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THE STUDY ON ALUMINA STABILIZED ZIRCONIA-WHITE CARBON BLACK NANOFILLERS IN RESIN COMPOSITE FOR DIRECT RESIN BONDED PROSTHESIS

Studi Tentang Nano Filer Zirconia terstabilkan Alumina Koloidal Silika dalam Komposit Resin Bonded Prosthesis

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ABSTRACT

Nanofilled composite, a resin composite consisting of a resin-based oligomer matrix and inorganic nanofillers, is one of the materials used to make a resin bonded prosthesis. The imported manufacture-made composite is expensive; therefore in this study, nanofilled composite have been prepared from the synthesized alumina stabilized zirconia-white carbon black nanoparticles as nanofillers and some resin-based oligomer matrixes. The aim of this study is to investigate the microstructure and the hardness between the prepared nanofilled composite as sample A and manufacture-made composite as sample B for direct application of resin bonded prosthes. However, the synthesized alumina stabilized zirconia consists of a tetragonal phase as a major phase. Microstructures of sample A show spherical and nanorod morphologies having an average size of about 149 nm. Whereas sample B has a spherical morphology with 153 nm in the average size. Since sample A was reinforced by polyethylene fiber with chitosan 6% as an adhesive material, it showed a smallest crack between nanofilled composite and fiber compared to the 2% and 4% of chitosan. This result is approaching to the investigation result of the sample B at the same manner. The hardness of sample A was 24.38VHN approaching to the hardness of sample B of about 27.48 VHN. This study shows the similar microstructure and hardness characteristics between the prepared nanofilled composites and manufacture-made composite.

Keywords : *Alumina Stabilized Zirconia, White Carbon Black, Nanofiller, Fiber Reinforced Composite, Hardness, Resin Bonded Prosthesis.*

ABSTRAK

Nanofilled komposit, komposit resin yang terdiri dari matriks resin berbasis oligomer dan nanofillers anorganik, merupakan salah satu bahan yang digunakan untuk membuat resin bonded prosthesis. Sedangkan komposit pabrikan impor cukup mahal; oleh karena itu dalam penelitian ini, nanofilled komposit dibuat dari bahan nanofiller hasil sintesis yaitu alumina stabilized zirconia dan white carbon black serta matriks resin berbasis oligomer senyawa organik. Tujuan penelitian ini ialah untuk membandingkan karakteristik mikrostruktur dan kekerasan antara nanofilled komposit

buatan sebagai sampel A dengan komposit pabrikan sebagai sampel B untuk aplikasi direct resin bonded prosthesis. Alumina stabilized zirconia yang digunakan memiliki fasa major tetragonal. Mikrostruktur pada sampel A memperlihatkan partikel-partikel berbentuk bulat dan batang nano dengan ukuran rata-rata 149 nm. Sedangkan sampel B terdiri atas partikel-partikel bulat dengan ukuran rata-rata 153 nm. Ketika sampel A diperkuat polyethylene fiber dengan dibantu penambahan bahan adhesif kitosan 6% menghasilkan celah paling kecil antara lapisan nanofilled komposit dengan polimer fibernya dibandingkan dengan konsentrasi bahan adhesif kitosan sebesar 2% dan 4%. Hasil ini mendekati ukuran celah antara sampel B dengan polyethylene fiber yang diolesi bahan adhesif pabrikan. Nilai kekerasan yang dihasilkan pada sampel A sebesar 24,38 VHN mendekati nilai kekerasan pada sampel B yaitu sebesar 27,48 VHN. Hasil penelitian menunjukkan adanya pendekatan karakteristik mikrostruktur dan sifat kekerasan antara nanofilled komposit hasil sintesis dengan komposit pabrikan.

***Kata kunci** : Alumina Stabilized Zirconia, White Carbon Black, Nanofiller, Fiber Reinforced Composite, Kekerasan, Resin Bonded Prosthesis.*

I. INTRODUCTION

A denture is a tool to replace missing teeth and surrounding soft tissues. It is generally made either by immediate dentures or conventional fixed dentures. Recently, a new design of fixed denture has been developed by using resin bonded prosthesis. It is known as resin bonded bridge or adhesive bridge [1,2]. Resin bonded prosthesis is a fixed denture that replaces one or two missing teeth using acid etching and bonding resin [3].

