Original Article

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# Effects of cyclic acid challenge on the surface roughness of various flowable resin composites

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**Objective:** The purpose of this study was to investigate the effects of cyclic acid challenge on the surface roughness (Ra) of various flowable resin composites.

**Materials and methods:** For this study, G-ænial® Universal Injectable, Beautifil Injectable X, Filtek™ Z350XT Flowable Restorative, and Filtek™ Z350XT Universal Restorative resin composites (Shade A2) were used. Twenty disc-shaped specimens were prepared per group and randomly divided into - control group and cyclic acid treated group. After specimen preparation, the surface roughness of the control group was measured with a contact profilometer. For the acid treated group, the specimens were immersed in 0.5% citric acid (pH=2.3) for 1 minute then in distilled water for 1 minute with a 10-second media-to-media transfer time. This cycle was repeated for 1,095 cycles to simulate 1 year of clinical acid exposoure. Following which, the surface roughness of the specimens were measured. The surface of the composite resins were also observed using SEM.

**Results:** For the control groups, there was no significant difference in Ra value among the various control resin composites ($p$=0.179), except Beautifil Injectable X. The highest Ra value was observed with Beautifil Injectable X (0.0448μm). After the cyclic acid challenge, only Beautifil Injectable X (0.0926μm) presented a statistically significant difference in Ra value when compared to the control. No significant differences in Ra values were observed before and after the cyclic acid challenge with other resin composites. All specimens showed a relatively smooth surface topography with the control and acid treated conditions, except Beautifil Injectable X which presented a comparatively rougher surface.

**Conclusion:** Surface roughness of some flowable resin composites was impacted by cyclic acid challenge, especially Beautifil Injectable X.

**Keywords:** acid challenge, dental material, injectable, material testing, resin composite, surface roughness

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## Introduction

Resin composites have various applications in dentistry and several types have been made available by manufacturers. Flowable resin composite is an easy-to-use material contained with a single syringe which is injected into the required surface. The number of filler loads in resin composite is decreased in order to reduce viscosity and increase flow rate, thus, enhancing its adaptability [1]. Flowable resin-based composites are composed of 37-50% filler load by volume which is lower than conventional resin composites. This composition results in decreased mechanical properties and increased polymerization shrinkage [2]; thereby, making flowable resin composites more suitable for low-stress areas or conservative occlusal restoration [3]. Due to these inherent weaknesses, several attempts have been made to

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develop new flowable resin composites with increased strength. Beautifil injectable x is a recently introduced flowable resin composite which can be used to restore both anterior and posterior teeth, as claimed by its manufacturer. It is composed of bioactive nano S-PRG fillers. G-ænial® Universal Injectable is a flowable high-strength resin composite and is based on ultra-fine barium particles (150 nm), which are strongly bonded into the resin matrix by the manufacturer's full-coverage silane coating technology. It is claimed to have excellent polishability and gloss retention. However, most of these data have been retrieved from the manufacturers, and have never been confirmed by studies. As the properties of injectable resin composites, such as gloss retention, color stability, and surface roughness, are equal to those of flowable resin composite, it can be used for direct veneers of anterior tooth restoration using injectable technique.

Surface roughness is an important aspect of dental materials. Rough surface promotes bacterial adhesion, plaque formation, staining of restorations, secondary caries, gingival irritation, and plaque-induced gingivitis [4, 5]. Surface roughness >0.2 µm promoted more plaque formations, hence, increasing the risk of caries and periodontal disease [6]. The roughness of resin restorations are dependent on several factors such as filler size, percentage of surface area occupied by filler particles, hardness, degree of conversion of polymer to resin matrix and filler/matrix interaction, as well as stability of silane coupling agent [7, 8]. Physical factors, such as tooth brushing, and intrinsic and extrinsic chemical factors, such as HCl from systemic disease eg. GERD [9] and frequent consumption of acidic food and alcoholic beverages [10, 11], can also alter the surface of restorative materials. Acidic solutions is considered to increase surface roughness of resin composite due to demineralization of the resin matrix and, consequently, to the displacement of filler particles [10, 12, 13]. In 2011, a study observed the effect of several beverages on restorative materials. The results showed that the restorative materials tested with orange juice (pH=2.85) and mango juice (pH=3.49) had higher roughness and greater dissolution than other beverages [14]. Flowable resin composites show different clinical performance depending on the oral environments [11]. However, no study has yet compared the surface roughness of resin composites from different manufacturers after cyclic aging in an acidic solution. The purpose of this study was to investigate the effects of cyclic acid challenge on the surface roughness of various flowable resin composites.

