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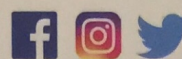
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- S1101** Powder/Liquid Ratio Effects on Mechanical Properties of Modified β -TCP Cement. Y. IDA*, J. BAE, K. SEKINE, F. KAWANO, K. HAMADA (Tokushima University, Tokushima, Japan)
- E1102** Microstructure Analysis and Hardness Test of Geopolymer Based Nano-Composite Materials. A. EVELYNA*, I. SURYATMOJO, I. ESSYTA, B. SUNENDAR PURWASMITA (Dental Material, Faculty of Dentistry, Maranatha Christian University, Bandung, West Java, Indonesia)

Seq#: 167 Friday, 24 June 2016, 2 p.m. – 3:15 p.m.
Poster Session, Hall B

Dental Materials 4: Adhesion – Universal Adhesives & Dentin Treatments in Adhesive Dentistry

- C1103** Application of Ethanol Wet-bonding in the Resin Restoration on Pulpless Permanent Teeth. Z. JIE* (Out-patient Center, Peking University School and Hospital of Stomatology, Beijing, China)
- I1104** Effect of Different Cross-Linkers on Dentin MMPs. L. BRESCHI*, V. ANGELONI, C. MAZZITELLI, M. CADENARO, L. TJADERHANE, F. TAY, D. PASHLEY, A. MAZZONI (Department of Medical Sciences, University of Bologna, Trieste, Italy)
- S1105** Effect of Different Aging Methods and Ethanol Wetting on Microtensile Bond Strength of a Self-etch Adhesive. Y. SESEN USLU*, S. HERGUNER SISO, N. DONMEZ (RESTORATIVE DENTISTRY, BEZMIALEM VAKIF UNIVERSITY, ISTANBUL, FATI, Turkey)
- C1106** Effect of MDP on Enamel Bonding Durability of Self-etch Adhesives. T. TAKAMIZAWA*, K. TSUCHIYA, Y. HOSOYA, W. BARKMEIER, K. TSUBOTA, A. TSUJIMOTO, M. LATTA, M. MIYAZAKI (Operative Dentistry, Nihon University, Tokyo, Japan)
- I1107** Enhanced Dentin Bonding Stability Using Blue-Light Activated Riboflavin. S.-F. CHUANG*, J.-P. HSIEH, Y.-L. CHEN (Institute of Oral Medicine, National Cheng Kung University, Tainan, Taiwan)
- S1108** SEM Analysis: UVA-activated Riboflavin Cross-link to Sound and Caries-affected Dentin. Y. AHN*, R. LAMY, C. DARLING, J. STEWART, L. PINZON (School of Dentistry, University of California, San Francisco, San Francisco, California, USA)
- I1109** Effect of DCC on Immediate Bond Strength. A. MAZZONI*, V. ANGELONI, V. CHECCHI, R. DI LENARDA, N. SCOTTI, F. TAY, D. PASHLEY, L. BRESCHI (Clinica Odontostomatologica, University of Trieste, Trieste, Italy)
- SE1110** Bond Strength of a Universal Adhesive with Different Application Protocols. P. SAIKAEW*, H. SANO, A.F.M. CHOWDHURY (School of Dentistry, Hokkaido University, Sapporo, Japan)
- S1111** Bond Strength of Multi-mode Adhesives to Dentin: Systematic Review and Meta-analysis. A. ELKAFFAS*, H. HAMAMA, S. MAHMOUD (Operative Dentistry, Faculty of Dentistry, Mansoura University, Mansoura, Egypt)
- S1112** Effects of Dentin Wetness and pH of Universal Adhesives on Dentin Bond Strength. A. CHOI*, S.-A. SON, J.-K. PARK, J. LEE (Conservative dentistry, Pusan national university, Yangsan, Kyungnam, Korea (the Republic of))
- C1113** The Evaluation of New All-in-one Self-etching Adhesives. T. HOSHIKA, Y. NISHITANI*, K. TAKAHASHI, T. KAJIHARA, M. YOSHIYAMA (Restorative Dentistry and Endodontology, Kagoshima University, Kagoshima, Kagoshima, Japan)
- I1114** Influence of Air-powder Polishing on Bond Strength of Universal Adhesive Systems. Y. TAMURA*, S. SHIBASAKI, K. TSUBOTA, T. TAKAMIZAWA, H. KUROKAWA, M. MIYAZAKI, K. HINOURA (Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan)
- SC1115** Adhesive Layer Thickness Of Universal Adhesives And Effect Of Air-thinning. K. CHO*, S.-H. PARK (Department of conservative dentistry, Inha University Hospital, Incheon, Korea (the Republic of))
- C1116** Comparison of Mode of Failure of New Universal Adhesives. M.T. PULIDO*, A. TANG, E. WIESNER, J. MITCHELL (College of Dental Medicine, Midwestern University, Glendale, Arizona, USA)
- C1117** Influence of Evaporation Time on Hydrophilicity of Different Universal Adhesives. A. MATSUMOTO, A. KASTRATI, R. HAMBURG, W. LOH, M. LECHMANN-DORN*, J. SCHWEPPE, A. LEYER (Heraeus Kulzer GmbH, Hanau, Germany)
- I1118** Dentin Etching Effects on Microshear Bond Strength of Universal Adhesives. X. HU*, H. ZHANG, J. SORENSEN, A. SADR (Restorative Dentistry, University of Washington School of Dentistry, Seattle, Washington, USA)
- I1119** Evolution of Recent Universal Adhesives – Chemistry and Performance. C. THALACKER*, H. LOLL, I. EDELMANN, R. GUGGENBERGER, B. KIM (3M Oral Care, 3M, Deutschland GmbH, Seefeld, Bavaria, Germany)
- I1120** Immediate Microtensile Bond Strength Evaluation of Universal Adhesives. N. SIRINSUKAN*, E. CAN SAY (Department of Restorative Dentistry, Yeditepe University, Istanbul, Turkey)
- S1121** Dentin Bonding in Physicochemical Properties of Adhesives Incorporated With Epigallocatechin-3-gallate. A. DE MACEDO*, M. LEMOS, N. SOUZA, V. FEITOSA, S. SANTIAGO (Restorative Dentistry, Federal University of Ceara, Fortaleza, Ceara, Brazil)
- I1122** Effect of Previous Acid Etching and Application Mode on Enamel Bonding Durability of Self-etch Adhesives. K. TSUBOTA*, T. TAKAMIZAWA, A. TSUJIMOTO, M. MIYAZAKI, M. LATTA (School of Dentistry, Nihon University, Tokyo, Japan)

