additive, the rounded shape of pores and size around 100 µm were determinant to the selection. The roughness were similar for all samples (Table 1), as shown on implant's surface low magnifications images by SEM (Fig. 3A, Fig. 4A, Fig. 5A, Fig. 6A).

Table 1. Porosity and roughness of the samples

Natural Polymer	Corn starch	Potato starch	Rice starch	Gelatin
Apparent Porosity	43%	40%	43%	38%
Roughness [Ra]	6,17 µm	5,36 µm	5,31 µm	4,71 µm

After sintering, there is only one phase, the α-phase in the structure (Fig. 2A). The sintering parameters used for this study were very efficient, there was successfully formation of necks between particles. The particle shape was roundish, as shown on implant's surface (Fig. 3B, Fig. 4B, Fig. 5B, Fig. 6B).

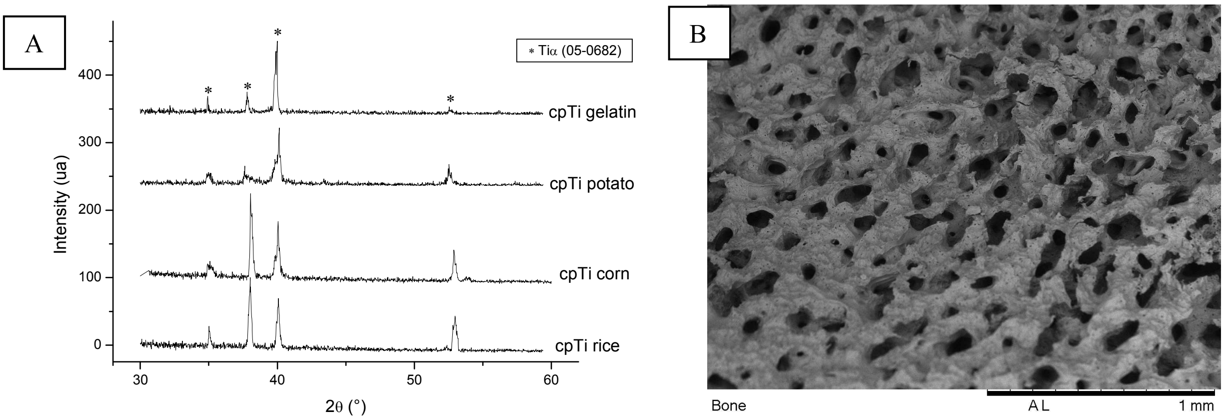


Figure 2. A) XRD of porous samples with respective natural polymer. B) SEM of trabecular bone.

The image of trabecular bone presented here (Fig. 2B), represents the goal of the mimicking metallic structures by the studied technique. Analyzing the image, there is an overt highly porous structure, it is possible to observe the microarchitecture, porosity, pore size, and distribution of trabecular bone.

Samples added of corn starch resulted in smaller and homogeneous pores in the structure (Fig. 3), on the other hand the addition of potato starch resulted in slightly larger pores, homogeneously and well distributed (Fig. 4). The rice starch provided a varied pore size, although well distributed in the structure (Fig. 5). The gelatin added, provided the formation of pores with heterogeneous size and distribution (Fig. 6).

The bone microarchitecture is an important issue because it relates directly to bone biomechanical properties. Besides, microarchitecture is one feature involved and determinant in the quality of formed bone. The bone natural microarchitecture variation, happens most to mechanically adapt the bone to support loads combined to a vary number of factors related to nutrition, metabolism, genetic, diseases and aging [17].

Considering the overall appearance based on the microstructure of samples, the rice starch promoted the most similar sample compared to the bone tissue microstructure, Fig. 2B. The corresponding pore size and similar design were achieved, ranging around 100 μm. As reported in literature the pore size is responsible to allow the proper bone deposition, resorption and nourishing though neoformed vasculature [7], it is important to allow the bone ingrowth during the healing process. The microarchitectural design drawn to mimic the bone tissue shape is intended to facilitate the osseointegration process due to its morphological resemblances.

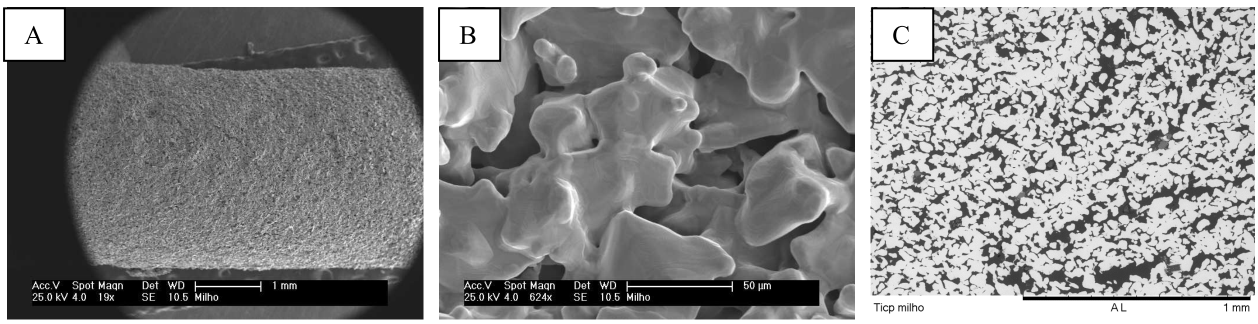


Figure 3. SEM of the samples added corn starch. A) Low magnification of the sample. B) Surface morphology. C) Microstructure of the sample.