## Materials and Methods

Three types of flowable resin composite and one conventional resin composite (Table 1), namely G-ænial® Universal Injectable (GC corp., USA), Beautifil Injectable X (Shofu Inc, Kyoto, Japan), Filtek™ Z350XT Flowable Restorative (3M ESPE, USA), and Filtek™ Z350XT Universal Restorative (3M ESPE, USA), were used in this study. All resin composites of Shade A2 were used. Twenty specimens of each resin composite were prepared and randomly divided into two groups: control group and cyclic acid treated group. After specimen preparation, the surface roughness of the control group was measured at baseline.

Table 1 Materials used in this study

| Materials | Composition | | Filler content (vol and wt%) | Lot |
|---|---|---|---|---|
| | Filler | Matrix | | |
| Filtek™ Z350XT Universal Restorative (3M ESPE, USA) | Zirconia/silica nanocluster silica particle | Bis-GMA Bis-EMA UDMA TEGDMA | 78.5 wt% (59.5% by volume) | N993826 |
| Filtek™ Z350XT Flowable Restorative (3M ESPE, USA) | 0.1-0.5 µm ytterbium triflouride 20, 75 nm silica 4-11 nm Zirconia | Bis-GMA UDMA TEGDMA | 65 wt% (46% by volume) | NA89645 |
| G-ænial® Universal Injectable (GC Corp., USA) | Silica 150nm Barium glass | Methacrylate monomer | 69 wt% | 1906141 |
| Beautifil Injectable X (Shofu Inc., Kyoto, Japan) | surface reaction type pre-reacted glass-ionomer (S-PRG) Aluminofluoro-borosilicate glass $Al_2O_3$ | Bis-GMA Bis-MPEPP TEGDMA | 64 wt% (42% by volume) | 91901 |

Specimen preparation was modified from a previous study [15]. Flowable resin composites were applied into disc-shaped plastic mold (5 mm in diameter, 2 mm height) covered with a Mylar matrix strip and placed between two glass slides (Figure 1). Following which, the specimen were light polymerized with a LED curing light unit (Bluephase N, Ivoclar Vivadent, Schaan, Liechtenstein) at 1,200 mW/cm$^2$ light intensity for 40 seconds per side of the specimen. For calibration, the intensity of the curing light unit was measured with a spectrophotometer (Bluephase® meter, Ivoclar Vivadent, Schaan, Liechtenstein) to confirm

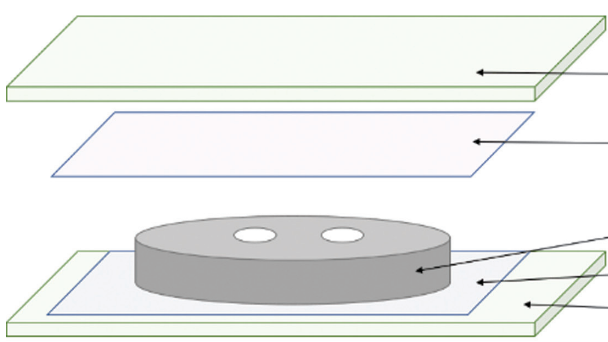


Figure 1 Specimen preparation

constant intensity. All prepared specimens were polished by hand with abrasive sandpaper of grid no. 800, 1200, 2000, and 2400, respectively, each grade of abrasive sandpaper was applied to the specimen under wet condition for 1 minute in linear motion then turn the specimen in perpendicular angle to the polishing line and repeated linear motion of polishing for another 1 minute, followed by ultrasonic cleansing for 10 minutes in distilled water before changing the grade of sandpaper. The samples were stored in distilled water at 37°C for 24 hours.

