Comparison of Wear Resistance and Surface Hardness of Dentoform Tooth and Silane-treated Alumina Reinforced Epoxy Resin

การเปรียบเทียบความต้านทานการขัดสี และ ความแข็งผิว ของฟันเดนโตฟอร์ม กับ อีพอกซีเรซินที่เสริมความแข็งแรงด้วยผงอะลูมินาที่ผ่านการทำไซเลน

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ABSTRACT

The purpose of this study was to compare wear resistance and surface hardness of dentoform teeth (Frasaco, Nissin) and epoxy composite (70 wt% untreated alumina and 50, 60, 70 wt% silane-treated alumina reinforced epoxy resin). Ten specimens of each group (n=10) were examined for pin-on-disk wear resistance and Vickers surface hardness. One-Way ANOVA statistic analysis indicated that wear resistance of 70 wt% and 60 wt% silane-treated alumina reinforced epoxy resin group (tx 70% and tx 60%) were significantly higher than those of the others (26.11 $\pm$ 1.17 mm^3 and 27.35 $\pm$ 1.22 mm^3 respectively). On the other hand, the surface hardness of tx 70% (29.62 $\pm$ 1.17 HV) was significantly higher than that of tx 60% (24.08 $\pm$ 0.91 HV). It could be concluded that tx 70% should be further developed as substituted material for dentoform tooth.

บทคัดย่อ

การศึกษานี้มีวัตถุประสงค์เพื่อเปรียบเทียบความต้านทานการขัดสี และความแข็งผิว ของฟันเดนโตฟอร์ม (Frasaco และ Nissin) กับอีพอกซีคอมโพสิต (อีพอกซีเรซินที่เสริมความแข็งแรงด้วยผงอะลูมินาร้อยละ 70 และผงอะลูมินาที่ผ่านการทำไซเลนร้อยละ 50, 60, 70) ตัวอย่าง (กลุ่มละ 10 ชิ้น) ถูกทดสอบความต้านทานการขัดสีด้วยวิธี พินออนดิสก์ และความแข็งผิวแบบวิกเกอร์ส ใช้สถิติการวิเคราะห์ความแปรปรวนแบบทางเดียวในการวิเคราะห์ข้อมูล กำหนดนัยสำคัญที่ระดับ 0.05 ผลการศึกษาพบว่า ค่าความต้านทานการขัดสีของอีพอกซีเรซินที่เสริมความแข็งแรงด้วยผงอะลูมินาที่ผ่านการทำไซเลนร้อยละ 70 และร้อยละ 60 (26.11 $\pm$ 1.17 มม^3 และ 27.35 $\pm$ 1.22 มม^3 ตามลำดับ) สูงกว่าค่าความต้านทานการขัดสีของทุกกลุ่มทดลองอย่างมีนัยสำคัญทางสถิติ ในทางกลับกัน ค่าความแข็งผิวของอีพอกซีเรซินที่เสริมความแข็งแรงด้วยผงอะลูมินาที่ผ่านการทำไซเลนร้อยละ 70 (29.62 $\pm$ 1.17 HV) มีค่าสูงกว่าอีพอกซีเรซินที่เสริมความแข็งแรงด้วยผงอะลูมินาที่ผ่านการทำไซเลนร้อยละ 60 (24.08 $\pm$ 0.91 HV) อย่างมีนัยสำคัญทางสถิติ จึงสรุปได้ว่า อีพอกซีเรซินที่เสริมความแข็งแรงด้วยผงอะลูมินาที่ผ่านการทำไซเลนร้อยละ 70 ควรถูกพัฒนาต่อไปเพื่อเป็นวัสดุทดแทนฟันเดนโตฟอร์ม

Keywords: Epoxy resin, Silane-treated alumina, Dentoform tooth

คำสำคัญ: อีพอกซีเรซิน อลูมินาที่ผ่านการทำไซเลน ฟันเดนโตฟอร์ม

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Introduction

Dentoform teeth or plastic teeth are frequently used in the preclinical laboratory practice by dental students in several subjects such as prosthodontic and operative practices until they have skill enough to treat their patients. Dentoform teeth have many advantages for example uniform anatomy, unlimited availability, and ease of placement into a simulated dental arch. However, they have some limits such as their tactility is not similar to that of natural teeth, their dento-enamel junction (DEJ) is absent and their bonding ability to resin-based materials is limited. Therefore, it is necessary to practice on the natural teeth in some procedures. However, natural teeth are needed to be disinfected prior to use and limited available. In addition, arranging natural teeth in the dentoform arch is difficult to get proper occlusion (Frazier et al., 1999). Therefore, dentoform teeth are the favorable choice for education of dental students.