The advantage of resin bonded prosthesis is as an alternative option to lose a little bit of teeth; because it is more efficient, not too much reduction in dental tissues, and easy to install compared to immediate denture or conventional fixed denture [4]. One of the materials which is being developed for direct resin bonded prosthesis is a fiber reinforced composite [5]. It consists of fibers as a basic frame and resin composite as a veneer that coats the fiber [6]. The previous study found that a resin composite reinforced with ultra high molecular weight polyethylene fiber (UHMWPE) had adequate flexural strength so that it would be used for resin bonded prosthesis [7].

One of the materials in dental restoration, especially for the composite, is made zirconia. Zirconia was used as filler in dental composite materials and strengthening materials by creating crowns and bridge [8]. Zirconia is chosen because of having good dimensional stability, bio-inert, high bio-compatibility, and high mechanical properties such as high strength, high hardness, high toughness, high modulus of elasticity and flexural strength [9, 10]. In addition, zirconia exhibits a great aesthetic value having similar color with the color of teeth [11].

Therefore, the study on resin

composite zirconia-based filler for the application of direct resin bonded prosthesis is very interesting. It also compares the characteristics of microstructures and the hardness of manufacture-made composite materials. In this study, nanoparticles of alumina stabilized zirconia and white carbon black are used as composite nanofillers for the applications of direct resin bonded prosthesis while the polyethylene fiber is selected as a reinforced material. In addition, chitosan materials used as an adhesive material in fabricating fiber-reinforced resin composites [10, 12].

White carbon black, $SiO_2.nH_2O$ is used in alumina stabilized zirconia-white carbon black nanofiller because this material is porous and insoluble in water. It has high temperature resistance, good electrical resistance, translucent, and the hardness resembles the carbon, and it has the structure of nano-sized stems (nanorod) that exhibits a large surface area [13, 14]. Due to a large surface of nanorod structures of white carbon black, $SiO_2.nH_2O$ generates an attraction between the larger particles and strengthens the bonds between the filler and the resin matrix. Based on this, it is expected that the mechanical properties of a material may increase. The combination of alumina stabilized zirconia-white carbon black nanoparticles is expected to be a good nanofiller in resin composites. Therefore, since they are reinforced with the fiber, they could be a good resin bonded prosthesis material [13, 14, 15].

II. MATERIALS AND METHOD

In this study, the sample tested were nanofilled composite consisting of alumina stabilized zirconia-white carbon black nanofillers (sample A) and manufacture-made composite (sample B). Each sample was characterized by their microstructure and tested its hardness.

2.1 Synthesis of Alumina Stabilized Zirconia

The synthesis steps of alumina stabilized zirconia by a sol-gel method are shown in Figure 1. A 0.1 M zirconium precursor solution was mixed with a 5 % aluminum precursor solution and a 2 % chitosan solution under vigorous stirring at 10000 rpm. The solution mixture was then aged and was dried on a hotplate. The dried precursors were calcined at 900°C to produce alumina stabilized zirconia.

