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Studying the Effect of Hydroxyapatite Coating on the Properties of Alumina

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Abstract. The expansion in the application of bio- materials leads to a wide variety of forms. The current study includes the preparation of a biological material in the form of a bio-coating that is represented by the, combination of a substrate with biological properties and at the same time high mechanical properties represented by alumina and the use of a coating from a material that provides high biological properties and low mechanical properties if it compared with the substrate, which is the hydroxyapeptite. The XRD and EDS technique showed an increase in the proportion of hydroxyapatite coatings formed and appears in calcium and phosphorous ions increasing with immersion time. A degradation test has been done after soaked the samples in Buffer solution for 7 days which proved an increment in degradation as the soaking time increase .

Introduction

Bio- materials are materials that have shown wide flexibility in the possibilities of using them in the repair, restoration and replacement of damaged and injured parts of the body of the living organism as well as showed wide progress when used in various medical applications and coatings[1]. This wide progress of applications and fields required the multiplicity of sources of obtaining these materials, whether natural or industrial, and the development of manufacturing methods to obtain abundant quantities and high purity. Biofilms are of a wide impact in the fields of applications of biological materials and differ according to the substrate, which can be ceramics, metals and their alloys, or polymers[2]. As well as the diversity of the coating material with the same types mentioned previously, or it may be a composite material, in addition to the methods and techniques of applying the paint, such as laser coating or wet or dry chemical deposition and others. Both the floor and the coating phase and the technique of applying the coating are chosen according to the specifications and requirements of the application, for example in orthopedics requires a porous floor that facilitates the penetration of vessels and nerves and the exchange of food, and at the same time the stiffness must be supported by a material that reduces the effect of brittleness resulting from the porosity in the ground phase[3]. While repair of teeth, for example, requires a solid material. Resist biting forces and also resist the effects of all oral fluids and fluids formed in the mouth and provide at the same time the appearance and aesthetics. All this has prompted the diversity of coating materials and techniques to provide all the required functions at the lowest cost and longest life while providing protection from side effects[4]. Controlling all the aforementioned is done through carrying out integrated checks to ensure obtaining the required properties in terms of physical and mechanical tests and most importantly, it must be done. There is a wide range of biological tests and toxicity tests such as immersion with body fluids or penetration of cells and other tests that prove the success of the coating as an effective part of the application[5].
Where previous studies related to the same research idea showed the use of bio-plating, a study showed that using titanium alloy floor and coating it with a metal material such as nickel alloy has a shorter life[6], if compared to a recent study in which the coating material is similar to ceramic alumina[7], where the results were better in terms of application age, lower cost and wear resistance. At the same time, a more recent study excelled, the flooring of alumina and the coating of zirconia showed high progress in all properties at the expense of biological and physical

properties, especially porosity, which is a basic requirement for most biological applications[8]. Regardless of the coating technique applied in most of the previously mentioned studies, but most of the previous research. The need for a variety of coatings and coatings has proven, such as the study that used a hydroxyapatite substrate coated with a bio-polymeric material[9].The current research represents a simple method for preparing a coating from ceramic biomaterials prepared from biological waste, and focuses on studying the properties of the resulting coating in terms of some biological tests to determine the success of the coating as a single integrated material that is easy to enter into areas of bio- applications.

Materials and Methods

1-Raw Materials

The alumina used as a material for the floor phase of the coating was purchased from the local market with a purity rate of approximately 98%. made in India.

Pure alumina powder and after examining it with X-ray technology to ensure the integrity of the installation, it was used to prepare models of a cylindrical diameter of 13 mm and 26 mm of height using a hydraulic press and pressure used up to 125 MPa and sinter the samples at a temperature of 1300C^o for a period of 3 hours and a rate of 5 C^o/min, and let them cool to be ready for the subsequent immersion process. The hydroxyapatite used as a coating material in this research was prepared from egg shells after collecting them in appropriate quantities, and chemically cleaning them with acetone solution and then leaving them to dry under the rays of the sun for two days. Then grind them dry milling balls(SFM-1, QM-3SP2) for 14 hours to turn into a very fine powder free of clumps followed by, a calcination process at 850°C for 2hours,this process helps egg shells powder to get rid of the organic materials, and transformation to the calcium-oxide. The resulting powder was mixed with phosphoric acid (as 1:1 weight ratio), to get homogenous mixing the mixtures were milled in the ball for 10 hours, sintering at a temperature of 1100 C^o for a period of three hours with a slow heating rate of about 7 C^o/min after mixing process has been done. Fig. 1 shows the stages of the process of obtaining hydroxyapatite.