For the acid treated group, cyclic acid challenge was performed by a thermocycler (TC400, King Mongkut's Institute of Technology Ladkrabang, Bangkok, Thailand). Specimens were immersed in 0.5% citric acid (pH=2.3) for 1 minute [16, 17]. Subsequently, the specimens were then immersed in distilled water for another 1 minute with 10 second media-to-media transfer time. The cyclic acid challenge was performed for a total of 1,095 cycles to simulate 1 year of clinical acid exposure, as modified from a previous study [18].

This study evaluated average surface roughness (Ra) of the specimens before and after the cyclic acid challenge. The surface roughness of the prepared specimens were measured with a contact stylus profilometer (Talysurf series 2, Taylor Hobson Limited, Leicester, England) with a 2 μm diamond stylus employing a cutoff length of 0.25 mm, with a measuring length of 2.5 mm at a speed of 0.5 mm/s. For each specimen the measurement was performed at 3 areas starting from a center then shifting to x and y-axis as shown in (Figure 2). The data gathered from each group was then analyzed.

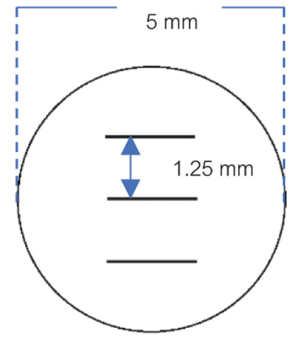


Figure 2 Surface roughness measurement process on a specimen 5 mm in length

The surface of the resin composites of the control and cylic acid treated group were also observed using SEM using secondary electron imaging mode (JSM-6610LV; JEOL Ltd., Tokyo, Japan) under 500x and 3000x magnification.

The means and standard deviations of Ra values of the restorative materials were calculated. The data were analyzed by two-way ANOVA for evaluating the effects of cyclic acid challenge on the surface roughness of the materials. Tukey's post hoc test was used for multiple comparisons. Statistical significance was established with level of significance $p \leq 0.05$.

## Results

The surface roughness of each specimen was represented by the Ra value as showed in (Table 2). Normal distribution and equality of variances of all data were analyzed with Kolmogorov-Smirnov test and Levene's test, respectively. Data were further analyzed with two-way ANOVA at 95% significant level. The analysis revealed a significant difference in surface roughness among tested resin

Table 2 Surface roughness measurement represented by Ra value (µm)

| Group | Mean (±SD) (µm) | |
|---|---|---|
| | Control | Cyclic acid treatment |
| Filtek™ Z350XT universal | 0.0287 (±0.0029) [a] | 0.0314 (±0.0054) [a] |
| Filtek™ Z350XT Flowable | 0.0327 (±0.0038) [a] | 0.0355 (±0.0071) [a, b] |
| G-ænial® Universal Injectable | 0.0351 (±0.0064) [a, b] | 0.0433 (±0.0063) [b, c] |
| Beautifil Injectable X | 0.0448 (±0.0055) [c] | 0.0926 (±0.0081) [d] |

Different letters indicate statistically significant difference at $p$=0.05.

composites ($p$<0.01). The surface roughness was affected by the cyclic acid challenge ($p$<0.01) and had an interaction between the two factors ($p$<0.01). Then, the data were analyzed by Tukey's post hoc test for multiple comparisons. For the control groups, there was no statistically significant difference in Ra value among the control resin composites ($p$=0.179), except control Beautifil Injectable X ($p$>0.05). The highest Ra value was observed in Beautifil Injectable X (0.0448 µm). After the cyclic acid challenge, only Beautifil Injectable X (0.0926 µm) presented a significant difference in surface roughness when compared to the control. However, no statistically significant difference was found between before and after the acid challenge in other resin composites.