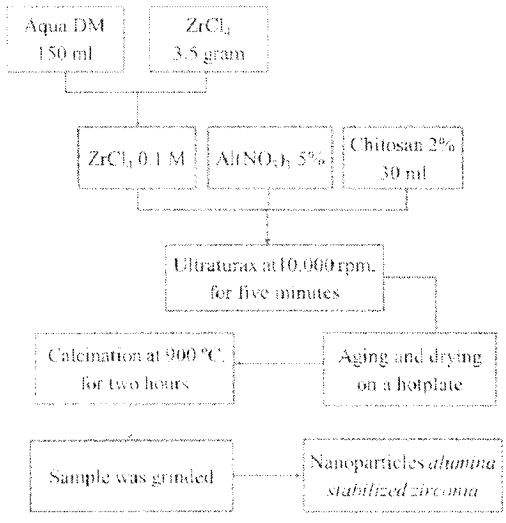

Figure 1. The synthesis steps of alumina stabilized zirconia

2.2 Synthesis of White Carbon Black

The synthesis steps of white carbon black by a sol-gel method are shown in Figure 2. About 12 ml of 0.5 M sodium silicates were dissolved in a mixture of 200 ml of ethanol and 20 ml of aqua DM. About 2.32 ml of 1% chitosan solution were added to the mixture under vigorous stirring at 10000 rpm. About 0.24 ml of 0.5% starch solution was added to the mixture under stirring. The final mixture was solidified using a centrifuge apparatus. The solid was dried in a vacuum condition and then was calcined at a temperature of 750°C for one hour. The obtained final product was white carbon black.

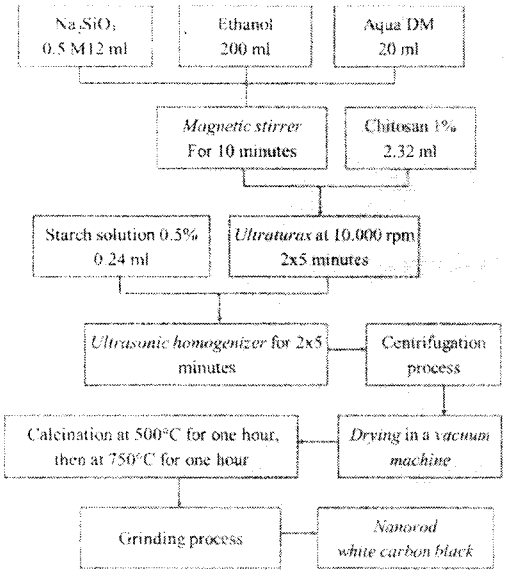

Figure 2. The synthesis steps of white carbon black

2.3 Preparation of Nanofilled Composites (Sample A) Using Alumina Stabilized Zirconia-White Carbon Black as Nanofillers.

Alumina stabilized zirconia and white carbon black as nanofillers were mixed at a ratio of 50:50. The fillers were immersed with 2% of chitosan and dried in an oven. The products were then mixed with a resin-based oligomer matrix such as UDMA (Urethane Dimetacrylate) (17%), TEDGM (Triethylene Glycol Dimethacrylate) 95% (5%), DMAEMA (Dimethylaminoethyl Methacrylate) 95% (5%), HEMA (Hydroxyethyle Methacrylate) 99+% (10%), and champorquinone 97% (1%). The mixture products were stirred until homogenous.

2.4 Sample Preparation for Hardness Test

Layers of nanofilled composites (sample A) and manufacture-made composite (sample B) were separately placed with a thickness of about 1 mm at the base of the mold with diameter of 6 mm and a height of 3 mm with pedestal matrix strip, then polymerized by light curing. Light curing process was conducted on the sample A for 4 minutes and 40 seconds for sample B.

The top of the first layer of the two groups was re-coated with 1 mm material that appropriate according to each group and polymerized by light curing, this step was repeated until it fills up the mold. At last, the entire sample was removed from the mold then did the polishing on the sample prior to testing.

2.5 Sampel Preparation for Microstructure Analysed

A layer of sample A and sample B was placed separately with a thickness about 0.5 mm in the bottom of the mold. Fiber was cut of to the appropriate length. Polyethylene fiber for sample A was wetted by chitosan in various concentrations of 2%, 4% and 6%. Meanwhile, polyethylene fiber for sample A was wetted with a spesific manufacture-made adhesive.

Each fiber which had been wetted with chitosan then put on the top of the synthetic nanofilled composite and the fiber that had been wetted with manufacture-made adhesive materials put in the mold and polymerized by light curing with 4 minutes illumination for sample A and 40 seconds for sample B. The fiber was then coated with materials that fit each group to fulfill the mold and then the top of the surface of the mold sealed with glass lab and pressed with a light pressure and pinned it on each side to suppress an excessive material then polymerized by light curing unit. The sample then polymerized using a light curing unit with a step that equal to the first layer. The entire sample was removed from the mold, then cutting and polishing was performed on samples before test it.