2- Preparing the coating solution from hydroxyapatite powder

The used coating solution was prepared from 100 grams of distilled water, and the addition of 50 grams of hydroxyapatite prepared in the manner mentioned previously. The mixture was mixed with a ball mill using an alumina container and mixing balls made of zirconia with a wide size gradient and a mixing speed of up to 300 revolutions per minute for 6 hours.

3- The Process of Coating Samples

The cylindrical aluminum samples used as a substrate for the coating in the research were immersed in a pre-prepared hydroxyapatite solution for periods ranging from (10,30, and 50) minutes, and then dried at 100 ° C for 12 hours ,then sintered at at 1200 C^o for one hours, to prepare them for subsequent tests.

Characterization

Analysis of the microstructure that shows the purity of the raw materials and making sure of the composition of the prepared materials and analyzing the layers of coating applied to the alumina samples was done by the XRD method, by x-ray diffractometer. EDS applied to explain the types of ions appear in the substrate surface.

The biological activity of the samples was determined after immersing the coated samples with saturated body liquid solution for a period of 14 days, studying the surface nature and analyzing the layers formed on the surface for the models. Degradation test by calculated the weight loss in sample, after immersing in Buffer solution Tris-HCl, Ph is 7.4 for seven days.

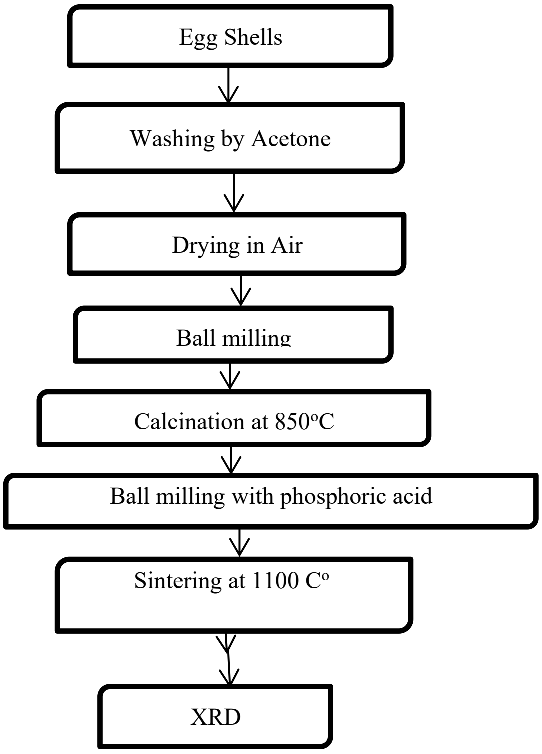

Figure 1 The stages of preparation of hydroxyapatite powder.

Results & Discussion

Fig. 2 and 3 show, the analysis by XRD which, appears the pure single phase of hydroxyapatite which prepared from egg shells, after matching the results with (JCPDS) card No. (09-0432), and XRD of commercial alumina shows pure single phase, accordance to (JCPDS) card No. (10-173)
Fig. 4(a,b,c), explains XRD which related to natural of the surface for the alumina substrate after soaking in a solution of hydroxyapatite for period(10,30,and 50) minutes, from this figure we can notice that, the peaks which related to the coating materials hydroxyapatite in figure (a) appeared, when the samples soaked for 10 to 30 minutes ,while this peaks and their intensity increase as the time of soaking increasing from 10 in figure(b) ,and we can see all the hydroxyapatite peaks with higher intensity as the time of soaking was 50 minutes in figure (c). It is proved that when the soaking time increase the coating layer and their thickness increases[10].

