Key Engineering Materials
ISSN: 1662-9795, Vol. 696, pp 74-79
doi:10.4028/www.scientific.net/KEM.696.74

Submitted: 2015-08-31
Revised: 2015-11-10
Accepted: 2015-12-29
Online: 2016-05-24

Hardness Evaluation of Dental Composite with Ceramic Fillers

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Keywords: Dental composite, zirconia filler, hardness

Abstract. In this study, new dental composites materials were developed. The two composites systems composed of zirconia (ZrO_2), alumina (Al_2O_3) and silica (SiO_2) (composites A) and zirconia (ZrO_2), calcium (CaO), and silica (SiO_2) (composites B) were synthesized through sol-gel method. These two systems were combined with urethane dimethacrylate and tetraethylene glycol dimethacrylate with 1% chitosan as coupling agent to build up the dental composites material. The resulting composites were subject to evaluation by microvickers hardness test and X-ray diffraction. The microvickers hardness test revealed that the hardness value for composites A and B were 24.48 and 21.9 VHN, respectively. Furthermore, the data were submitted to t-test (α=0,01) and it showed t count of both samples was 0,871 which means between the $-t_{1-½\alpha} < t < t_{1-½\alpha}$ thus showing statistically the same average hardness value of both samples. Eventually, the new dental composites could be anticipated to apply in dental composites filler. The hardness results support the XRD result revealed that tetragonal crystal phase will help the transformation toughening mechanism and cubical crystal phase of zirconium dioxide. Both of the crystal phases were formed to stabilize the zirconia.

Introduction

Dental composites materials have been develop as a restorative materials that could be used in biological tissue in term of appearance and functions [1]. The main components which developed the properties and characteristics of composites i.e polymerizable resin, filler, and the filler-resin interface [1]. Filler materials have several functions including enhancing modulus, hardness, strength and also translucency. Recently strontium glass, barium glass, quartz, borosilicate glass, ceramic, and silica particles have been as filler particles [2,3,4,5]. These fillers materials will affect to the resultant properties and characteristics of composites resin [6].

Zirconia is a crystalline dioxide of zirconium. The development of zirconia as ceramic material has increased along with the development of the dentistry technology and the need of material that required a high strength and aesthetic appearance. The other benefit is to avoid the use of certain metals that generally cause allergies in an effort toward metal-free dentistry [7,8]. The mechanical properties of zirconia are similar to those of metals (stainless steel). This particles also could protect the structure against crack propagation. Recently, the development of the combination of zirconia with other elements as raw materials in dentistry become gaining interest [9]. The addition of the other minerals will make tetragonal form more stable and be more effective in inhibit and seals crack propagation, Started in the late of 1980s, ceramic engineers learned to stabilize the tetragonal form of zirconium oxide at room temperature by adding small amounts (3–8 mass %) of calcium.

Other minerals that could be used as stabilizers are alumina, magnesium, and yttrium, or cerium [8,10,11,12]. Furthermore, the optical properties of zirconia need to be improve due to its poor translucency properties [9,13]. Thus, in order to maintain the aesthetic natural appearance, silica is combined with zirconia to improve the translucency characteristics. Moreover, there are some restorative material products using silica nanofiller or zirconia/silica nanocluster, while others contain a blend of a proprietary inorganic bariumalumino fluoroborosilicate (BAFG) glass with nanosized silicon dioxide particles to enhance the translucent shades of the restorative materials [14,15,16].

Hardness of materials is one of mechanical properties which frequently used as a parameter in order to evaluate the surface resistance of materials due to plastic deformation by penetration [6,17]. Synthesized filler could added in order to increase hardness as mechanical properties of dental composite.

In this study, two systems of zirconia (ZrO_2), alumina (Al_2O_3) and silica (SiO_2) (system A) and zirconia (ZrO_2), calcium (CaO), and silica (SiO_2) (system B) were synthesized through sol-gel method. The resulting composites were subject to evaluation by microvickers hardness test and X-ray diffraction to evaluate the hardness properties and crystallography characteristics of these dental material filler.

Materials and Method

Synthesis of ZrO_2-Al_2O_3-SiO_2 (Composites A). Zirconium chloride, aluminum nitrate and tetraethyl orthosillicate (TEOS) (60:20:30 % mol) were dissolved in 100 ml distilled water. 1% w/v Chitosan and starch were also added to the solution and mixed for 30 minutes. The solution was homogenized by ultra stirring. After that, the solution was then heated from 80°C until became char flakes. The char flakes thus produced was grinded using mortar and pestle then calcined at 900 °C for 2 hours. The resulting powder was then cooled and grinded again. Eventually, ethanol was added into the powder and followed by homogenization using ultrasonic homogenizer for 10 minutes.

