

# In Vitro Study for Yttrium addition on titanium - 15 Molybdenum after Immersion in Simulated Body Fluid

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## Abstract

Multiple attempts are made to develop bioactive alloy for the construction of the implant to enhance osseointegration and support implant functions. The titanium – 15 Molybdenum alloy with and without the addition of Yttrium makes an organic-inorganic hybrid composite that has the ability to make a network with or without covalent bonds between components. In this study the immersion of Titanium – 15 Molybdenum disc and titanium – 15 Molybdenum- 1.5 Yttrium in simulated body fluid.

The unique biomaterial multi-component Ti-Mo-1.5Y alloys were created as a way to improve the bioactivity characteristics and lessen the toxicity of biomaterials for implants in order to create new biomaterials for implant applications. The purpose of this study is to look at how adding yttrium (Y) changes the alloy's bioactivity. Through the use of powder metallurgy, Ti-Mo-1.5Y alloys are created. Atmospheric pressure high-purity argon gas is used to sinter the metals. samples were immersed in SBF in closed containers and placed in incubators at a temperature of 370 C for 30 days. Analysis of the surface includes thickness measurement, X-ray diffraction (XRD), and an optical microscope. The results of showed an increase in the thickness of (HA) with the addition of yttrium after immersion in SBF compared with thickness before the addition of yttrium. The results of XRD showed the appearance of new peaks of HA, and showed there are additional accumulates of HA granules when adding yttrium content after immersion in SBF.

**Keywords:**  $\beta$ -Titanium · Yttrium · Powder metallurgy · Bioactivity · SBF.

## INTRODUCTION

The success of dental implants depends on many factors; implant material, design of the implant, and design of implant surface (1). Multiple attempts are made to develop coating layers on the implant surface to enhance osseointegration and support implant functions(2). The coating layer should be biocompatible and have osteoconductive properties which means that it must have the ability to attract osteoblasts to the implant surface and enhance bone formation to secure the bone-implant interface(3). The wide use of Titanium and its alloys as dental implants due to its biocompatibility, good strength, resistance to corrosion, and ability to enhance osseointegration(4). Much research has been done to modify titanium implant surfaces by coating them with materials that enhance stabilization and osseointegration or by adding an alloying element (5). Hydroxy apatite is commonly used as a coating for titanium implants; since it is a biomaterial that resembles natural teeth and bones(6).

Yttrium element is one of the representative members of rare earth elements (REEs). The application of yttrium and its compounds had been reported previously to involve many aspects of industrial photocatalysis, photoluminescence properties, biological corrosion retardation, hemocompatibility, and re-endothelialization [7–9]. In order to improve the photocatalytic performance of TiO<sub>2</sub>, REE doping had long been known as one of the most effective approaches to promote photocatalytic performance. Previous studies had reported that the prepared low-cost Y-doped TiO<sub>2</sub> nanosheet film enhanced photocatalytic removal of Cr and methyl orange in cathode ray tube waste [7]. Another investigation focused on the synthesis of Y-TiO<sub>2</sub> nanotubes, their photoluminescence, and photocatalytic properties, indicating that the photocatalytic properties increased with the increase of Y-doping content [8].

Also because of its biocompatibility, which enhances the growth of bone when implanted inside the body, and its acceptable mechanical properties so it is widely used as an alloying element with titanium alloy (10)

Numerous experiments are being conducted to create Ti alloys containing yttrium. Ti alloys containing yttrium are biocompatible [11]. Recent studies have focused on -type alloys that minimize the stress-shielding effect by having low elastic moduli. These alloys are created using stabilizers and non-cytotoxic materials such as molybdenum, tantalum, niobium, zirconium, yttrium, and manganese indium [12,11-17]. Modern alloys have an elasticity modulus that is three to four times greater than that of human bone, which can be uncomfortable for the patient and increase the risk of implant failure. This is due to a condition called stress shielding [18], in which the implant's heavy mechanical strain causes degradation and decreased bone density, leaving the bones exposed. Dental implants' therapeutic effectiveness is improved by the addition of yttrium to Ti due to improvements in biocompatibility, mechanical characteristics, and corrosion resistance [19].

