RINGKASAN

Sintesis Titanium-Hidroksiapatit : Suatu Upaya Peningkatan Kualitas Hidroksiapatit Sebagai Tulang Sintetis.

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Dua dasa warsa terakhir ini banyak dikembangkan material Hidroksiapatit (HA) dengan rumus kimia $Ca_{10}(PO_4)_6(OH)_2$ sebagai tulang sintetis. Di samping sifat-sifat yang menonjol dari Hidroksiapatit adalah berpori, terserap ulang (resorpsi), bioaktif, tidak korosi, inert dan tahan aus, ada sifat-sifat yang kurang baik pada Hidroksiapatit adalah getas, mudah patah, ini menjadi kendala dalam desain.

Upaya untuk itu telah dilakukan penelitian sintesis Ti-HA dengan mensubstitusi titanium pada Hidroksiapatit dengan cara basah/ larutan di Laboratorium. Penelitian ini bertujuan untuk meningkatkan sifat mekanik ditinjau dari aspek porositas, densitas, daya serap air, kuat tekan, kekerasan, kuat tarik dan modulus elastisitas.

Pada penelitian ini dilakukan sintesis Hidroksiapatit yaitu mereaksikan suspensi 0,5M $Ca(OH)_2$ dan 0,3M H_3PO_4 (M_0). Sintesis Ti-HA dilakukan dengan mereaksikan 0,3M H_3PO_4 dengan konsentrasi tetap yang telah dicampur dengan larutan $TiCl_3$ dengan variasi ; 0,490M $Ca(OH)_2$ dan 0,010M $TiCl_3$ (M_1); 0,485M $Ca(OH)_2$ dan 0,015M $TiCl_3$ (M_2) ; 0,480M $Ca(OH)_2$ dan 0,020M $TiCl_3$ (M_3) ; 0,475M $Ca(OH)_2$ dan 0,025M $TiCl_3$ (M_4).

Reaksi dilakukan dalam reaktor dengan pengadukan terus menerus dan dimonitor pH, saat reaksi suhu dipertahankan 40-50°C. Reaksi dilakukan selama $2^1/_2$ jam dan dilakukan replikasi 4 kali. Hasil dicuci dengan aquades supaya bebas khlor, disaring kemudian dipanasi pada suhu 110°C. Hasilnya dihaluskan berupa serbuk dan dicetak dalam bentuk kubus untuk uji kuat tekan, silinder tipis untuk uji kekerasan dan sifat fisika, bentuk balok untuk uji degradasi. Dari kelima senyawa hasil sintesis M_0, M_1, M_2, M_3, M_4 diidentifikasi komposisi kimianya dengan sinar-x.

Pada senyawa hasil sintesis Titanium-Hidroksiapatit substitusi 0,025M $TiCl_3$ (M_4) dilakukan analisis DTA dan TGA untuk menentukan suhu pembakaran yaitu 800 °C, 1000 °C, 1100 °C, 1200 °C.

Hasil pembakaran, pada Hidroksiapatit suhu 1200 °C, dan hasil sintesis Titanium-Hidroksiapatit suhu (M_4) 1000 °C, 1100 °C, 1200 °C diidentifikasi komposisi kimianya dengan sinar-x. Hasil komposisi kimia dari Hidroksiapatit adalah $Ca_5(PO_4)_3OH$; $Ca_3(PO_4)_2.xH_2O$; $Ca_3(PO_4)_2$., sedang hasil komposisi kimia dari sintesis Titanium-Hidroksiapatit (M_4) adalah $Ca_5(PO_4)_3OH$; Ca $Ti(PO_4)_6$; $Ca_3(PO_4)_2$ .xH_2O ; $Ca_3(PO_4)_2$.

Kemudian dilakukan pengujian sifat mekanik yang meliputi porositas, densitas, daya serap air, kuat tekan, kekerasan, kuat tarik dan modulus elastisitas. Pengujian didapat makin tinggi suhu pembakaran dan makin tinggi substitusi titanium pada HA makin kecil porositasnya. Harga densitas makin besar dengan makin tingginya suhu pembakaran dan makin tingginya substitusi titanium pada HA.

Harga daya serap air makin kecil dengan makin tingginya suhu pembakaran dan makin tingginya substitusi titanium pada HA. Harga daya serap Ti-HA (M_4) pada suhu 1200°C adalah 0,49% lebih kecil dibanding dengan Hidroksiapatit (M_0) adalah 3,28%.