Seq#: 168 Friday, 24 June 2016, 2 p.m. – 3:15 p.m.
Poster Session, Hall B

Dental Materials 5: Biocompatibility and Biologic Effects of Materials – Antimicrobial Effects of Dental Materials

- I1123** Synthesis and Characterization of a Novel Antibacterial and Bioactive Root Canal Sealer Precursor. X. CHENG, T.-J. QU, Q. YU, X. LIU* (Baylor College of Dentistry, Dallas, Texas, USA)
- C1124** Biofilm Formation on Dental Restorative Biomaterials. S. DENG*, D. CHAN, K.-H. CHUNG, R. FRANCISCO, J. BRYERS (Department of Conservative Dentistry, Affiliated Hospital of Stomatology, Zhejiang University, Hangzhou, Zhejiang, China)
- I1125** Antibacterial Activity of Dental Restorative Materials Containing Urushiol Extracts From the Korean Lacquer Tree. H. MOON, D.-H. SHIN* (College of Dentistry, Dankook University, Chuncheon, Choong-Nam, Korea)
- SE1126** Surface Property Change of Titanium After Bacterial Inoculation and Debridement. G.-S. SHIN*, B.-S. LEE, Y.-W. CHEN (National Taiwan University, Taipei, Taiwan)

Research Reports

SYNTHESIS AND MICROSTRUCTURE ANALYSIS of Al-Mg-Si-Zr GEOPOLYMER BASED NANO-COMPOSITE FOR DENTAL MATERIALS APPLICATION

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Abstract.