Many attempts have been made to develop an alloy that establishes the ability to form bone-like apatite in simulated body fluid (SBF)(20). A cellular (SBF) that has ions concentrations almost similar to that of human plasma, it has the ability to formulate bone apatite on the surface of the bioactive materials, therefore it is used for in vitro estimation of the bioactivity of the materials and evaluation of the formation of bone-like apatite on the implant surface(21). The procedure of measuring the HA formation on the coated surface is considered an in vitro procedure that has many advantages over the conventional in vivo procedure in that is the simplest, cheaper, quicker procedure, reproducible method, and less affected by experimental conditions(22). The study aimed to evaluate the formation of new hydroxyapatite after adding yttrium alloy to the basic alloy samples in SBF.

## Experimental Procedure

In this work, titanium, molybdenum, and yttrium powders were employed; they were bought from Lemandou Ltd. Co. China. As determined by X-ray fluorescence (XRF) type (Bruker S8 Tiger) and particle size analyzer type (NanoBrook 90Plus) for the beginning materials' particle size, the purity of the employed powders is displayed below in Table 1.

Table (1): Average size and Purity of the Powders

Powder	Purity %	Average particles size
Ti	99.95	28.62
Mo	99.98	23.59
Y	99.99	19.43

### Preparation of Sample

Powder metallurgy was used to create the Ti-15Mo-1.5Y in an inert atmosphere (Argon). According to Table 2, two samples were created, each of which had two samples of Titanium-15Molybdenum-1.5Yttrium, the proportion of powders in each sample, and its composites.

Table 2: Chemical composition of the prepared alloys (wt%).

Specimen	Mg	Al	Si	Ca	Ti	Mn	Fe	O	Mo	Y
85Ti-15Mo	0.08	0.09	0.32	0.24	84.40	0.07	0.11	0.35	14.35	0.00
83.5Ti-15Mo1.5Y	0.09	0.10	0.04	0.30	82.93	0.05	0.18	0.23	14.68	1.37

Ti, Mo, with and without Y, and their respective elemental metal powders were blended in a planetary ball milling machine for 8 hours at a rotational speed of 300 rpm and a ball-to-powder weight ratio of 20:1. According to the speed, type, and milling

operation, the temperature would typically increase during the milling process. To reduce heating, the mill was run in cycles of 60 minutes of grinding and 30 minutes of idle time. After eight hours of grinding, the powder mixture was cold compressed under 850 MPa for three minutes with a constant loading rate (0.3 t/s) to produce a green compact, whose size varied depending on the test. Green compacts were sintered at 950 °C at a rate of 5 °C/min for six hours, after which they were cooled in the vacuum furnace.[23]

The following steps are included in the sintering process samples: ( Figure 1 )

1. heating at a rate of 5 C°/min, up to 500 C, starting at room temperature
2. one hour of soaking at 500 C.
3. Heating increases at a rate of 5 C°/min from 500 C to 950 C.
4. six hours of soaking at 950 C.
5. In a furnace under constant argon conditions, the temperature is gradually lowered to room temperature.