2.6 Microstructures Characterization and Hardness Test

The prepared sample was analyzed for its morphological characteristics by using SEM, hardness testing was performed using microvickers hardness tester.

III. RESULT AND DISCUSSION

3.1 The results of X-Ray Diffraction (XRD) on Alumina Stabilized Zirconia

The results of XRD characterization from alumina stabilized zirconia powder after calcined at a temperature of 900°C are shown in Figure 3.

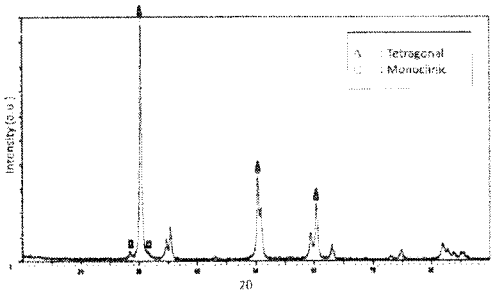

Figure 3. XRD results of alumina stabilized zirconias after calcined at a temperature of 900°C

The XRD analysis showed that alumina stabilized zirconia consisting of a tetragonal major phase and a minor phase of monoclinic. Based on the XRD results, the addition of alumina to zirconia was expected to stabilize the tetragonal phase. Zirconia with a tetragonal crystal structure particularly is expected having good mechanical properties that identical with formation of a short fiber system in nanofilled composite compared to the other two crystal structures of monoclinic and cubic.

3.2 Microvickers Hardness Test Results

Hardness value of nanofilled composite (sample A) with filler and matrix ratios of about 60:40 (A1), 70:30 (A2), and manufactured composite (sample B) are shown in Table 1.

Table 1. Hardness Value for Group Samples A1, A2, and B

Sample	Average Hardness Values (VHN)
A1	8,4
A2	24,38
B	27,48

Hardness value of the resin composite used in dentistry ranging were ranging 30-90 VHN [16]. Based on this, the synthetic nanofilled composite has not been proper for use as a filler for dental composite because the average hardness value was 24.38 VHN. However, the value was already approaching the hardness value of manufacture-made composite. In this study, the hardness value was 27.48 VHN.

The low hardness value of the synthetic nanofilled composite might be caused by homogenous particle sizes on the synthetic nanofilled composite. The homogenous particle sizes cause the formation of inter-particle space. The existence of unallocated space causes the mechanical properties of the composite particles will decrease. Other possible causes of the low hardness value of the synthetic nanofilled composite occurred when the process of mixing the filler and matrix was not homogeneous. The process of mixing matrix and filler on manufacture-made composite is made by automatic machine [17], while the research was conducted manually in the laboratory scale so that the filler and matrix mixing processes should have approached the optimal result.

3.3 Characterization Results Using Scanning Electron Microscopy (SEM)

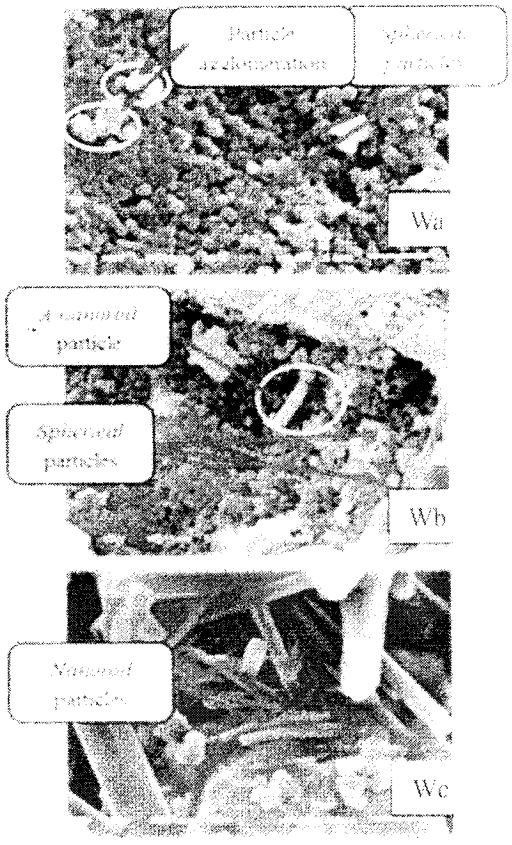

Figure 4. SEM characterization results on white carbon black powder. (Wa) Before calcined, (Wb) Calcined at a temperature of 500°C for two hours, and (Wc) Calcined at a temperature of 500°C for one hour then continued at a temperature of 750°c for one hour.