A large number of dentoform teeth are used each year and the price of the dentoform teeth increased continuously. The aim of this study was to develop the new substitute materials with quality resemblance in order to reduce import and education expenses of dental students.

According to previous study (Chamchong et al., 2010), the analysis of dentoform teeth components by nuclear magnetic resonance (NMR) technique found that Frasaco dentoform teeth (Frasaco®, Germany) comprised melamine as an essential component whereas Nissin dentoform teeth (Nissin dental products inc., Japan) was epoxy resin. Moreover, themogravimetric analysis (TGA) revealed that Frasaco dentoform teeth were composed of polymer approximately 40 wt% and fillers approximately 60 wt%. Alumina reinforced epoxy resin was chosen as substitute material and five experimental groups were studied including dentoform teeth, 50, 60, 70 wt% alumina reinforced epoxy resin, and polymethyl methacrylate (formatray®, Switzerland). It was shown that surface hardness of Frasaco dentoform teeth (57.04 HV) was the highest and surface hardness of alumina reinforced epoxy resin increased with increasing alumina content. Nevertheless, more than 70 wt% of alumina could not be used because the materials were unable to mix homogeneously.

The facts that main component of Nissin dentoform tooth is epoxy resin and 60 wt% fillers of Frasaco dentoform teeth are still an interesting issue. Epoxy resin had many usable qualities including low shrinkage, easy mold ability, chemical erosion resistance, good affinity to homogeneous materials, and thermal stability (Senthikumar et al., 2012; Ji et al., 2004) Mechanical properties and tribological behavior of epoxy resin can be improved with various materials such as glass or carbon fiber, Al_2O_3, SiO_2, TiO_2, and $CaSiO_3$ particle (Chamchong et al., 2010; Senthikumar et al., 2012; Ranganatha et al., 2013; Ramesh et al., 2014; Zhang et al., 2002; Wetzel et al., 2003; Chung et al., 2005; Kumar et al., 1998; Namie et al., 2011). However, fiber reinforcement was a cause of reduction in flowability of the composite (Ji et al., 2004). Therefore, using particle was a better choice for construction of dentoform tooth with casting method. When considering types of particle, Al_2O_3 was more appropriate for improving mechanical particle than any others because of its high surface hardness, availability and low price (Ramesh et al., 2014; Namie et al., 2011).

Silane coupling agent (SCA) is a substance being able to attach organic polymer to inorganic substrate such as glass, mineral filler, metal and metallic oxide. It creates stable bond between organic and inorganic substrate which is significantly improve properties of the composite (Michael et al., 2008). In addition, silane treatment on alumina particle provides low surface energy result in reduction of particle agglomeration and improvement of flowability (Jallo et al., 2010). From previous studies, silane treatment on alumina particle surface could increase mechanical and tribological properties of the composite and could help alumina particle to disperse homogeneously in epoxy resin (Kim et al., 2012; Yu et al., 2011; Rashid et al., 2011; Vassileva et al., 2006).

In this study, silane-treated alumina reinforced epoxy resin was chosen as substitute material for dentoform teeth. Wear resistance and surface hardness of these materials were evaluated.

Objective of the study

The purpose of this study was to compare wear resistance and surface hardness among dentoform teeth, silane-treated alumina and untreated alumina reinforced epoxy resin.

Materials and methods

Silicone mold fabrication

Dentoform teeth were cut by low speed diamond saw (Isomet 40003™, Buehler, USA) and polished by polishing machine (Ecomet 3™, Buehler, USA) to achieve a rectangular prism shape, 5x5x13 mm in size (Figure 1). Then, the rectangular prisms were attached on a tile at the distance of 1 cm between one another. The tile was boxed around by pink wax. Afterwards, silicone and its hardener were mixed in the vacuum mixer (Combination unit, Whip-Mix corporation, USA) for 1 minute. Silicone mixture was poured in the box and left for 2 days until it solidified. Then rectangular prisms were removed out of the silicone mold (Figure 2).