Synthesis of ZrO_2-CaO-SiO_2 (Composites B). Synthesis of ZrO_2-CaO-SiO_2 were conducted using the aforementioned procedure for the preparation of ZrO_2-Al_2O_3-SiO_2, with different percentage of components (ZrO_2:CaO:SiO_2 = 60%:10%:30%)

Preparation of filler coating. Each ceramic filler system of ZrO_2-Al_2O_3-SiO_2 and ZrO_2-CaO-SiO_2 were mixed with 1% w/v chitosan solution until all filler particles mixed homogeneously. Furthermore, the composites were homogenized using ultrasonic bath for 30 minutes. The composites were dried in an oven at 70ºC for 10 hours. Further, the samples were then cooled and grinded using a mortar and pestle resulting ceramic powders.

Hardness Test. The hardness tests was conducted using microvickers hardness test with the calculation of the slopping surface of the indentation. The two diagonals of indentation left in the surface of the material after removal of the load were measured using microscope and their average calculated. Hardness test was done using LECO Brand Hardness Tester-Japan-M-400 H1/H2/ H3 with a load of 200 grams for 15 seconds. Prior to the hardness test, the samples were prepared. Firstly, urethane dimethacrylate (UDMA) was mixed with tetraethylene glycol dimethacrylate (TEGDMA) for 30 minutes. Furthermore, 2-(dimethylamino)ethyl methacrylate (DMAEMA) and camphorquinone were then added into the solution and then mixed it for 1 hour. The as-prepared filler was inserted gradually into the solution and stirred constantly until paste-like consistency was reached. Afterwards, the resulting paste was poured into 3 mm in height x 6 mm in diameter size of teflon mold which has been coated with Mylar strip on the bottom until 1.5 mm height, followed by cured it using light for 20 seconds and coated it again with Mylar strip on the top. Furthermore, the samples were then press until it became solid and cured it again with light for another 20 seconds. After the composite became fully hard, Mylar strip was taken out and cured it again for 20 seconds.

The composites were then removed from the mold and immersed them in distilled water at room temperature.

Results and Discussion

The hardness test results for ZrO_2-Al_2O_3-SiO_2 (Composites A) and ZrO_2-CaO-SiO_2 (Composites B) are summarized in Figure 1, respectively.

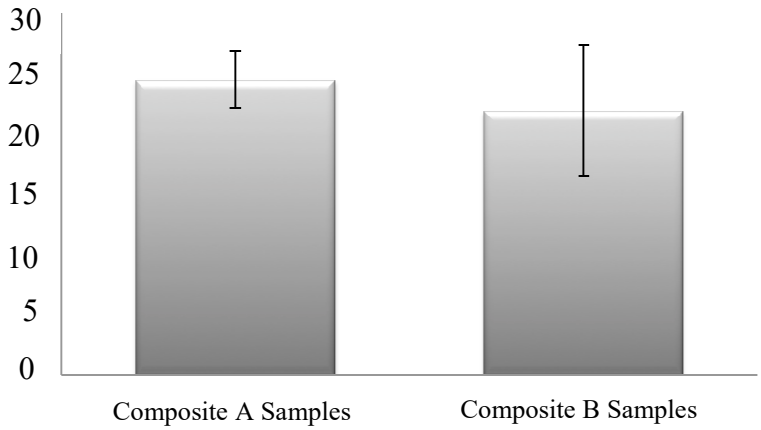

Fig 1. Comparison of Vickers hardness number results of both samples ($t_{count\ is\ between}\ t_{1-½\alpha} < t < t_{1-½\alpha}$)

As shown in Figure 1, the hardness value of ZrO_2-Al_2O_3-SiO_2 filler (Composites A) is higher than ZrO_2-CaO-SiO_2 filler (Composites B).

The hardness data were evaluated by statistical analysis using t-test compared to the tables for 0,01 significance level. The statistic result showed that t count of both samples was 0,871 which means between the $-t_{1-½\alpha} < t < t_{1-½\alpha}$ thus showing statistically the same average hardness value of both samples. Figure 1 shows comparison of Vickers hardness number results of both samples. The hardness data were evaluated by statistical analysis using t-test compared to the tables for 0,01 significance level. The statistic result showed that t count of both samples was 0,871 which means between the $-t_{1-½\alpha} < t < t_{1-½\alpha}$ thus showing statistically the same average hardness value of both samples.

In accordance with dental composites, the filler features an important role of hardness as its mechanical properties. This also in agreement with Moraes *et al.* and Poggio suggested that the size, shape, and content of fillers tends to affected the hardness; and all materials generally presented different results in comparison with one another. Filler content, with stable phase that will increase transformation toughening mechanism and good translucency, in resin composite is desirable. It has always been a subject of development in order to use it as the material under constant masticatory stresses, with better mechanical properties, and also successful aesthetic restorations [6,16,18].

The result was corresponding with the SEM and XRD characterization from the filler of those two different particle stabilizers [8].