Science and technology of dental materials development provides variety of dental products that fulfils the needs of aesthetics and physical properties. There are wide ranges of aesthetics dental materials, one of them is dental composite. The use of inorganic ceramic nanoparticle filler on dental composite system has provides many advantages. However it have severals limitations due the use of polymer resins as matrix such as a low mechanical property. To overcome the problem, geopolymer nanocomposite materials of Al-Mg-Si-Zr used to substutute resin polymer. Geopolymer materials have reliable properties such as low density, easily processed, and low cost.

The objective of this study is to synthesis four different types of inorganic ceramic fillers, alumina, magnesia, silica, and zirconia using sol-gel technique, with two differents alkali activator concentrations (8 M and 12 M) and then cooperate them with geopolymer as matrix to characterized its microstructure through XRD and SEM to know the phase characterization, crystal structure, and size.

The X-Ray Diffraction and Scanning Electron Microscopes Characterized that, Al-Mg-Si-Zr have been formed with nano sized particles. Microscopic morphology of Mg-Al-Si-Zr

nanocomposite with alkali activator concentration 8 M has more porosity than 12 M that lead to better mechanical property.

Keywords: Al-Mg, Si, Zr, geopolymer, nanocomposite, dental materials

INTRODUCTION:

Individual or community oral health is the key to achieve a good quality of life. Most of Indonesian people have known oral health educations and get the access to oral health services. But somehow, the optimal health service could only perceived by the middle-high economic group of the community. This happens because inability of dental manufacturer in Indonesia to produce variety dental products with good quality, therefor most of dental material products are still imported from the other country, which results in high cost of dental health services. Dental parctice could not be seperated from the use of various dental materials to suport dental health care and therapy. One of the mostly used dental materials are dental composites.^{1,2}

Generally, composites are combination of two or more different materials to get a better property. Dental composite is a restorative resin material that consists of inorganic fillers such as amorphous silica, glass, and crystalline materials that combine with organic matrix resins by the use of coupling agent. Most of dental composite contains synthetic resin matrix to increase binding properties between polymers and fillers. The matrix resins that being used are aliphaticdimethacrylate monomer (bis-GMA), triethylene glycol dimethacrylate (TEGDMA), and urethane dimethacrylate (UDMA). Synthetic resins have a low physical and mechanical property.³

Geopolymer is an inorganic polymer material, can be activated by silica-alumunium rich mineral. Geopolymerization occur through complex exotermis process. Geopolymer beginning to be liked and improved because its excellent physical and mechanical properties, compared with synthetic polymer resin. Geopolymeric materials can be used on extremely high temperature condition, it can reach 1000 °C. The firing gas product is not combustibile and poisonous.^{4,5}

Addition of nano-particle ceramic filler material has been much used in the process of making dental composite material restoration. Ceramic filler material which been used is alumina (Al_2O_3) because of its crack propagation resistention and good thermal expansion coefficient. Magnesium Chlorida (MgCl) been used as alumina stabilizer. Silica (SiO_2) has good strength and esthetic. Zirconia (ZrO_2) is an inert material and has good fracture

toughness. All of the four kind ceramic fillers are nano-sized powder blend by optimal proportioning weight percentation then combined with polymer matrix through the use of coupling agent to make nano composite material. Coupling agent is a substance applied at the surface of filler particle to create adhesivity between fillers and matrix. Chitosan is a kind of organic polymer made from sea creatures shells such as crustacea. Chitosan can be used as a coupling agent because its adhesivity to ceramic materials. Chitosan has been widely used as biomaterials. ⁶⁻⁹

Nanotechnology has led us to improvement of biomaterials engineering. Nanosized is the smallest size that can be reached by synthesis method existed until now. The smaller the particle size, the more filler particles can be inserted to the composite, so the better mechanical property can be reached. One technique that has been chosen to synthesize ceramics nanoparticles is sol-gel technique (precursor) that mainly involved two processes, hydrolysis and condensation. This technique was widely chosen because it is more efficient and cheaper, it only requires some relatively simple instruments compared with other technique. Inorganic ceramic nanoparticles fillers can be synthesized using sol-gel technique. ¹⁰

This study attempts to synthesize nanocomposite based geopolymer alumina-silica-magnesia-zirconia materials for dentistry application and characterized its microstructure.