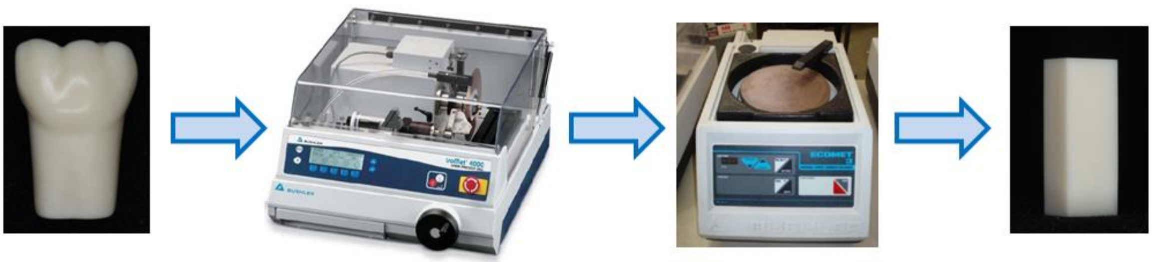


Figure 1 Dentoform teeth were trimmed and polished to a rectangular prism shape

a. The rectangular prism in boxing wax

b. Silicone

c. Vacuum mixer

d. Pour the silicone in boxing wax

e. Wait for the silicone completely solid about 2 days

f. Silicone mold

Figure 2 Silicone mold fabrication

Sample preparation

Silanization of alumina powder

Spherical-shaped alumina with mean particles size of 4.5 μm (Indal Calined Alumina, HTM30, Loxley, Thailand) was selected as a filler. 3-Aminopropyltriethoxysilane (APTES) (Sigma-Aldrich®, USA) was used for silanization of the alumina particle. The amount of APTES used was 0.3 wt% which was calculated from Arkle's equation to create monolayer of silane coating on the filler surface.

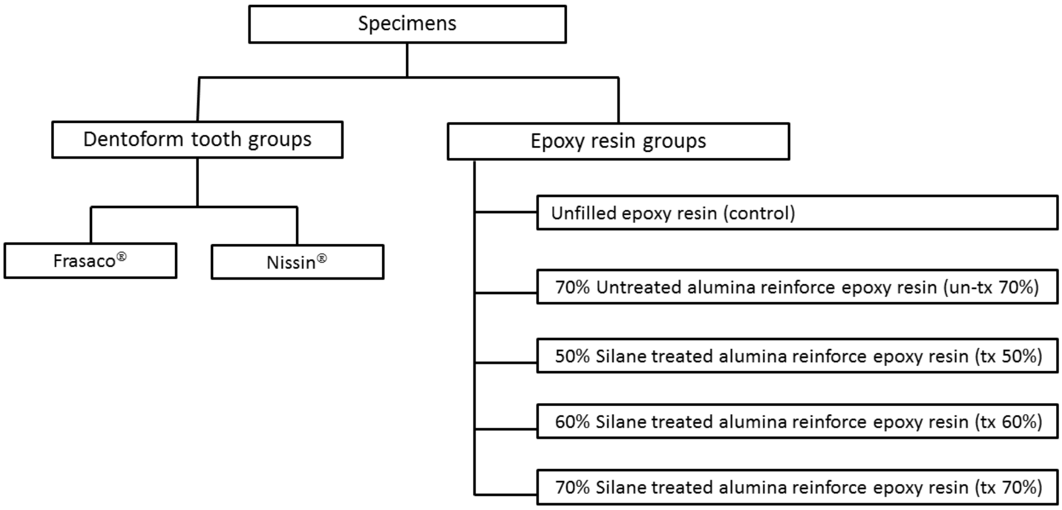
$$\text{Amount of silane (g)} = \frac{\text{Amount of filler (g) x Surface area } (m^2/g)}{\text{Minimum coating area of silane coupling agent } (m^2/g)}$$

Where: Surface area = 1 m^2/g

Minimum coating area of APTES = 353 m^2/g

0.3 g of APTES was added in the 70 vol% ethanol and stirred. These solutions were stored in a polyethylene cup with a cover and allowed to hydrolyze for 5 min. After that, 100 g of the alumina powders were added into APTES solution. The mixture was stirred until the solvent was evaporated entirely, dried at room temperature for 14 days and dried in the hot air oven at 50°C for 24 hr. Finally, silanization of alumina powders was verified by energy dispersive spectroscopy (EDS: EMAX x-act, Horiba, Japan)

Specimens were divided into 7 groups according to diagram as follows;



Fabrication of alumina reinforced epoxy resin

Epoxy resin was heated 70°C for reduce viscosity. Then, alumina powders were gradually added into epoxy resin and stirred until it was homogeneous. After that, the mixture was left to cool to 40°C. Hardener was added at an epoxy to hardener ratio of 2:1 and mixed all ingredients with vacuum mixer machine for 2 min. The mixture was poured into the silicone mold and left for 6 hr at room temperature and another 8 hr at 80°C for complete curing. Finally, the substitute materials were removed from the silicone mold and inspected. Inclusion criteria was specimens have no porosity on their entire surface and have a rectangular prism shape, 5x5x13 mm in size thoroughly when measured by digital vernier caliper.