Harga kuat tekan makin tinggi dengan makin tingginya suhu pembakaran dan makin tingginya substitusi titanium pada HA. Harga kuat tekan pada Ti-HA (M_4) suhu 1200°C adalah 36,268 MPa dan lebih tinggi dibanding dengan Hidroksiapatit (M_0) 24,162 MPa . Harga kekerasan makin tinggi dengan makin tingginya suhu pembakaran dan makin tinggi substitusi titanium pada HA. Harga kekerasan pada Ti-HA (M_4) pada suhu 1200°C adalah 18,093 MPa dan pada HA (M_0) adalah 11,878 MPa. Harga kuat tarik makin tinggi dengan makin tingginya suhu pembakaran dan makin tingginya substitusi titanium, demikian pula harga modulus elastisitas makin naik dengan makin naiknya suhu pembakaran dan makin tinggi substitusi titanium pada HA. Harga kuat tarik pada Ti-HA (M_4) pada suhu 1200°C adalah 4,690 MPa dan pada HA (M_0) adalah 2,542 MPa. Adapun harga Modulis Elastisitas pada Ti-HA (M_4) pada suhu 1200°C adalah 53,295 MPa dan pada HA (M_0) adalah 30,622 MPa

Pengujian degradasi dilakukan dengan merendam senyawa hasil dengan larutan SBF/ *Syntetic Body Fluid* selama 12 minggu, hasil bentuk fisik HA maupun Ti-HA tetap seperti semula, tidak terdekomposisi dan tidak terdegradasi.

Pengujian biokompatibilitas dilakukan dengan pengujian toksik dan penumbuhan sel *fibroblast* (*cell line BHK 21 clone 13*) secara in vitro, dengan hasil pengujian HA(M_0) maupun Ti-HA (M_4) pada suhu 1200 °C tidak toksik dan dapat ditumbuhi sel, pertumbuhan sel yang terbanyak adalah Ti-HA (M_4) pada suhu 1000 °C.

Hasil sintesis substitusi titanium pada Hidroksiapatit pada penelitian ini adalah meningkatkan sifat mekanik meliputi porositas, densitas, daya serap air dan kuat tekan, kekerasan serta memenuhi persyaratan sebagai biomaterial tulang sintetis. Adapun senyawa Ti-HA yang diperoleh M_1, M_2, M_3, M_4 dapat digunakan sebagai tulang sintetis alternatif, yang dalam aplikasinya harus disesuaikan dengan peruntukan dan tempat implannya perlu dikaji lebih lanjut.

SUMMARY

Titanium- Hydroxyapatite Synthesis : The Effort to Improve Quality of the Hydroxyapatite As Synthetic bone

Wahidin Nuriana

During thus last decade Hydroxyapatite compound (HA) wich chemical formula $Ca_{10}(PO_4)_6(OH)_2$ as syntetic bone.

Hydroxyapatite compound has some prominent properties, porous, resorption, bioactive, non-corrosive, inert and wear resistance. On the other hand there are some unforable properties, brittle, tend to break, that is become constraint in design.

The effort had been research conducted Titanium- Hydroxiapatite synthesis with substitution titanium of hydroxiapatite by solution method in Laboratory. Purpose of the research for improving physics and mechanical properties that including compressive strength, hardness, tensile strength and modulus elasticity of Hydroxyapatite.

On this research conducted Hydroxyapatite synthesis that is by reacting suspension of $Ca(OH)_2$ by concentration 0,5M and 0,3 M solution of H_3PO_4 (M_0).

Titanium- Hydroxiapatite (Ti-HA) compound synthesis conducted by reacting solution of H_3PO_4 by concentration 0,3M into the mixture various solution concentration of $TiCl_3$; $Ca(OH)_2$ 0,490M and $TiCl_3$ 0,010M (M_1); 0,48 $Ca(OH)_2$ 0,485M and $TiCl_3$ 0,015M (M_2) ; $Ca(OH)_2$ 0,480M and $TiCl_3$ 0,020M (M_3) ; $Ca(OH)_2$ 0,475M and $TiCl_3$ 0,025M (M_4).

Reaction conducted in the reactor, conducted mixing continuously and monitored early pH, keep stabile temperature of 40-50°C during reacting. Reacting conducted 21/2 hours in batch and replication conducted fifth time. Yield washed with aquadest in order to release chlor, filtered, dryed in temperature of 110 °C. Yield refined as powder and molding as in thin cylinder for compressive strength, tensile strength test, bar shape for degradation test and cube for hardness.

Five product synthesis of compound M_0, M_1, M_2, M_3, M_4, identified of chemical composition by x-ray. The product Titanium- Hydroxiapatite compound synthesis of substitution $TiCl_3$ 0,025M (M_4) conducted DTA and TGA analysis for determine of burner temperature is 800 °C, 1000 °C, 1100 °C, 1200 °C.

Combustion yield of Hydroxiapatite temperature of 1200 °C (M_4) temperature of 1000 °C, 1100 °C, 1200 °C identified of chemical compocition by x-ray. The chemical composition of Hydroxiapatite is $Ca_5(PO_4)_3OH$; $Ca_3(PO_4)_2.xH_2O$; $Ca_3(PO_4)_2$. The chemical composition of Titanium-Hydroxiapatite (M_4) is $Ca_5(PO_4)_3OH$; $Ca\ Ti(PO_4)_6$; $Ca_3(PO_4)_2\ .xH_2O$; $Ca_3(PO_4)_2$.