The results from SEM observations at 500x and 3000x magnifications revealed surface irregularities (Figure 3) corresponding to the results of the surface roughness test. In general, the representative surfaces of the resin composites revealed observable differences from each other. All specimens showed a relatively smooth surface topography of the control and acid treated groups, except Beautifil Injectable X which presented a comparatively rougher surface. Scratch lines from abrasive sandpapers could be observed in all groups. Beautifil Injectable X group demonstrated greater surface irregularities and porosities when compared to the control group.

## Discussion

Generally, the properties of most restorative materials can be altered by temperature changes and the pH of the environment [19]. The surface characteristics of the restorative materials, such as roughness, determine the clinical outcomes and performance of restorative materials during restorative procedures [19]. The roughness and irregularities of restorative surface tend to increase plaque accumulation which causes gingival irritation, decreased esthetic outcomes, and reduced longevity of the restorations [19, 20]. Moreover, wear resistance and surface roughness in the oral environment are important criteria to determine and predict the clinical degeneration of restorative materials [19, 20]. Surface roughness can be measured by using contact or non-contact profilometer. The parameter used to represent the surface roughness in this study was Ra which was obtained from a contact profilometer. It is suited for glossy materials as the ones tested

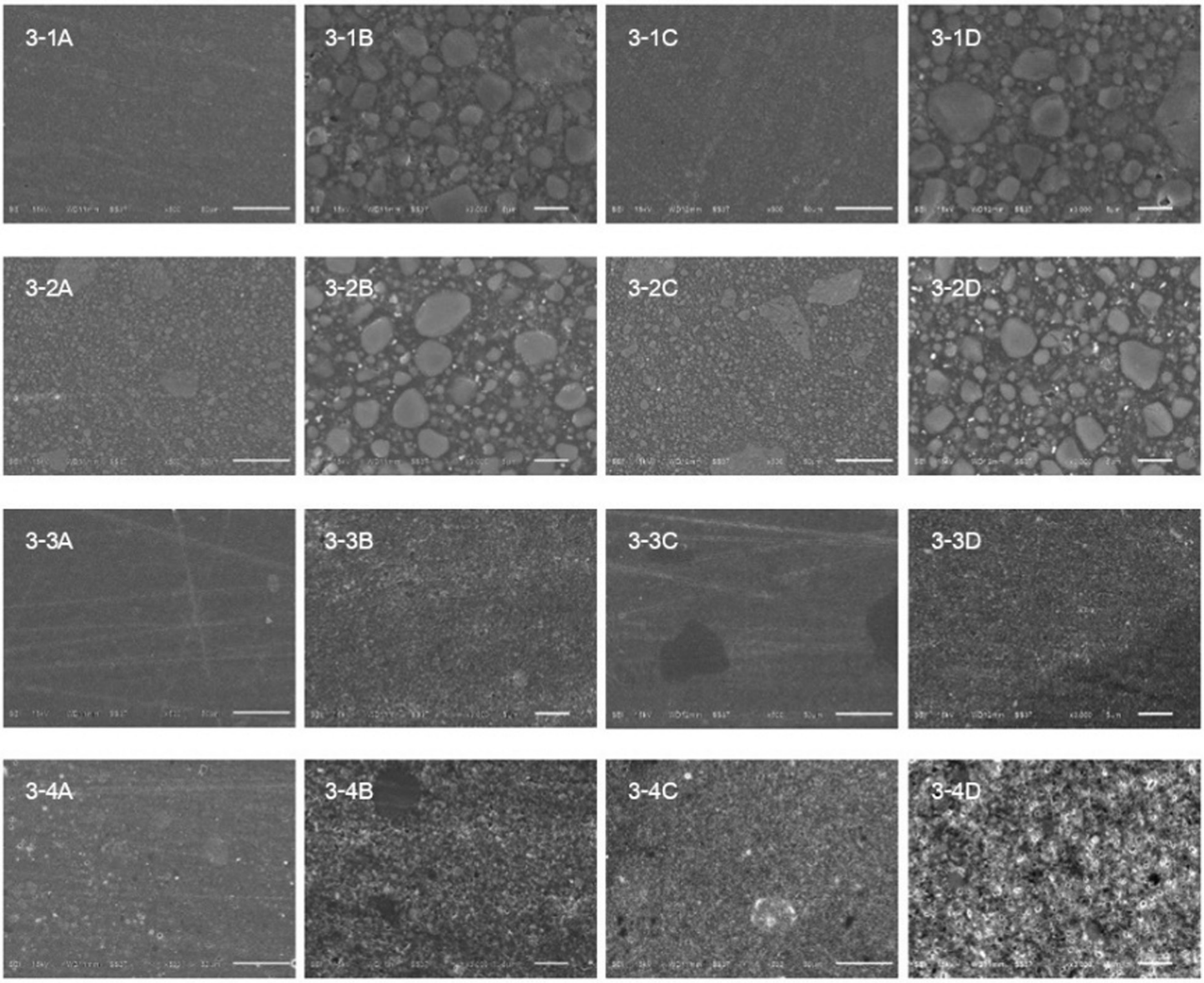


Figure 3 Representative SEM images of Filtek™ Z350XT Universal Restorative (3-1), Filtek™ Z350XT flowable Restorative (3-2), G-ænial® Universal Injectable (3-3), and Beautifil Injectable X (3-4); with (A) control group at 500x, (B) control group at 3000x, (C) cyclic acid treated group at 500x, and (D) cyclic acid treated group at 3000x