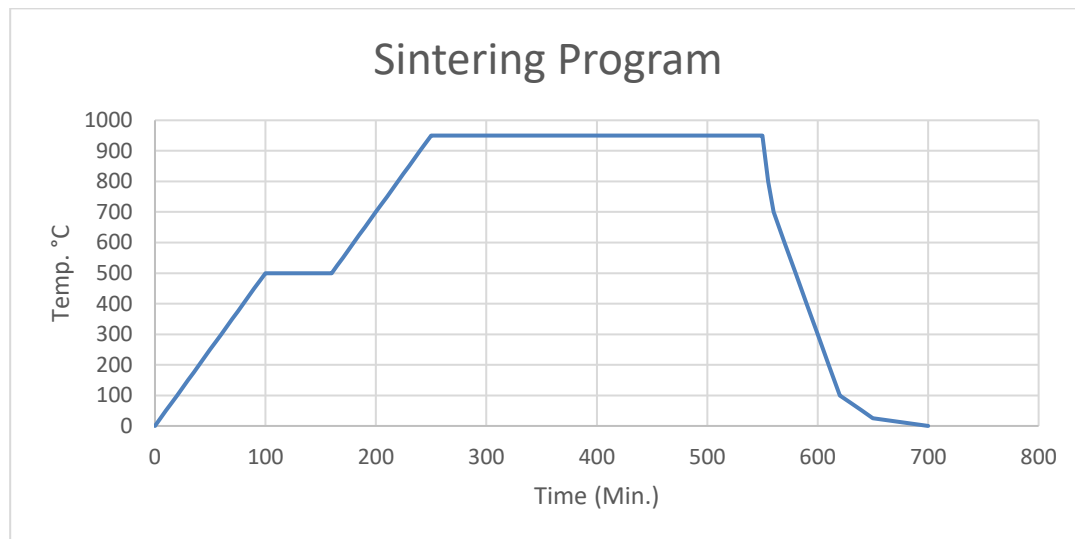


Figure 1: Heating Cycle in Sintering

#### Characterization of the Prepared Specimens

X-ray diffraction (XRD) was used to determine the phases and crystal structures of the produced specimen (Shimadzu XRD-60000 X-Ray diffractometer). It used Cu K radiation at 40 kV, 30 mA, and 2° of 20°–80° wavelength (=1.5409 Å).

A light optical microscope (Olympus/542037.Japan) and a scanning electron microscope (CARL ZEISS ULTRA PLUS GEMINI FESEM) was used for the microstructure analysis. Wet grinding with grades of (600, 800, 1000, and 2000) and polishing with diamond paste (1 μm particle size) were completed for the specimens. With "10 g FeCl<sub>3</sub>+ 25 ml HCL+ 100 ml distilled water" [24], the specimens were etched. X-ray diffraction (XRD) calculations were done on the specimen's phases and crystal structures both before and after the addition of the yttrium (Shimadzu XRD-60000 X-Ray diffractometer). It was employing CuK radiation at 40 kV, 30 mA, and 2° of 20°–80° wavelength (=1.5409 Å).

## Result and Discussion:

### X-ray diffraction analysis:

As appeared in fig. 2 a and b compared different alloy samples after immersion in SBF for 30 days which illustrates the appearance of new peaks of HA. Peaks of HA were indexed according to the standard pattern (JCPDS09-0432).

Small amounts of yttrium addition have been extensively investigated for their grain refinement efficiency in Ti and its alloys. In general, for commercial purity Ti, trace amounts of yttrium have provided significant grain refinement in both the prior  $\alpha$ - and  $\beta$ -grain sizes due to its strong segregating ability. (25)

Yttrium is an element that the body can metabolize, and the addition of Y can refine the grain and improve the mechanical properties of titanium alloys. Enhancement of osseointegration by improvement of bioactivity and biocompatibility of the titanium molybdenum alloy by addition of yttrium element. It is widely believed that the adhesion and spreading of osteoblasts on the surface of implants is the first step in osseointegration. Hence, the biocompatibility of implant materials is critical for successful implantation. from the result above the yttrium stimulate to formation more of HA layers.