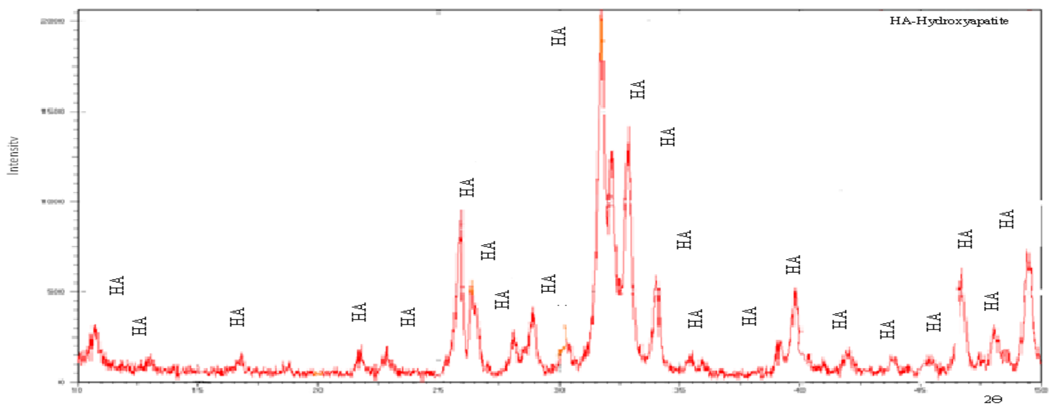

Figure 2 XRD analysis of hydroxyapatite.

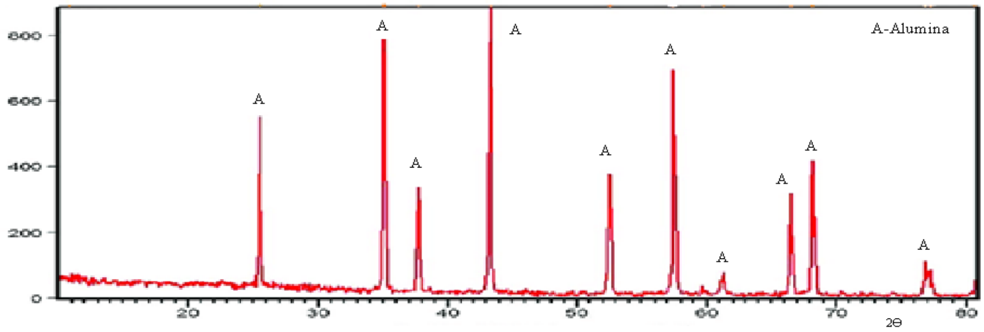

Figure 3 XRD investigation of alumina.