in this study as the surface roughness is difficult to measure with a non-contact profilometer owing to the light scattering effect of shiny surfaces. Besides carious lesions, acidic erosion can also occur over time [21]. Acidic food and beverage not only affect tooth structure but also restorative materials, which relate to their properties [22]. Recommendations made in a guideline from the US Food and Drug Administration was used to select the solution for the cyclic acid challenge [23]. As food simulators, citric acid is found in citrus fruits, juices, and beverages. The pH value of 0.5% citric acid used in this study (pH=2.3) is representative of the common pH of most soft drinks and acidic beverages [16, 24]. This study was designed to stimulate the intermittent acidic conditions that can occur inside the oral cavity. The cyclic acid challenge represented acidic food or beverage consumption thrice a day for a cumulative time of 1 year, as modified from previous studies [16, 17, 18]. To standardize the specimens, abrasive sandpapers were used in series of grit no. 800, 1200, 2000, and 2400, respectively, to produce a smooth surface across all specimens and represented polished restorations in a clinical situation. The polished surfaces with the filler-exposed areas resulted in a greater degree of roughness, especially Beautifil Injectable X. This consequence corresponded to its larger average particle sizes (0.8 µm) as compared to smaller average particle sizes (0.1-0.5 µm) found in G-ænial® Universal Injectable, Filtek™ Z350XT Universal Restorative, and Filtek™ Z350XT flowable Restorative [23]. In this study, the result showed that Beautifil Injectable X had the highest surface roughness when compared to other restorative materials, even in the control and cyclic acid treated groups. Furthermore, only the surface roughness of Beautifil Injectable X was affected by the cyclic acid challenge. The results found that the Ra of Filtek™ Z350XT Flowable, G-ænial® Universal Injectable, and Filtek™ Z350XT universal in cyclic acid treated groups were not different from the controls. As the restorative materials used in this study were not exposed to mechanical forces, any changes that were observed could be attributed to chemical dissolution. Surface roughness was directly dependant on the structure of the resin matrix, coupling agent, and characteristic of filler particles [25]. Citric acid has been known to be destructive to dental hard tissues and resin-based restorative materials [26]. In a low pH situation, the matrix of the restorative material gradually dissolves due to the influence of the acid, along with any unstable glass particles [26]. High acidity might have a greater softening effect on the resin matrix, consequently promoting the dislodgement and elution of filler particles and reducing the mechanical properties of restorative materials [26]. Many studies have reported that acidic solutions (pH=2.67-3.79) increased the surface roughness of resin composite. Due to the acidity of the solutions, the surface hardness may be decreased because of the demineralization of the resin matrix, resulting in the displacement of filler particles and eventual formation of a rough surface [10, 12, 13]. In comparison to giomer, resin composite was found to be less affected by low pH beverages or acid solution [26]. For this study, Beautifil Injectable X was the only material composed of bioactive nano S-PRG (Surface Pre-Reacted Glass Ionomer) which could have resulted in greater filler dissolution than any other