MATERIALS AND METHODS

MATERIALS

Materials used in this research are $MgCl_2$, Tetraethyl-orthosilicate (TEOS), $Al(NO_3)_3$, $ZrCl_4$ as precursors, Chitosan as coupling agent, a solvent of demineralization aqua liquid (Aqua DM), crystalline NaOH and waterglass/ sodium silicate (Na_2SiO_3).

METHODS

This study is a pure laboratory experimental research study. Sample used in this study are Mg-Al-Zr-Si nanocomposite bars with different mass fraction and activator alkali with different molarity to see morphological characteristics.

The research procedure divided into several stages. The first stage is producing NaOH solution, followed by making alkaline activator solutions and 2% Chitosan solution. Mg-Al-Si-Zr prepared by using sol-gel methods. The last stage is to characterize its microstructure and filler distribution by x-ray diffraction (XRD) and scanning electron microscope (SEM) analysis to analyze the phase and structure of crystal lattice by using

philips analytical x-ray B.V, diffractometry type PW1710 and the scanning electron microscopy (SEM) JSM 6510 LV. ^{11,12}

RESULTS AND DISCUSSION

The Mg-Al-Si-Zr geopolymer based nanocomposite by firing temperature of 800 °C XRD characterization results shows diffractogram as follow (fig. 1).

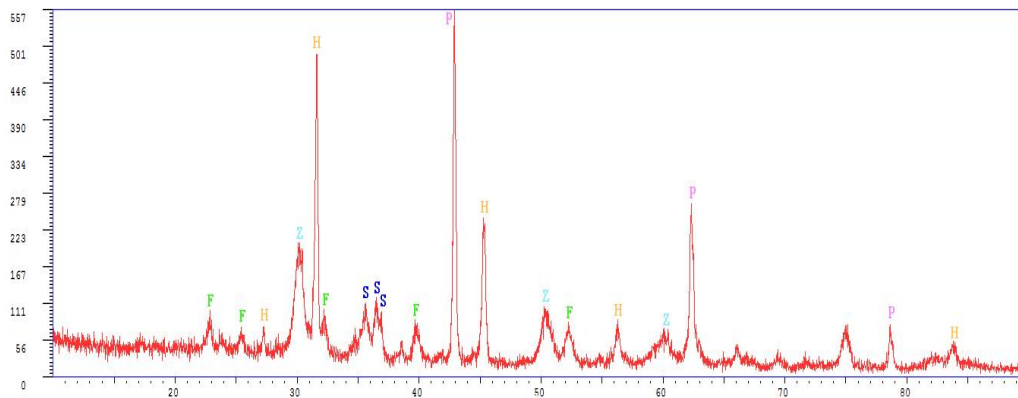


Figure 1. Diffractogram of Mg-Al-Si-Zr powder

Synthesize product diffractogram compared with diffraction pattern from Joint Committee on Powder Diffraction Standard (JCPDS) to determine which crystal lattice formed.

Diffractogram results analyzed using xpowder software in order to obtain information about crystal lattice. Crystalline phase of MgAlO_4 spinel (JCPDS no. 21152) have been successfully synthesize, shown with the emergence of diffraction pattern at 37° peak. MgAlO_4 spinell fractions produced were still to view (8.2 %). This could be happen because the calcination temperature has not steady yet. ZrO_2 phase (JCPDS no. 270997) indicate by diffraction patterns at 30°, 50° and 60° peak. Beside those compounds there are also another compound such as MgO periclase (JCPDS no. 450946), Mg_2SiO_4 forsterite (JCPDS no. 340189), and NaCl halite (JCPDS no. 050628). MgO periclase shown by 43°, 62° and 79° diffractions peak. Mg_2SiO_4 forsterite shown by 23°, 25°, 32.5°, 40° and 52° diffractions peak. NaCl halite shown by 27.5°, 31.5°, 25.5° and 56.5° diffractions peak.

SEM figure of Mg-Al-Si-Zr nanocomposite with 6:2:1:1 filler proportion and alkaline activator concentration addition of 8M and 12M at 500, 1.000, and 2.500 magnifying shows patterns as bellow.