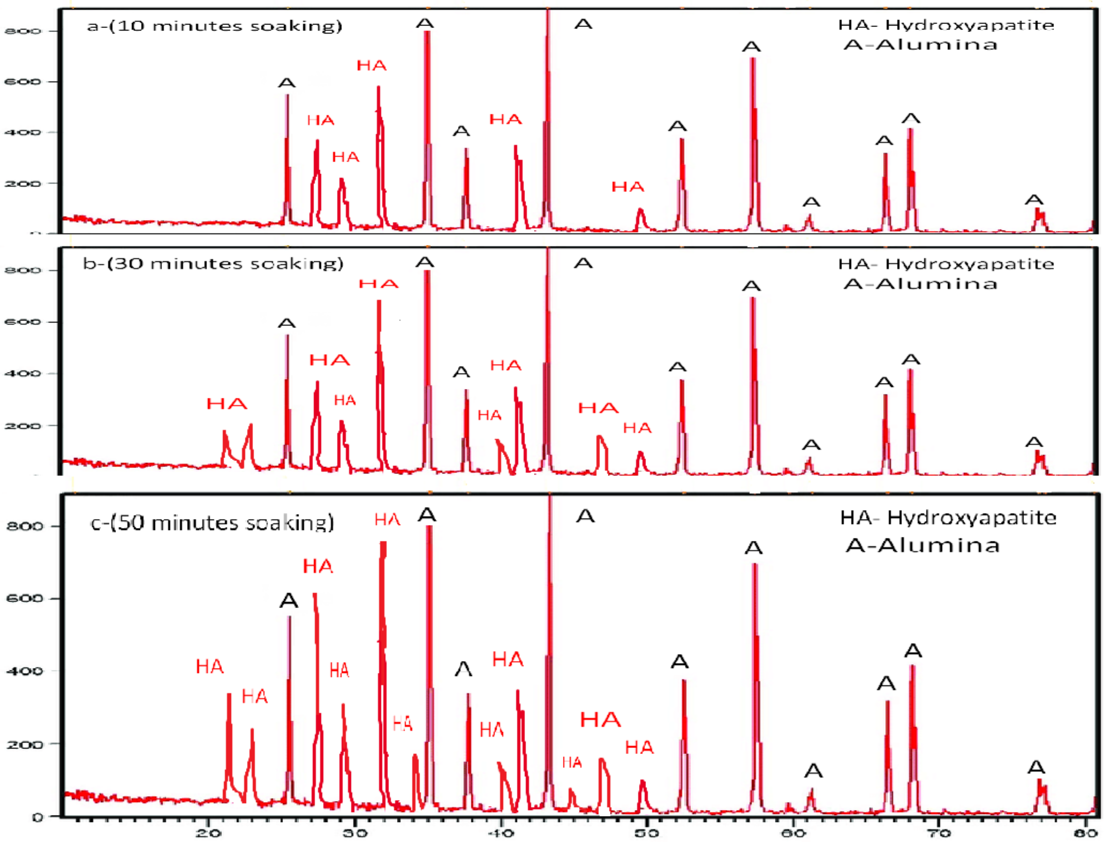

Figure 4 XRD examination of alumina surface after soaking in hydroxyapatite solution for: (a)10 minutes,(b)30minutes,(c)50 minutes.

Fig.5 (a,b,c) related to the EDX test for alumina samples, after a periods time of soaking. It will be interesting to know that Ca, P peaks appeared on the alumina surface after 10 minutes from soaking and this can show in figure(a),while in (b,c) figures the peaks of Ca, P noticed diffusion in a large areas for substrate with high intensity ,which explained the effect of increase soaking times on the increment of coating layers and their thickness[11] . Fig. 6 mentions, EDX test for coating samples after immersed in SBF solution for (14) days which showed in (a) peaks related to hydroxyapatite phase as (P,Ca) that proved formation of HA layers cover the samples surface ,and this peaks appearance and intensity increase for the sample soaking in the HA solution increase as shown in (b and c).Which gives high bioactivity of the prepared samples, and ability to perfect react with body solution[12].

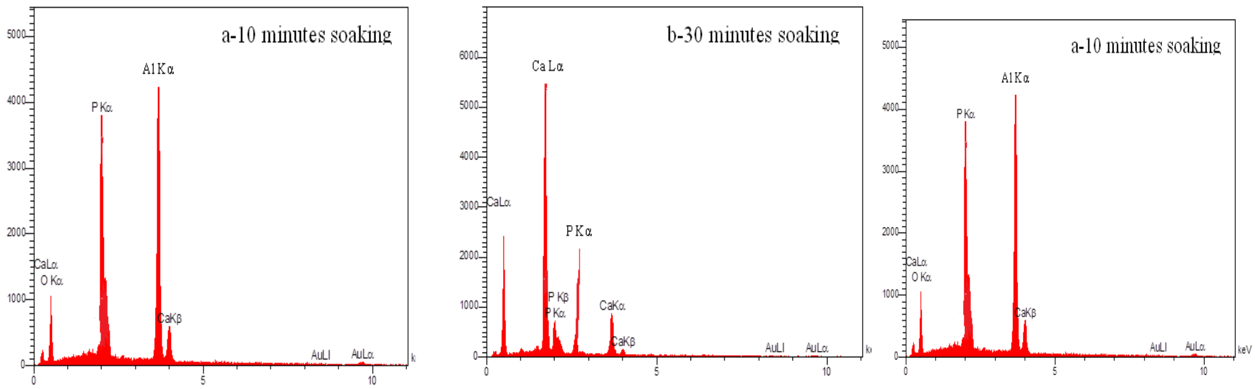

Figure 5 EDX of alumina surface after soaking in hydroxyapatite solution for: (a)10 minutes,(b)30minutes,(c)50 minutes.