Characterization result using SEM on white carbon black powder is shown in Figure 4. Based on SEM characterization results of white carbon black powder

before calcined showed that the particles formed in the spherical shape with a particle size of around 39-114 nm. The results of SEM characterization on white carbon black powder calcined in the temperature of 500 °C for two hours showed that the nanorod particles began to form but was still dominated by the spherical shape, whereas the white carbon black powder calcined in a temperature of 500 °C for one hour followed by a temperature of 750 °C for one hour showed that in general the nanorod particles had formed the smallest diameter up to 35 nm.

SEM characterization results of white carbon black powder showed rod shape in the form of nano-sized particles (nanorod) with the smallest diameter up to 39 nm. The process of making white carbon black was carried out by adding 1% chitosan and 0.5% starch solution. Chitosan in this case acts as a surfactant that acts to cover the particle surface on the phase before calcination. Chitosan has amino (NH_2) groups and cations behavior (positive). This causes chitosan to become bioadhesive, which easily binds to the negatively charged surface. Na_2SiO_3 hydrolysis reaction produces Si(OH) which is negative (anion) due to the OH- group. The surface of the negatively charged particles will soon be covered by the positively charged chitosan after the addition of chitosan. This led to the growth of particles to be restricted so that the result of particle size was small [10,18].

The addition of starch in this study acts as a soft template to direct the growth of the particles in the direction of the nanorod shape and stabilize the nanorod shape. Starch which is a cellulose polysaccharide type composed of two different polymers, namely 10-20% amylose and 80-90% amylopectin. Amylose has a linear structure so it can direct the growth of particles in the direction of the nanorod shape. In addition, the presence of amylopectin which is stable leads to stabilize the nanorod shape [14,19].

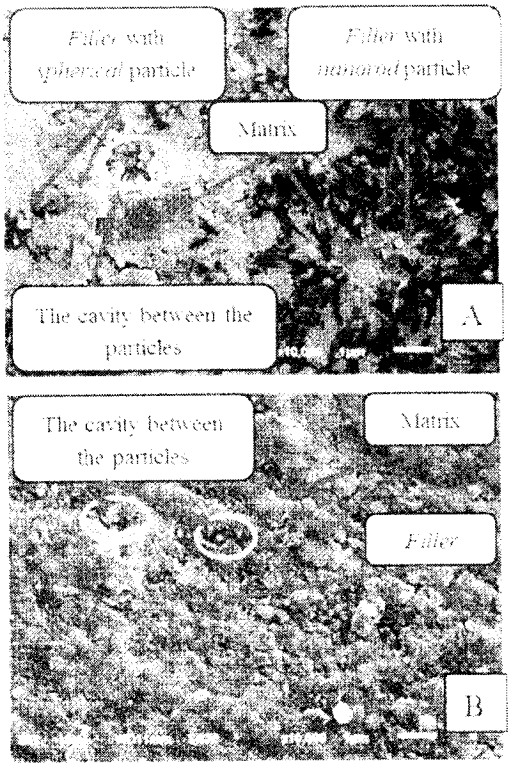

Figure 5. SEM characterization results (A) alumina stabilized zirconia-white carbon black nanofilled composite, (B) Manufacture-made composite.

SEM characterization results in alumina stabilized zirconia-white carbon black nanofilled composite (sample A) and manufacture-made composite (sample B) are shown in Figure 5.

Table 2. Comparison of results between the SEM characterization of nanofilled composite with the ratio of the filler and matrix about 70:30 and manufacture-made composite.