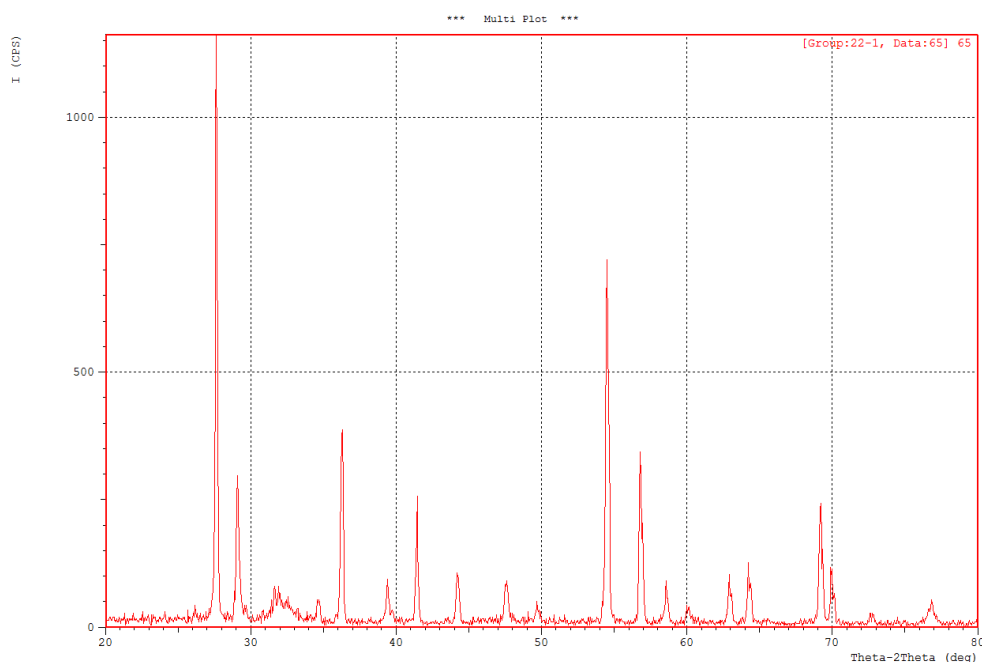


Figure 2A: X-ray diffraction of tested samples

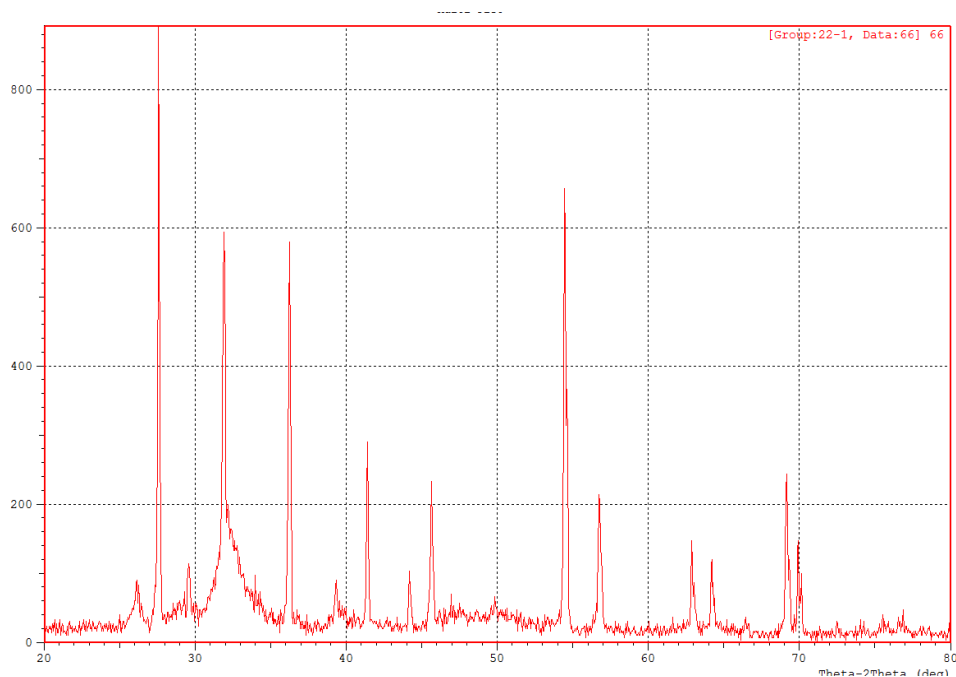
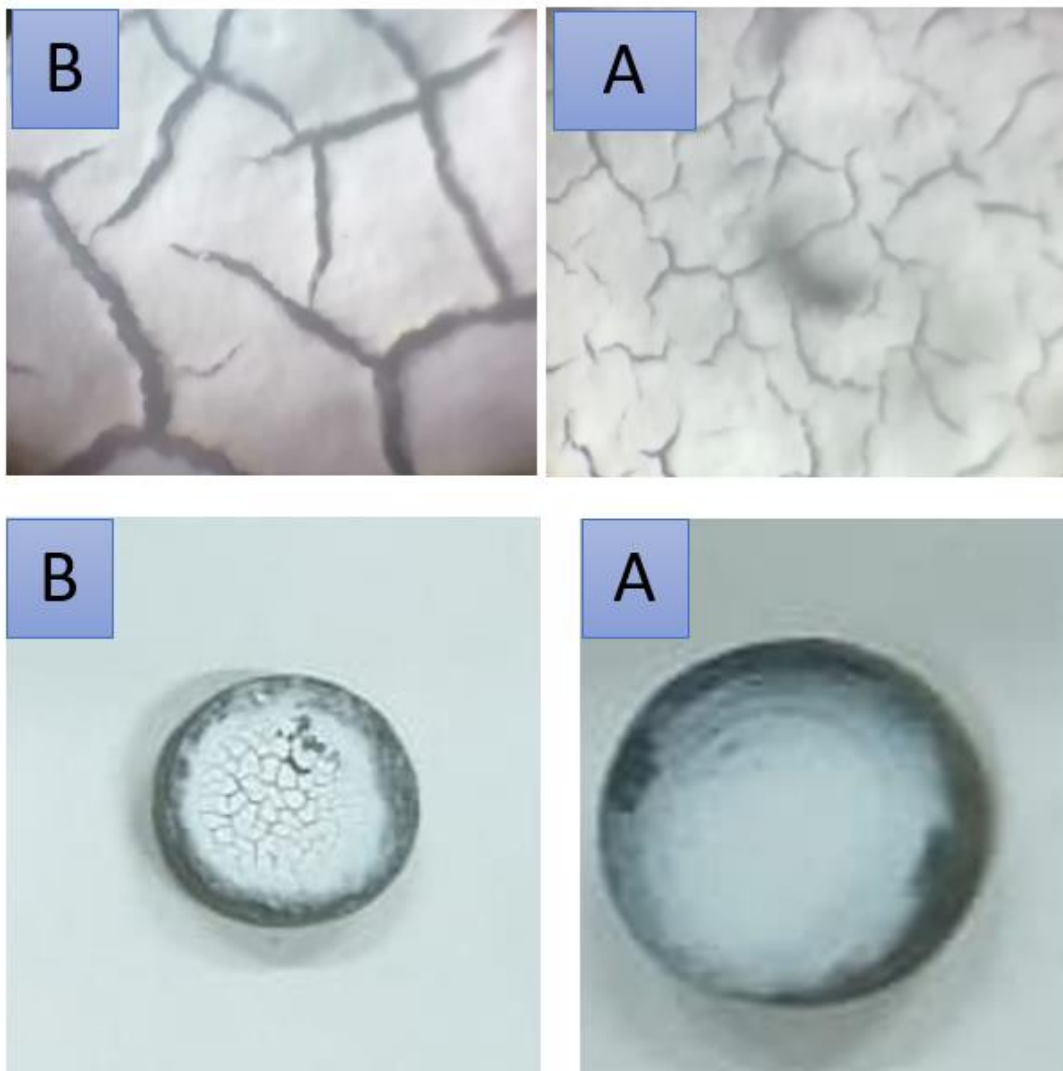


Figure 2B: X-ray diffraction of tested samples

#### Optical Light microscope (SEM):

The surface feature of base alloy samples after 30 days immersed in SBF was compared with the surface feature of alloy with yttrium addition samples after immersion in SBF. As appeared in fig. 3 a and b there is an additional accumulation of HA granules on the surface of the alloy with yttrium addition (3b). The newly formed crystals of HA accumulated as layers of flaks which differed from the manner of accumulation after immersion in SBF for the base alloy without the addition of yttrium (3a), these differences in the manner of accumulation were due to the differences in the condition, PH, and temperature of HA formation. Also, the sufficient time of immersion in SBF led to complete coverage of titanium samples with layers of HA even cracks areas, and the appearance of a random accumulation of layers because of unrestricted formation and growth of HA crystals (26). This manner of nucleation and formation of new apatite is an indication of the bioactivity of the Ti- 15 Mo- 1.5Y sample(27). It has been reported that the proliferation, adhesion, mineralization, and changes in gene expression in osteoblasts are very sensitive to micro/nanoscale interfaces. Previous studies have shown that the adhesive capacity of osteoblasts on Y-doped HA increased with increasing molar percentage (mol %) of yttrium (28-29). From above, the results of the previous study agreed with our that the addition of the yttrium increased bone-implant contact.



