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Hardness test of Polymethyl Methacrylate (PMMA) Filled with Self Synthesized Ceramic Fillers of SiO_2-Al_2O_3-ZrO_2 for Indirect Restorative Materials. (Two different Filler/PMMA powder ratios)

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Abstract- Polymethyl methacrylate (PMMA) is a synthetic polymer that often used as temporary restoration of jacket crowns and bridges. The purpose of this study is to measure the hardness of Heat-cured PMMA which filled by a ceramic filler system SiO_2-Al_2O_3-ZrO_2 with different ratio of filler PMMA in order to increase the mechanical properties (hardness) whichwill prolong the restoration self life.

An experimental descriptive study was used to analyse SEM and XRD characterization of ceramic filler SiO_2-Al_2O_3-ZrO_2 system which synthesized using the sol-gel technique. The filled PMMA samples were then tested using a micro vickers tester LECO-Japan-tipeM-400 H1/H2/H3 with 200 gram force 15 for seconds.

The SEM result showed the particle size of 83 nm with uneven and less homogeneous distribution due to the agglomeration and its particles showed tetragonal zirconia and α-alumina crystallites according to the XRD . The hardness of filled PMMA using ratio of 1 : 2 and 1 : 4 were 14,80 VHN and 16,07 VHN respectively. Meanwhile control showed 15.27 VHN.

As a conclusion ceramic system of SiO_2-Al_2O_3-ZrO_2 synthesized by sol-gel technique can be used as fillers. And the filled PMMA with filler PMMA powder ratio of 1 : 4 has the highest hardness value of 16.07 VHN.

Keywords: Hardness; Polymethyl Methacrylate; Filler; Sol Gel

1. Introduction

Acrylic resin or Polymethyl methacrylate (PMMA) is a polymer that is commonly used in dentistry especially for indirect restorations such as denture base material and provisional crown jackets[1]. PMMA was chosen because of its aesthetic and has a good physical and biological properties, biological and aesthetically good, but its clinical use as restorative materials lead to cracks due to the low ability to resist the pressure and flexibility. PMMA consisting has two main components that are polymer (powder) and monomer (liquid). Polymer and monomer ratio has an affect the mechanical properties. Generally the polymer/monomer ratio used is based on volume 3: 1 or 2 - 2.5: 1 by weight[2],[3].

In order to improve the mechanical properties fillers are commonly added to the polymer. Some fillers mostly used in dentistry including ceramics, glass and resin. Some examples of ceramics are widely used as raw materials in the field of dentistry such as silica (SiO_2), zirconia (ZrO_2) and alumina (Al_2O_3)[4],[5],[6]. Silica in the nature usually form of quartz. Pure silica containing high levels of translucency so it is often used as one component of a porcelain body material[7]. Silica has a better translucency properties than

hardness[8]. While Zirconia widely used to reinforcing material because it has the mechanical properties comparable to steel[4],[9]. The Alumina additions in the manufacture of dental ceramics improve the mechanical properties of tetragonal zirconia phase namely fracture resistance as a result of the transformation thoughening mechanism. For these reasons, Alumina is often used as a stabilizer[4],[10].

One of the techniques for manufacturing glasses and ceramics is Sol gel technique which produce nano particle[11].

The research purposed is to increase the mechanical properties of PMMA by addition self synthesized ceramic fillers of SiO2-Al2O3-ZrO2 using sol-gel technique.

2. Materials and Methods

The research procedures were divided into 2 stages consist of sol-gel technique to synthesized ceramic particles of SiO_2-Al_2O_3-ZrO_2 and to test PMMA samples filled with the ceramic particles.

- The Sol-gel technique for synthesizing SiO_2-Al_2O_3-ZrO_2 ceramic particles[5].

1. Synthesis of ceramic systemof Silica-Alumina-Zirconia

The precursors used were $ZrCl_4$, $Al(NO_3)_3$ and TEOS with ratio of (20%: 10%, 70%) diluted with 100 ml aquabidest + chitosan 1% using a magnetic stirrer for ± 45 minutes then homogenized the solution using ultraturax and ultrasonic subsequently placed on the oven at a temperature of 80° C. Put the sample in furnace at 900° C to remove carbon oxide. Next the powder was homogenized in beaker glass with ethanol using ultrasonic and ultraturax homogenizer for 6 times with 5 minutes interval with 1 minute pause after each interval. Finally put the beaker glass above an oven until it dry become powdery

2. Characterization using SEM and XRD

Some powder then characterized with SEM (JEOL-JSM-6510LV) and XRD.

3. The rest of ceramic system filler Silica-Alumina-Zirconia was silanizated by chitosan 1%

Preparation of chitosan 1% solution materials and 98 ml aqua bidest. Mix the chitosan powder and acetic acid in aqua bides use magnetic stirrer for ± 30 minutes until all the chitosan has dissolved.. Put the filler into Beaker glass and chitosan 1% until all the powder was soaked then put it in a water bath for 1 hour then dry it above the oven.