Sample	Morphology of particle	Particle Size (nm)	Average Particle Size (nm)
Nanofilled composite with the ratio of filler and matrix 70:30	Spherical and nanorod	57-241	149
Manufacture-made composite	Spherical	117-189	153

Based on SEM characterization results, some differences were found on alumina stabilized zirconia-white carbon black nano composite with the ratio of filler and matrix 70:30 after compared to the manufacture-made composite; and presented in Table 2.

In observations using SEM for the results of synthetic samples, alumina stabilized zirconia white carbon black with filler and matrix ratio of 70:30 was selected because it produces the average value of hardness greater than the sample with a ratio of filler and matrix of 60:40. This phenomenom might be caused by chitosan as a surfactant that less evenly covers the particle surfaces before calcination. Therefore, the particle size that formed becoming less homogeneous [10].

3.4 Interface between Composite and Polyethylene Fiber

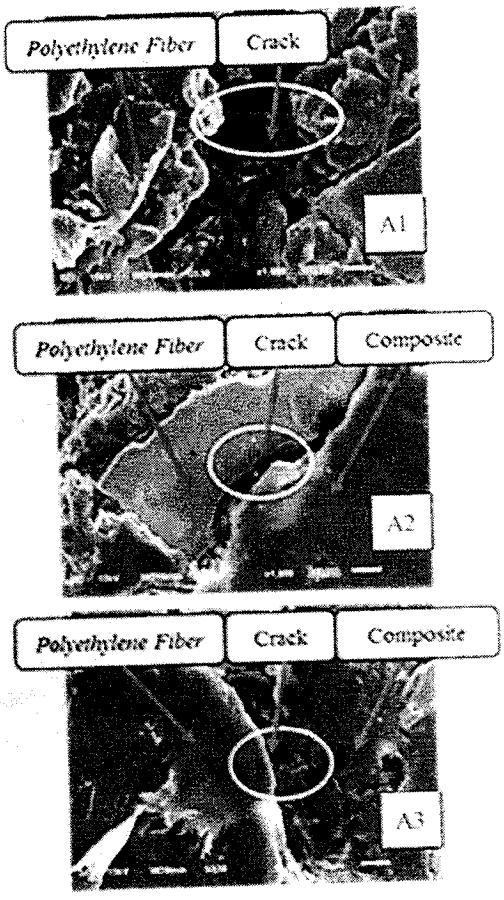

Figure 6. SEM characterization results of alumina stabilized zirconia-white carbon black nanofilled composite and polyethylene fiber with adhesive material (A1) 2% chitosan, (A2) 4% chitosan, and (A3) 6% chitosan.

The microstructures of Alumina stabilized zirconia-white carbon black dan polyethylene fiber sample with adhesive materials, 2% chitosan (A1), 4% chitosan (A2), and 6% chitosan (A3) are shown in Figure 6.

SEM characterization results in Figure 6 shows a big gap difference between the polyethylene fiber nano composite that previously smeared with 2% chitosan, 4% chitosan, and 6% chitosan. That huge gaps difference is presented in Table 3.

Table 3. Huge gaps difference on samples A1, A2, and A3.

Samples	Huge Gaps Between Nanocomposite and Polyethylene Fiber (µm)
A1	28.645
A2	2.665
A3	2.279

Sample of manufacture-made composite and polyethylene fiber with manufacture-made adhesif is shown in Figure 7.

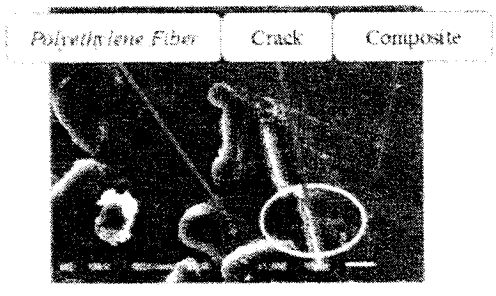

Figure 7. SEM characterization results of manufacture-made composite and polyethylene fiber with manufacture-made adhesive material.
Source: Primary Research Data, 2013

SEM characterization results in Figure 7 shows that between manufacture-made composite with polyethylene fiber that previously wetted with manufacture-made adhesive material had a crack which was around 0.600-0.825 µm, with average value of 0.7125 µm.