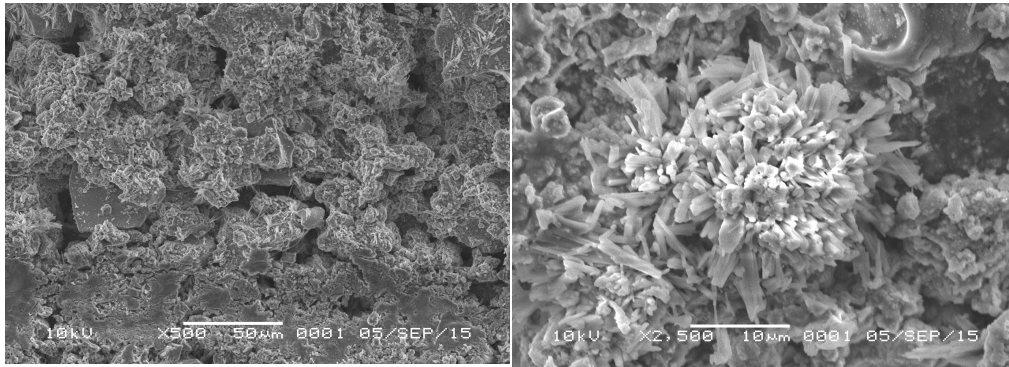


Figure 2. SEM Figure of Alkaline Activator 8M (500x and 2500x)

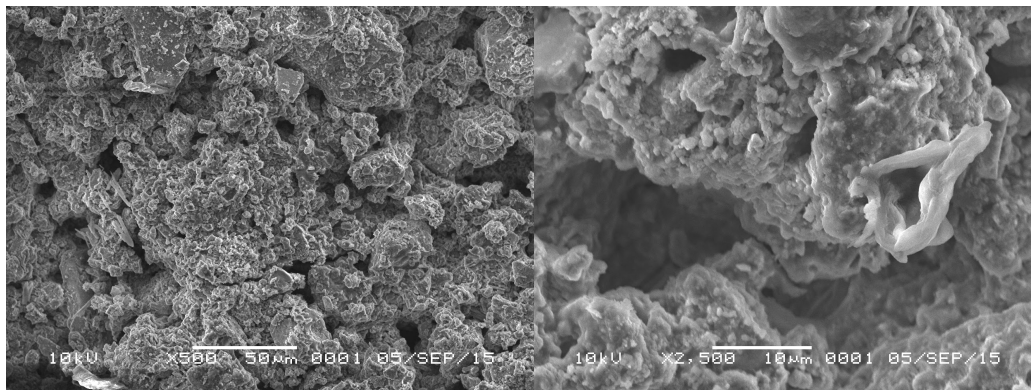


Figure 3. SEM Figure of Alkaline Activator 12M (500x and 2500x)

SEM characterization figures shows, Mg-Al-Si-Zr nanocomposite with 8 M concentration is more homogeneous compared with Mg-Al-Si-Zr nanocomposite with 12 M concentration. This can be seen from figure 2 where fillers are equally distributed, there are only few tufts (agglomerations) and pores compared to figure 3, which is more globules and fillers are not distributed equally and homogeneous.

Therefore, Mg-Al-Si-Zr nanocomposite with 8 M alkaline activator concentration has a better mechanical properties compared with 12 M. SEM figures also shows rods morphology at figure 3 which may results from silica particles. Silica filler particles of Mg-Al-Si-Zr nanocomposite 12 M are not shown so much, which can lead better bonding ability between silica filler and geopolymer matrix, however there are more cracks on Mg-Al-Si-Zr nanocomposite 12 M so the mechanical properties are lower.

CONCLUSION

Mg-Al-Si-Zr nanocomposite has been successfully synthesized using sol-gel method through calcination process at 800 °C for two hours and formed MgAlO₄ spinell (JCPDS no. 21152) crystal lattice, but the spinell fractions are still too few.

SEM characterization shows Mg-Al-Si-Zr nanocomposite with 8 M alkaline activator concentration looks more homogeneous compared to 12 M. So it can be concluded that the mechanical properties of 8 M possibly better than 12 M.

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