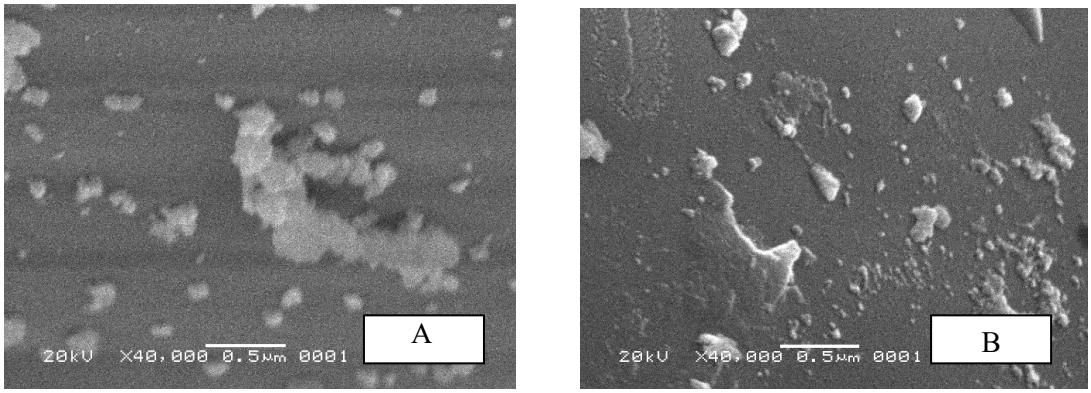

Fig 2. SEM Results of ZrO_2-Al_2O_3-SiO_2 (A) and ZrO_2-CaO-SiO_2 (B) Fillers [8]

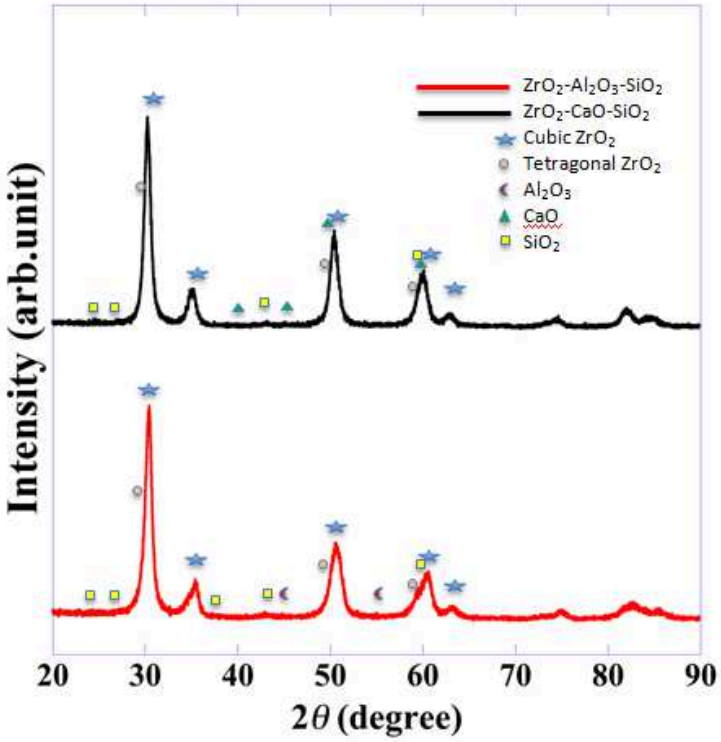

Fig 3. XRD Results of ZrO_2-Al_2O_3-SiO_2 and ZrO_2-CaO-SiO_2 Fillers

The SEM results on figure 2 showed that the particle size of both composites were successfully formed in nanometer size with minor agglomeration. XRD results on figure 3 revealed that the composites of ZrO_2-Al_2O_3-SiO_2 was successfully formed with the growing of tetragonal phase among the cubic phase, meanwhile the composites of ZrO_2-CaO-SiO_2 all formed in cubic phase. All of those composites had 6-12 nm variety crystal size. The ZrO_2-Al_2O_3-SiO_2 filler shows 8,6% intensity in tetragonal phase, which produced Partly Stabilized Zirconia (PSZ) that will help the toughening mechanism of crack shielding results from the controlled transformation of the metastable tetragonal phase to the stable monoclinic phase. Meanwhile, the powder sample of ZrO_2-CaO-SiO_2 filler almost formed all of cubic phase that produced Fully Stabilized Zirconia (FSZ) [8,19]. This cubic phase is resistant to most molten metals and thus used to make crucibles, but the combined of cubic and tetragonal phase results in a stronger material due to closure of cracks by expansion of the precipitates [20].

Conclusions

We fabricated new composites system composed by ceramic-based materials which could be developed as a dental composite filler. Based on the hardness test, the mean value of dental composite contains of ZrO_2-Al_2O_3-SiO_2 filler was higher than dental composite contains of ZrO_2-CaO-SiO_2 filler, even though there was no difference according to statistical calculations. Therefore, it can be concluded that the use of both systems (ZrO_2-Al_2O_3-SiO_2 and ZrO_2-CaO-SiO_2) are acceptable as dental composite filler.

Acknowledgements

We would like to thank Ministry of Research, Technology, and Higher Education, Directorate General of Higher Education, Republic of Indonesia whom funded the research through Padjadjaran University.

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