Synthetic nanofilled composite that reinforced with polyethylene fiber which wetted with 6% chitosan produced gaps that was smallest when compared to the use of 2% and 4 % chitosan. This is because chitosan that smeared on fiber bonded with the synthetic nanofilled composite. The higher concentration of chitosan, the more the amino group being open, the larger the ability of chitosan to bind [20].

Cracks found on the interface between the composite synthesis results and polyethylene fiber can be caused due to the lack of bonding between the functional groups of chitosan with the functional groups on polyethylene fiber, wherein the chitosan amino groups (NH_2) cannot bind with polyethylene fiber that has a functional group that CH_2. Therefore, both of them are positively charged. This can be fixed by adding a coupling agent from natural materials that are biocompatible and have suitable functional groups with chitosan and polyethylene fiber that can form a bond between them [10, 19, 21].

IV. CONCLUSION

Based on the results of this study, it can be concluded that:

1. The synthesized alumina stabilized zirconia consists of a tetragonal crystal structure as a major phase.
2. Microstructures of sample A show spherical and nanorod particles having an average size of about 149 nm. Whereas sample B has spherical morphologies with 153 nm in the average size.
3. Nanofilled composite (sample A) with alumina stabilized zirconia-white carbon black as nanofillers at the filler and matrix ratio of about 70:30 has a smaller particle size and better than manufacture-made composite.
4. The distance between nanofilled composite and polyethylene fiber with 6% chitosan adhesive material has the smallest gap size compared to the use of 2% and 4%, adhesive chitosan and is approaching the a gap size in the fiber reinforced manufacture-made composite.
5. Nanofilled composite at the filler and matrix ratio of 70:30 has a hardness value which is closed to the hardness value of manufacture-made composite.

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   b. Penulis bertanggung jawab penuh terhadap isi tulisan.
   c. Tulisan belum pernah diterbitkan di media cetak lainnya.

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      2) Tinjauan atau kajian ilmiah
   b. Jumlah halaman naskah maksimal 15 halaman, berjarak 1,5 spasi, dengan format: font Arial, Times New Roman, Helvetica atau Universe dengan ukuran 12. Untuk seluruh naskah semua jenis hurufnya harus sama. Judul tulisan diketik tebal (bold) dengan huruf besar font 14. Sub judul diketik tebal dengan huruf besar font 12.
   c. Naskah harus mengandung unsur-unsur sebagai berikut : judul, nama penulis, lembaga asal penulis, "abstract", "key word", pendahuluan, metode penelitian, hasil dan pembahasan, kesimpulan, saran (bila ada) daftar pustaka.
   d. "Abstract" ditulis dalam bahasa Inggris dan bahasa Indonesia. "Abstract" ditulis dengan huruf miring (italic) dengan jarak 1 spasi, maksimum terdiri atas 200 kata.
   e. Cara penulisan persamaan, formula, tata nama, satuan, harus mengikuti aturan baku yang telah lazim digunakan.
   f. Naskah disampaikan ke Dewan Redaksi dalam kertas ukuran A4, dan naskah ditulis dalam bentuk dua kolom berukuran 8 cm dengan jarak kolom 1 cm. Khusus untuk judul tulisan, nama penulis, lembaga asal penulis "abstract" dan "key word", hanya terdiri dari satu kolom.
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      Contoh : Roy W. Rice, *"Mechanical Properties of Ceramics and Composities"*, Marcel Dekker, Inc, New York, 2000.
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      Contoh : James V. Bothe, and Paul W. Brown, "Low Temperature synthesis of Porous NZP Ceramics", *J.Am. Ceram. Soc.* 91 [6] 2059-2063 (2008)
      Dari internet urutan penulisannya : Penulis, judul, http://www.tanggal.
      Contoh : Anonumous, Silicon Carbide, http://en.wikipedia.org/wiki/silicon carbide,2008
      Daftar pustaka disusun berurutan sesuai permunculannya dalam naskah atau sistem "end note".
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