The test smaller porocity value the higher of its combustion temperature and higher of its titanium substitution of the Hydroxiapatite. The test higher density value the higher of its combustion temperature and higher of its titanium substitution of the Hydroxiapatite. The smaller its absorption value of water with the higher of its combustion temperature and higher of its titanium substitution of

the Hydroxiapatite. Absorption value of water got for Ti-HA(M_4) compound of 1200 °C temperature is 0.49% smaller than that is HA(M_0) compound is 3.28%.

The higher its compressive strength value with the higher of its combustion temperature and the higher of its titanium substitution of the Hydroxiapatite. Compressive strength value got for Ti-HA(M_4) compound of 1200 °C temperature is 36.268 MPa. Highest than that is HA(M_0) compound is 24.162 MPa. The higher is hardness value with the higher of its combustion tempersture and the higher of its titanium substitution of the Hydroxiapatite. The highest hardness value Ti-HA (M_4) compound of 1200°C temperature is 18.093 MPa and on HA(M_0) 11.878 MPa . The tensile strength highest value on Ti-HA in 1200°C temperature is 4.690 MPa and on HA 2.542 MPa. The highest modulus elasticity value Ti-HA on 1200°C temperature is 53.295 MPa and on HA 30.622 MPa.

Degradation testing conducted by soaking the sample into SBF/Synthetic Body Fluid solution for 12 weeks, physics form of HA and Ti-HA yields remain to like from the beginning, no decomposition, and degradation.

Biocompatibility testing conducted with toxicity testing and emergence of fibroblast cell (cell line BHK 12 clone 13) in an in vitro manner, in yield of testing either of HA (M_0) compound, Ti-HA(M_4) compound of 1200 °C temperature is no toxic and cell will grow. The higher its grow is Ti-HA (M_4) compound of 1000 °C temperature.

Titanium substitution synthesis yield for HA in this research is increase physics properties such as : porocity, dencity, absorption of water and also mechanical properties such as: compressive strength, hardness, tensile strength, modulus elasticity is requirement biomaterial syntetic bone. Titanium-Hydroxyapatite compound can usefull as biomaterial alternative syntetic bone which application must adjusted with design implantation place and need to be further study.

ABSTRACT

Titanium- Hydroxyapatite Synthesis : The Effort to Improve Quality of the Hydroxyapatite As Synthetic bone

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Human bone and teeth as well as animal contains hydroxyapatite compound with $Ca_{10}(PO_4)_6(OH)_2$ formula, hydroxyapatite found in 65-70% composition in bone and 90-95% in teeth.

Hydroxyapatite is ceramics compound or inorganic compound with most formed from calcium and phosphate. Hydroxyapatite compounds synthesis can be made by artificial in laboratory that reacting calcium hydroxide $Ca(OH)_2$ and phosphate acid with ratio Ca/P = 1.33 – 1.67.

The features that prominent from hydroxyapatite compound are porous, resorption, bioactive, non-corrosive, inert and worn-out resistance. The purpose of this research is improving physics and mechanics of the nature that including compressive strength, hardness, tensile strength and modulus elasticity of hydroxyapatite by substitute titanium metal by solution method.

On this research conducted hydroxyapatite synthesis that is reacting suspension of $Ca(OH)_2$ by concentration 0.5 M and 0.3 M solution of H_3PO_4 and titanium-hydroxyapatite synthesis.

Substitution reaction of titanium conducted by reacting various solution concentration of $Ca(OH)_2$ into the mixture of various solution concentration of $TiCl_3$ that already mixed into H_3PO_4 solution.

Reaction conducted in the reactor by temperature of 40-50°C, with mixing continuously and monitored early pH, when reaction and settlement. Reaction conducted 2½ hours in batch. Yield washed, filtered, dried in temperature of 110°C and molding as sample in thin cylinder, cube and bar form. Sample burned in temperature 800°C and variation on 1000°C, 1100°C, 1200°C.

Testing yield has got porosity value, the higher of its combustion temperature the smaller its porosity. The higher of combustion temperature the higher density value Hydroxyapatite and Titanium-Hydroxyapatite in temperature 1100°C for HA 3.512 g/cm^3 and Ti-HA 3.600g/cm^3. Absorption value of water got for HA higher that is 3.28% compared by water Ti-HA absorption that is 0.49%.

As for testing yield compressive strength have highest value on Ti-HA and HA in 1200°C temperature is 36.268 MPa, the highest hardness value Ti-HA on 1200°C temperature is 18.093 MPa and on HA 11.87 MPa, the tensile strength highest value on Ti-HA in 1200°C temperature is 4.690 MPa. The highest modulus elasticity value Ti-HA on 1200°C temperature is 53.295 MPa.

Degradation testing conducted by soaking the sample into SBF/Synthetic Body Fluid solution for 12 weeks, physics form of HA and Ti-HA yields remain to like from the beginning, no decomposition, and degradation.

Biocompatibility testing conducted with toxicity testing and emergence of fibroblast cell (cell line BHK 12 clone 13) in an invitro manner, in yield of testing either of HA is good, Ti-HA no toxic and cell will grow.

Key words : Hydroxyapatite, substitute titanium metal, synthetic bone