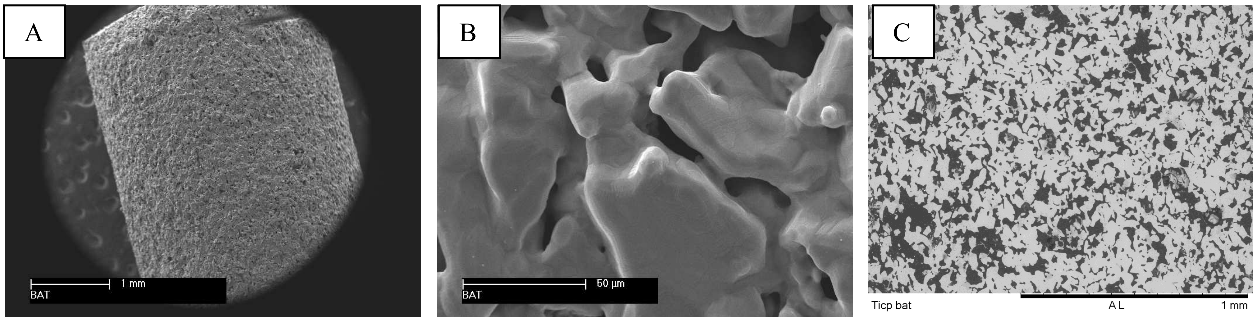


Figure 4. SEM of the samples added potato starch. A) Low magnification of the sample. B) Surface morphology. C) Microstructure of the sample.

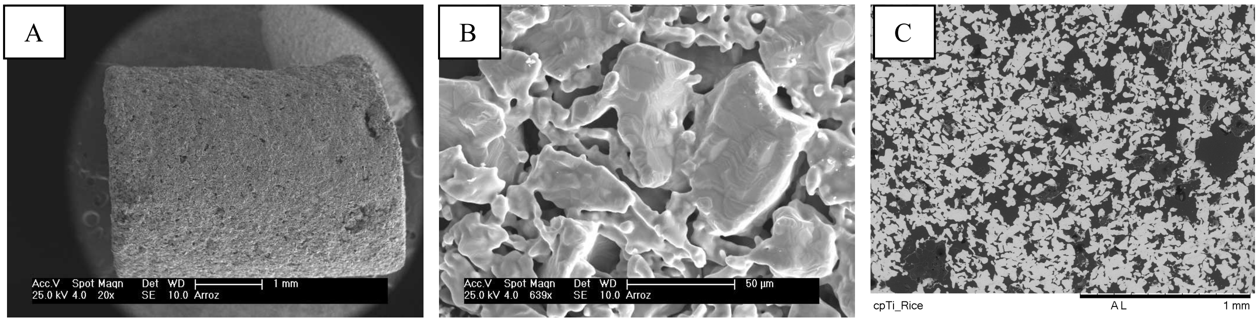


Figure 5. SEM of the samples added rice starch. A) Low magnification of the sample. B) Surface morphology. C) Microstructure of the sample.

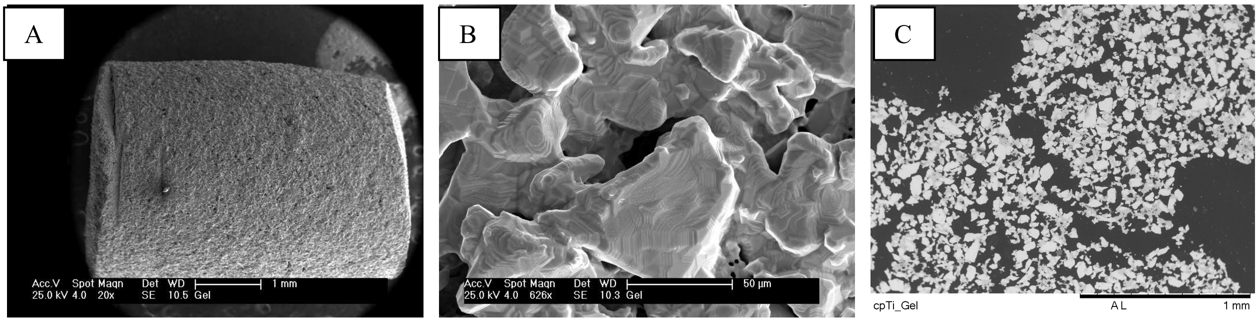


Figure 6. SEM of the samples added gelatin. A) Low magnification of the sample. B) Surface morphology. C) Microstructure of the sample.

Conclusions

The ability of gelling, easy degradation and wide source of these natural polymers, make them a reasonable, low cost and accessible choice, besides its greener feature of being an environment friendly additive.

The processing technique allowed the open pore formation, in which the pores mimics the trabecular bone structure and resemble to bone.

Although the different polymers provided a similar aspect of porosity, the varying pore size and microstructure distribution, will determine the meets and needs to application of these porous metallic biomaterials based on the specific polymer, that holds the proper implant intrinsic features to achieve repair and replacement success.

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