- Sample preparation of PMMA added by filler ceramics[6].

1. PMMA sample preparation.

Nine samples were made using conventional method as making heat-cured acrylic dentures. The samples formed in disc shape 6 mm diameter x 3 mm thicknes. The specimen was divided into three groups. Firstly PMMA powder/fillers ratio 1:2. Secondly PMMA powder/fillers ratio 1:4 and thirdly PMMA without filler as control group. All samples were then soaked in aquabidest for 24 hours at 37 ° C in the incubator[5].

2. Hardness Test

Mechanical tests were carried out in the form of hardness test by means of mickrovickers performed on a test specimen. Tests conducted at the Laboratory of metallurgical physics and ceramics, Faculty of Mining and Petroleum Engineering, Institute of Technology Bandung. Tests using the test machine LECO-Japan-M-400 H1 / H2 / H3 with a load of 200 grams for 15 seconds.

3. Result and Discussion

Results of the research were a qualitative data characterization of filler SiO_2-Al_2O_3-ZrO_2 using Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD) while quantitative data in the form of the results of hardness test specimen made of PMMA filled with SiO_2-Al_2O_3-ZrO_2 in different filler/PMMA powder ratio (1: 2 and 1: 4 respectively).

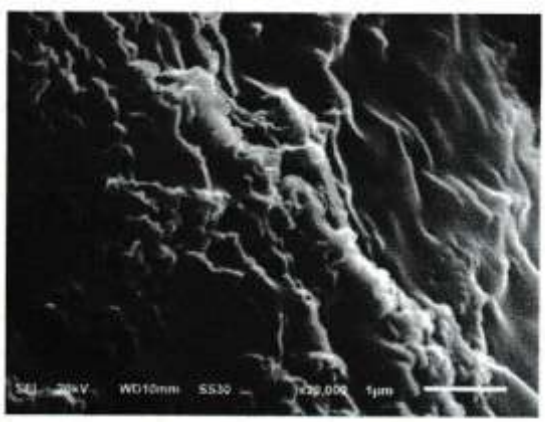

Fig. 1. SEM Characterization of SiO_2-Al_2O_3-ZrO_2 Filler calcination

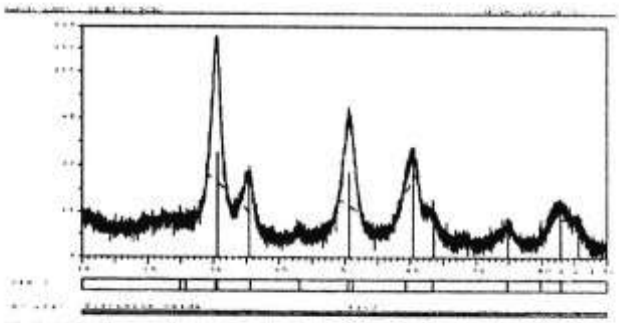

Fig.2. XRD Chatacterization Result with calcination temperature 900°C

1 : 4

SEM characterization of particles showed size of particle is 83 nm and a description of the agglomeration of the particles while the XRD results has a similar peak to PDF number zirconia = 42-1164[12] and PDF number Alumina = 46-1212[13] show phase of α-alumina and tetragonal zirconia. Tetragonal phase of zirconia crystallites have transformation thoughening mechanism which has an ability to withstand crack propagation to improve the mechanical properties (hardness) a restoration. The formation of tetragonal phase of zirconia crystalite in the SiO_2-Al_2O_3-ZrO_2 produced system showed that the ceramic can be used as a filler for PMMA.

The mean value of hardness for group A (without filler) amounted to 15.27 VHN higher compared with group B which filled PMMA (1: 2) with a mean value of 14.8 VHN hardness. Theoretically, the group B should has a higher value than group A because in group B were added another material (filler) which should serve as a reinforcement material for PMMA. Results of Karlina research in 2014 also uses chitosan 2% as a coupling agent for polymethyl methacrylate filler produces heat-cured hardness value is also lower (13.3 VHN) compared to controls. From the results obtained it can be stated that the using of different concentration coupling agent also affects the mechanical properties of a particular restoration surface hardness.

Group C is filled PMMA (1: 4) with a mean value of 16.07 VHN hardness which is higher than the group B filled PMMA (1: 2) with an average value of 14.8 VHN hardness. This is probably due to different ratio of the filler and PMMA that were used. Refference [14] shows one of the factors that affect the mechanical properties (hardness) is the ratio of filler and matrix. Filler ratio of often used in the manufacture of dental restorations in a different volume or weight ratio. Percentages in volume was 50% and the ratio is equal to the ratio of the percentage by weight of 75%. In this research, the weight ratio of the group B has a weight ratio of filler and PMMA by 1: 2, while in group C having the weight ratio between filler and PMMA of 1: 4. altough Group C have less weight ratio they showed high average value of hardness presumably it caused the amount of filler with more ratio may hinder bonding between the particles monomers into polymers in polymerization so that the bond formed is not strong enough.