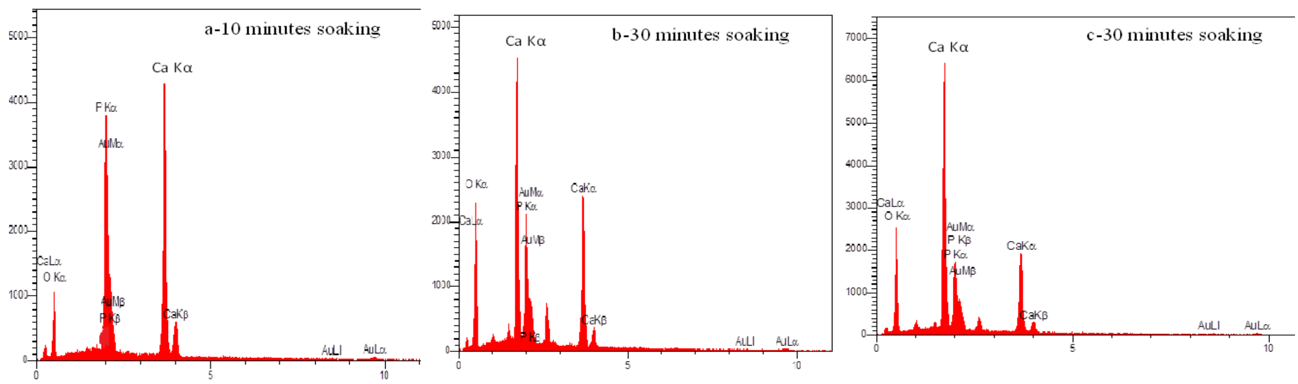

Figure 6 EDX analysis for coated samples ((a)10 minutes,(b)30minutes,(c)50 minutes) after 14 days soaking in SBF solution.

Fig. 7 describes the relative between the ion concentration for the ions covered the surface of the alumina coated samples after soaking for fourteen days in simulated body fluid ,and the samples soaking days in hydroxyapatite solution for coating. The figure displays the appeared and addition in the ions like, Ca^{2+}, $PO_4{}^{3-}$. which pointed to formation of hydroxyapatite layers on the coated alumina samples which stayed for a long time in HA solution .Also it shows that Ca/P ratio increased as the soaking time increase which give 1.4 for sample before immersing and (1.6,1.7,1.78) respectively for each soaking time. The above result leads to, perfect reacted with body fluid and important bioactive property for prepared coating[13].

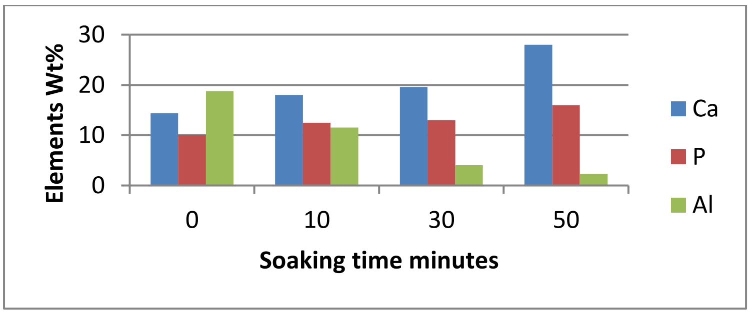

Figure 7 Difference in ion weight formation on the alumina coating ,sample for different period of time after 21 days immersing in SBF.

Fig. 8 presents one of the most important biological test, that give the amount in weight loss for alumina samples after ten to fifty minutes of immersing in HA solution. From this test we can see that the loss in weight increase when immersing time increase, as a result of formation of hydroxyapatite layer ,and this material has high degradation which means achievement one of a good biological properties[14].

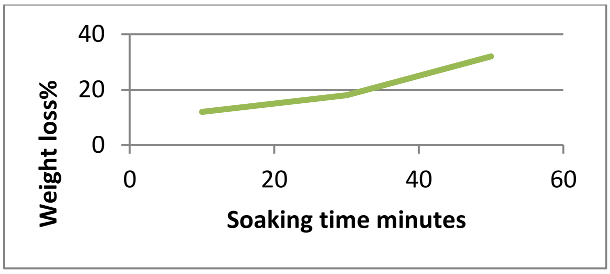

Figure 8 Weight loss after soaking the coated, sample in Buffer solution for seven days.

Summery

Biological behavior tests for alumina substrate immersed in hydroxyapatite solution for (10,30,50) minutes, explain perfect properties when coating samples immersed in SBF solution for 14 days. The test results proved by XRD, and EDX which showed formation of HA layers on the sample surface, and appeared of Ca,P ions and this increased with increment in Ca/P ratio , when soaking time in coating solution increase. Also the coating samples have long soaking time in HA solution, gave good degradation, after soaked in buffer solution for seven days .

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