Characterizations

Wear resistance test

The specimens were tested using pin-on-disc technique by pressing the specimens against a rotating counterpart which was sandpaper 280-grit (Buehler, USA) on the polishing machine (Ecomet 3™, Buehler, USA) at room temperature (Figure 3). In each test, load of 1 kg was applied and a rotation velocity of 0.12 m/s and testing time of 2 min were employed. Wear resistance was determined by calculating the volume loss of the specimens by measuring the reduction in length of the specimens after test and calculating the volume loss.

Figure 3 Specimen holder and polishing machine that used for wear resistance test

Surface hardness test

Vickers microhardness test was done by digital microhardness testing machine (FM-800, Future-Tech®, Japan). The diamond indenter pressed into the specimens under a load of 300 gf for 15 s. Each specimen was tested 3 times at the distance of at least 1 mm between each point (Figure 4). Then, diagonal lengths were measured and used to calculate Vickers hardness as follows:

$$HV = 1854.4 \times P/d^2$$

Where:

HV = Vickers hardness number (gf/ μm^2)

P = Force (gf)

d = Mean diagonal length of indentations, d1 and d2 (μm) (Figure 5)

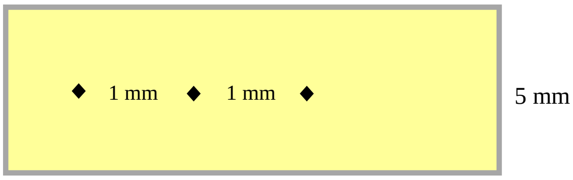


Figure 4 Indentation location in each specimen

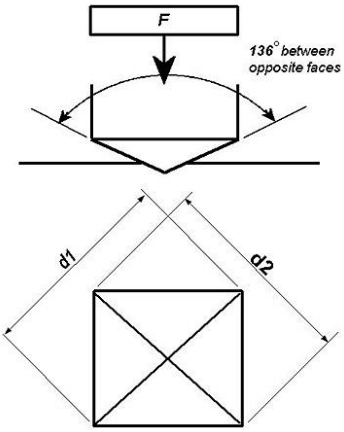


Figure 5 Determination of Vickers hardness

Results

Figure 6 and Figure 7 show the results of the wear volume loss and Vickers hardness in terms of the mean and standard deviation (X±SD). One-Way ANOVA and post hoc test (Bonferroni) were applied as statistical analysis of this study at p-value=0.05

Unfilled epoxy resin which was served as control group showed the largest volume loss among study groups indicating its lowest wear resistance. Tx 70% had higher wear resistance than that of un-tx 70% and was also significantly higher than those of dentoform groups and epoxy composite excepting tx 60%.

Unfilled epoxy resin had the lowest surface hardness. Whereas, the surface hardness values of both dentoform groups were significantly the highest among the study groups. However, within the epoxy composite groups, the surface hardness of tx 70% was significantly the highest.