Table 1. Hardness Test Result

Indentatio		1	2	3	4	5	Average (VHN)	Total Average (VHN)
A Group	A	14,7	15,2	14,9	14,9	15,4	15,0	
(-*filler*)	B	15,2	15,4	15,5	15,2	15,4	15,3	**15,27**
	C	15,1	15,7	15,6	16,1	15,2	15,5	
B Group	A	13,3	15,4	14,8	16,0	14,4	14,8	
Filler **:**	B	15,0	14,6	15,1	14,0	15,4	14,8	**14,80**
PMMA	C	15,0	14,3	13,3	15,3	16,2	14,8	
1:2								
C Group	A	16,4	16,2	16,0	15,8	16,3	16,1	
Filler **:**	B	13,5	15,1	17,2	16,4	16,6	15,8	**16,07**

4. Conclusion

As a conclusion ceramic system of SiO_2-Al_2O_3-ZrO_2 synthesized by sol-gel technique can be used as fillers of PMMA based on SEM and XRD characterization. And the filled PMMA with filler PMMA powder ratio of 1 : 4 has the highest hardness value of 16.07 VHN. Further research needed to find out which the right filler/PMMA powder ratio in order to obtain optimal result.

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[1] Evelyna, A., Sintesis dan Analisis Mikrostruktur Al_2O_3-SiO_2- ZrO_2 Berukuran Nano Sebagai Bahan Pengisi Restorasi Mahkota Jaket Resin Polymethylmethacrylate Heatcured Serta Uji Sifat Mekanisnya, Thesis, Program Pendidikan Magister Program Studi Ilmu Kedokteran Gigi, *Universitas Padjadjaran*: 2-4, 6-9, 13-17, Bandung, Indonesia. 2010.

[2] Sharma, P. R., et al., The Effect Of Incorporation Of Non Metallic Fillers On The Strength Of Polymethymethacrylate, Journal, *India Dental Journal of Dental Sciences*, India, 2014.

[3] Hamza, T., A. G Wee, H Alapati, S. R Schricker, The Fractur Thougness of Denture Base Material Reinforced with Different Concentrations of POSS, USA. *Marcel Dekker, Inc*: 898- 904, 907-1000, 2004.

[4] Anusavice, K. J, Phillips' Science of Dental Materials, 11th ed. St. Louis, Missouri, *Elseviers Saunders* : 656-657, 2003.

[5] Hasratiningsih, Z., dkk, Evaluasi Sifat Mekanis Komposit Gingiva Buatan Sendiri dengan Filler Hasil Sintesis Sistem ZrO_2-Al_2O_3-SiO_2 Menggunakan Teknik Sol-Gel dan Di-Coating Kitosan Satu Persen, *Proceeding Book RDM&E 6th ed*: 41-48, Medan, Indonesia, 2014.

[6] Karlina, E., dkk, Evaluasi Bahan Restorasi Indirek Pengganti Porselen Dengan Penggunaan Polymethyl Methacrylate Yang Ditambahkan Filler Keramik Nanopartikel ZrO_2-Al_2O_3-SiO_2 dan Coupling Agent Kitosan, *Kumpulan Abstrak Seminar Nasional Keramik XII* : 4-17, Bandung, Indonesia, 2014.

[7] Abdelaaziz, M. A, Synthesis of Nanocomposite with Nano-TiO2 Particles and Their Aplications as Dental Materials, Thesis, Magister Degree of Technology: Dental Technology, *Cape Peninsula University of Technology*: 16-20, 2012.

[8] Mizrahi, B, All-Ceramic Silica/Glass-Based Crowns Clinical Protocols, UK, *British Dental Journal*: 257-260, 2011.

[9] Vagkopoulou, T., S. P Koutayas, P Koidis, J.R Strub, Zirconia in Dentistry: Part 1. Discovering the Nature of an Upcoming Bioceramic, Germany; *The European Journal of Esthetic Denstistry*: 134-137, 2009.

[10] O'Brien, W. J., Dental Material and Their Selection. 3rd ed. Canada, *Quintessence*: 13-28, 32-40, 2002.

[11] Brinker C.J., G.W Scherer, Sol-Gel Science: The Physics and Chemistry of Sol-Gel Processing. USA. *Academic Press*: 2-3, 1990.

[12] Zhao, Y., Li, W., Zhang, M., Tao, K., A Comparison of Surface Acidic Features between Tetragonal and Monoclinic Nanostructured Zirconia, China, Elsevier: 240-244, 2002.

[13] Sherikar, B, N., Umarji, M, A, Synthesis of γ- Alumina by Solution Combustion Method Using Mixed Fuel Approach (Urea + Glycine Fuel), India, International Journal of Research In Engineering and Technology: 434-438, 2013.

[14] Roberson, T, M., *et al*. Sturdevant's Art & Science of Operative Dentistry, 4th ed. USA. *Mosby*: 192-195, 2002.