## REFERENCES

1. Fawad J, Hameeda B, Roberto C, Georgios E. Romanos4: Role of primary stability for successful osseointegration of dental implants: Factors of influence and evaluation, *Interventional Medicine & Applied Science*. 2013; 5 (4): 162–167.
2. Bobyn J, Thompson R, Lim L. Local alendronic acid elution increases net periimplant bone formation: Amicro-CT analysis. *Clin. Orthop. Relat. Res.* 2014; 472: 687–694.
3. Albrektsson T, Johansson C. Osteoinduction, osteoconduction and osseointegration. *Eur. Spine J.* 2001; 10: S96–S101.
4. Yang Y, Kim KH, Ong JL. A review on calcium phosphate coatings produced using sputtering process—An alternative to plasma spraying. *Biomaterials*. 2005;26: 327–337.
5. Yang Y, Oh N, Lui Y. Enhancement of osseointegration using surface modified titanium implant. *JOM*. 2006;58: 71–76.
6. Chou BY, Chang E. Influence of deposition temperature on mechanical properties of plasmasprayed hydroxyapatite coating on titanium alloy with ZrO<sub>2</sub> intermediate layer. *J Therm Spray Technol* 2003; 12: 199–207.
7. [21] Q. Zhang, Y. Fu, Y. Wu, Y. Zhang, T. Zuo, Low-cost Y-doped TiO<sub>2</sub> nanosheets film with highly reactive {001} facets from CRT waste and enhanced photocatalytic removal of Cr(VI) and methyl Orange. *ACS Sustain. Chem. Eng.* 4 (2016).
8. [22] J. Rao, H. Xue, W. Zhang, X. Li, X. You, Z. Xing, Synthesis of yttrium doped TiO<sub>2</sub> nanotubes by a microwave refluxing method and their photoluminescence properties and photocatalytic properties, *Int. J. Electrochem. Sci.* 11 (2016) 2408–2418.
9. [23] P. Wang, P. Xiong, J. Liu, S. Gao, T. Xi, Y. Cheng, A silk-based coating containing GREDEVY peptide and heparin on Mg-Zn-Y-Nd alloy: improved corrosion resistance, hemocompatibility and endothelialization, *J. Mater. Chem. B* 6 (2018).
10. Gunawarman, Mulyadi I, Arif Z, Nuswantoro N, Af J, Niinomi M (2021) Effect of particle size on adhesion strength of bovine hydroxyapatite layer on Ti-12Cr coated by using electrophoretic deposition (EPD) method. In: *IOP conference series: materials science and engineering*, vol 104, p 012054
11. Fajri H, Ramadhan F, Nuswantoro NF, Juliadmi D, Tjong DH, Manjas M, Af J, Yetri Y, Gunawarman (2020) Electrophoretic deposition (EPD) of natural hydroxyapatite coatings on titanium Ti-29Nb-13Ta-4.6Zr substrates for implant material. *Mater Sci Forum* 1000:123–131. <https://doi.org/10.4028/www.scientific.net/msf.1000.123>.