material, as the fluorosilicate glass filler of this material has greater susceptibility to degradation by weak acids [27]. Beautifil Injectable X had many glass filler exposed on the restorative surfaces. The glass fillers were easily attacked by $H^+$ ion from citric acid then released ion ($Al^{3+}$ and $Ca^{2+}$) to form citrate complex which also could be continuously dissolved by acid [28]. This dissolution process caused more surface roughness in giomer. Moreover, all materials in this study contained bis-GMA as part of their resin matrix. When it absorbed water, silane coupling agents were induced to cause hydrolysis and loss of chemical bond between fillers and the resin matrix [29]. Consequently, the surface roughness of Beautifil Injectable X, which contained both fluorosilicate glass fillers and bis-GMA, was observed to be more affected by the cyclic acid challenge as compared to the others [26]. The results of a previous study that observed the effects of acidic drink on the surface roughness of flowable composite showed that the surface roughness of flowable composite was significantly increased [11]. However, the previous study operated the acid challenge by continuously soaking all specimens in acidic drinks (pH=2.55 and 4.15) for 14 days. In contrast, the cyclic acid challenge in this study was carried out in intermittent cycles between distilled water and acidic solution. No significant increase in surface roughness was observed after cyclic acid challenge for Filtek™ Z350XT Universal Restorative, Filtek™ Z350XT flowable Restorative, and G-ænial® Universal Injectable. This might be due to the time required for the acid to degrade the filler-resin matrix. When compare between the injectable resin composite and conventional composite, higher surface roughness was observed in the injectable resin composite without statistical difference. The injectable resin composite is a flowable high-strength resin composite with injectable application. Due to their composition, the injectable resin composite composed of ultra-fine barium particles (150 nm), which highly loaded up to 61 to 71 wt%. However, the fiilers in the injectable composite were lesser in size and quantity than the conventional resin composite. Their properties might not be equal to the conventional type [30]. Therefore, it can be considered that not only the concentration, pH, and type of the acidic solution but also the frequency and continuation of acid challenge, can affect the surface roughness of restorative materials [31].

Practically, the intraoral situation could not be completely imitated because of the limitations of in-vitro experimental conditions. According to the results of the present study, conventional composite, flowable composite, and injectable resin composite were all able to survive the acid challenges. Despite the fluoride-releasing ability of giomer and its help in reducing secondary carious lesion under the restoration, dental practioners are recommended to consider resin composites as alternative materials for use in esthetic areas as well as in patients with dental erosion due to their acid-resistant properties. Nevertheless, further evaluation is still recommended for long-term clinical observation and in-vitro performances.

## Conclusion

Within the limitations of this in-vitro study, it can be concluded that the surface roughness of various flowable resin composites was impacted by cyclic acid challenge, especially for Beautifil Injectable X.

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