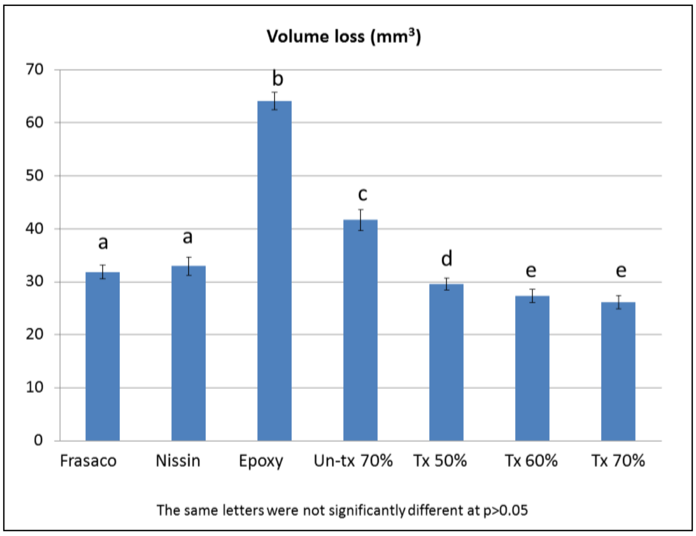


Figure 6 Comparison of volume loss of all experimental groups

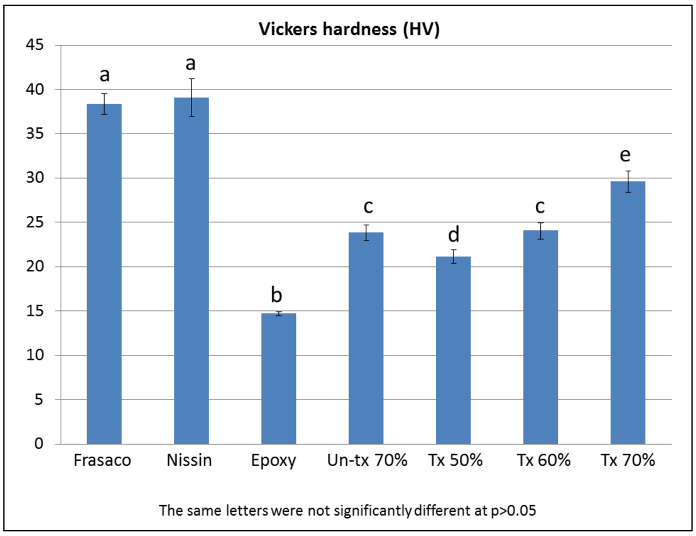


Figure 7 Comparison of Vickers hardness of all experimental groups

Discussion

The results in this study agreed well with the results shown previously that untreated alumina reinforced epoxy resin had the surface hardness lower than that of the dentoform teeth (Chamchong et al., 2010). The mechanical properties of alumina reinforce epoxy resin was improved by silane treatment with 3-Aminopropyltriethoxysilane (APTES). Consequently, wear resistance and surface hardness of the silane-treated alumina groups were comparable to the dentoform teeth groups. Statistical analysis showed significant differences among the groups in both wear resistance and surface hardness investigations.

From silanization point of view, there are various types of silane coupling agents (SCA) available. However, APTES was selected based on its functional group and application in the previous studies. APTES reaction had been accomplished by its silanol groups with the hydroxyl groups on the alumina surface, which thus formed covalent bonds. Moreover, APTES can chemically react via its amino groups with the epoxy groups of the epoxy resin (Vassileva et al., 2006). The silanization process in this study had been followed the protocol of previous studies (Chaijareenont et al., 2012). This wet technique was chosen because of easy manipulation and uniform coverage of filler particles. Amounts of APTES were selected based on the Arkle's equation, which were the minimum amount to create monolayer of APTES on alumina particle. It was shown previously that mechanical properties of composite can be improved by the silanized alumina when used the amount of SCA according to Arkle's equation. Moreover, the mechanical properties of materials had been reduced when using SCA over the amount calculated from the equation (Chaijareenont et al., 2012). In this study, EDS analysis showed silicon deposition on silanized alumina particle (data not shown) indicating that APTES effectively reacted with alumina particle.

Wear resistance of materials was characterized by their volume loss (Fig. 6). It means that low volume loss indicates high wear resistance. The result revealed that when increasing amount of alumina particle, wear resistance was increased. Wear resistance of un-tx 70% was 37% lower than tx 70%. Moreover, within epoxy composite groups, the wear resistance of tx 70% was significantly the greatest and even significantly higher than that of the dentoform teeth groups (frasaco® and Nissin®, about 18 and 20 % respectively). Therefore, silanization could improve wear resistance effectively. It might be related to the chemical bond between epoxy matrix and silanized alumina particles. This agrees with previous studies which revealed that wear resistance of silane-treated alumina reinforced epoxy resin was greater than that of untreated alumina reinforced epoxy resin (Kim et al., 2012; Vassileva et al., 2006).

Similar to several previous studies, the result of this study showed that when the proportion of alumina was increased, the surface hardness was comparatively increased as well (Senthikumar et al., 2012; Ranganatha et al., 2013; Chung et al., 2005; Namie et al., 2011). Nevertheless, more than 70 wt% of alumina could not be added even if alumina was treated with SCA, because of the materials were too high viscosity and difficult to mold. Unfortunately, surface hardness of silane-treated alumina reinforced epoxy resin has been not many studied and the result revealed that surface hardness of untreated alumina and silane-treated alumina reinforced epoxy resin were not significant difference. However, we found that the surface hardness of tx 70% was significantly greater than un-tx 70%. It conforms to previous study which showed that surface hardness of dental composites with silane-treated fillers is significantly greater than those of composite with untreated filler (McCabe et al., 1999).

From the result, the surface hardness of tx 70% was significantly the highest within the epoxy composite groups. Even if it was still lower than the dentoform teeth, but the wear resistance of tx 70% was significantly the greatest and was also significantly higher than those of dentoform groups.

Conclusion

From the results of this study, we can conclude that Tx 70% is a promising substituted material for dentoform teeth. Further study of this material at the application level should be evaluated in term of the satisfaction of dental student.

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