12. Sudhagara Rajan S, Jithin V, Geetha M, Nageswara Rao M (2020) Heat treatment of metastable beta titanium alloys, welding - modern topics, Sadek Crisóstomo Absi Alfaro, Wojciech Borek, and Błażej Tomiczek, IntechOpen. Available from: <https://www.intechopen.com/chapters/72078>
13. Wang ZM, Ma YT, Zhang J, et al. (2008) Influence of yttrium as a minority alloying element on the corrosion behavior in Fe-based bulk metallic glasses. *Electrochim Acta* 54: 261–269.
14. Xu Z, Wang Y, Xu R, Hu Q, Shi D, Lu X (2020) Research on microstructure and properties of Ti-15Mo-3Al alloy with high oxygen content. *Mater Res Express* 7(11):116528
15. Bahador A, Hamzah E, Kondoh K, Abubakar TA, Yusof F, Umeda J, Ibrahim MK (2018) Microstructure and superelastic properties of free forged Ti-Ni shape-memory alloy. *Trans Nonferrous Met Soc China* 28(3):502–514
16. Han MK, Im JB, Hwang MJ, Kim BJ, Kim HY, Park YJ (2015) effect of indium content on the microstructure, mechanical properties, and corrosion behavior of titanium alloys. *Metals* 5(2):850–862
17. Ho W-F, Wu S-C, Hsu S-K, Li Y-C, Hsu H-C (2012) Effects of molybdenum content on the structure and mechanical properties of as-cast Ti–10Zr-based alloys for biomedical applications. *Mater Sci Eng C* 32:517–522.
18. Hansen DC (2008) Metal corrosion in the human body: the ultimate bio-corrosion scenario. *Electrochem Soc Interface* 17(2):31.
19. Ammar Hassan Khilfa, (2015) ‘Effect of Y and Ge addition on Mechanical Properties and Corrosion Behavior of Biomedical CoCrMo Alloy (F75)’, MSC thesis, Submitted to the Council of the College of Materials Engineering / University of Babylon.
20. SH Rhee, YK Lee, BS Lim. Evaluation of chitosan nano – hybrid material containing silanol group and calcium salt as a bioactive bone graft *Key Eng. Mater.* 2005; 284–286, 765. 16.
21. Kong L J, Gao Y, Lu G Y, et al. A study on the bioactivity of chitosan/nano-hydroxyapatite composite scaffolds for bone tissue engineering. *European Polymer Journal*, 2006; 42(12): 3171– 3179. 17.
22. Lee J, Leng Y, Choe, K, Ren F.; Ge X. Cell culture medium as an alternative to conventional semmulated body fluid, *Acta Biomaterialia*, 2011; 7:2615 – 22.
23. P. Xu, X. Tang, S. Yao, J. He, G. Xu, J. Mater. Process. Technol. 208 (2008) 549–555.
24. V. de Castro, T. Leguey, A. Muñoz, M.A. Monge, R. Pareja, *Mater. Sci. Eng., A* 422 (2006) 189–197.
25. Kailiang Zhang (S.M.M), Baoping Zhang (M.D), Chunjuan Huang (S.M.M), Shuting Gao (S.M.M), Bo Li (S.M.M), Rui Cao (S.M.M), Jingyang Cheng (S.M.M), Ruiping Li (S.M.M), Zhanhai Yu (M.D), Xiaodong Xie (M.D), (2019) Biocompatibility and antibacterial properties of pure titanium surfaces coated with yttrium-doped hydroxyapatite *Journal of the Mechanical Behavior of Biomedical Materials* 100 , 103363.
26. Lu X, Leng Y. Theoretical analysis of calcium phosphate precipitation in simulated body fluid. *Biomaterials* 2005;26:1097–108.
27. Kaneko H, Uchida M, Kim HM, Kokubo T, Nakamura T. Process of apatite formation induced by anatase on titanium metal in simulated body fluid. *Key Eng Mater* 2002; 218–220:649–52.
28. R. Olivares-Navarrete, P. Raz, G. Zhao, J. Chen, M. Wieland, D.L. Cochran, R.A. Chaudhri, A. Ormoy, B.D. Boyan, Z. Schwartz, Integrin alpha2beta1 plays a critical role in osteoblast response to micron-scale surface structure and surface energy of titanium substrates, *Proc. Natl. Acad. Sci. U. S. A.* 105 (41) (2008) 15767–15772.
29. T.J. Webster, C. Ergun, R.H. Doremus, R. Bizios, Hydroxylapatite with substituted magnesium, zinc, cadmium, and yttrium. II. Mechanisms of osteoblast adhesion, *J Biomed Mater Res B Appl Biomater* 59 (2) (2002) 312–317.