

Research Report

Shear bond strength of self-adhering flowable composite on dentin surface as a result of scrubbing pressure and duration

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ABSTRACT

Background: Self-adhering flowable composite is a combination of composite resin and adhesive material. Its application needs scrubbing process on the dentin surface, but sometimes it is difficult to determine the pressure and duration of scrubbing. **Purpose:** This study was aimed to analyze the effect of scrubbing pressure and duration on shear bond strength of self-adhering flowable composite to dentin surface. **Methods:** Fifty four mandibulary third molar were cut to get the dentin surface and divided into nine groups ($n = 6$). Dentin surface was scrubbed with 1, 2, and 3 grams of scrubbing pressure, each for 15, 20, and 25 seconds respectively. Composite resin was applied incrementally and polymerized for 20 seconds. All specimens were immersed in saline solution at 37° C for 24 hours. Shear bond strength was tested for all specimens by using Universal Testing Machine (Shimadzu AG-5000E, Japan) at a crosshead speed of 1 mm/minute and analyzed by ANOVA and Post Hoc Test Bonferonni. The interface between self-adhering flowable composite and dentin was observed with a Scanning Electron Microscope (JEOL JSM 6510LA). **Results:** The highest shear bond strength was obtained by 3 grams scrubbing pressure for 25 seconds or equal to applying the brush applicator in 0° relative to dentin surface. **Conclusion:** Increasing the scrubbing pressure and duration will increase the shear bond strength of self-adhering flowable composite resin to dentinal surface. The highest shear bond strength was obtained when the applicator in 0° relative to dentin surface.

Key words: Scrubbing technique, shear bond strength, self-adhering flowable composite

ABSTRAK

Latar belakang: Self-adhering flowable composite merupakan gabungan resin komposit dengan material adhesif yang dalam penggunaannya memerlukan teknik scrubbing pada permukaan dentin, namun sulit untuk menentukan besar tekanan yang tepat saat scrubbing. **Tujuan:** Tujuan dari penelitian ini adalah untuk menganalisa pengaruh tekanan dan lama scrubbing saat aplikasi self-adhering flowable composite terhadap shear bond strength pada permukaan dentin. **Metode:** 54 gigi molar-3 rahang bawah dipotong sampai permukaan dentin dan dibagi atas 9 kelompok ($n = 6$). Scrubbing pada permukaan dentin dilakukan dengan besar tekanan 1, 2, dan 3 gram, masing-masing selama 15, 20, dan 25 detik. Resin komposit diaplikasikan secara inkremental dan dipolimerisasi dengan sinar selama 20 detik. Spesimen direndam dalam larutan salin dan disimpan dalam inkubator pada suhu 37° C selama 24 jam. Shear bond strength diuji menggunakan Universal Testing Machine. Analisa data melalui uji ANOVA diikuti Post Hoc Test Bonferonni. Antarmuka self-adhering flowable composite resin dan dentin diamati melalui Scanning Electron Microscope (SEM). **Hasil:** Shear bond strength tertinggi antara self-adhering flowable composite resin dan dentin diperoleh pada tekanan scrubbing 3 gram selama 25 detik atau kuas membentuk sudut 0° dengan permukaan dentin. **Kesimpulan:** Semakin besar tekanan dan lama waktu scrubbing akan meningkatkan shear bond strength material self-adhering flowable composite resin terhadap permukaan dentin. Kekuatan paling tinggi diperoleh bila kuas aplikator membentuk sudut 0° dengan permukaan dentin selama proses scrubbing.

Kata kunci: Teknik scrubbing, shear bond strength, self-adhering flowable composite

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INTRODUCTION

The latest adhesive system on dentin surface is a combination of etchant, primer, and bonding agent in one product or one step application.¹ Self-adhering flowable composite that combines the resin technology of composite and adhesive has also developed into one product. This self-adhering flowable composite eliminates the need for a separate bonding application step with composite for direct restorative procedures. This material contain of glycerol phosphate dimethacrylate (GPDM) that act as an etchant and dentin bonding agent in the same time. The bonding to dentin is a result of chemical reaction between phosphate group and calcium ion in tooth structure and mechanically as a result of hybrid layer formation. Self-adhering flowable composite is indicated for small class I and base or liner for class I and II restorations. Additional indications include pit and fissure sealant, repair of enamel defects, blocking of undercuts, incisal abrasions, and porcelain repairs. The application of this material on the dentin surface needs scrubbing process with moderate pressure using brush applicator.² The application of self-etch adhesive materials with scrubbing technique on dentin surface will increase the bonding strength to dentin surface by modified the smear layer, and increase chemical interaction between self-adhesive materials and dentin.^{3–5} The same reason expected in application of self-adhering flowable composite. However practically it is hard to determine the pressure and duration of scrubbing process using brush applicator to reach the optimal bonding strength.

The objective of this study were to analyze the effect of scrubbing pressure and duration on shear bond strength of self-adhering flowable composite to dentin surface and that, to develop a guideline of brush applicator usage to obtain the appropriate scrubbing pressure.

MATERIALS AND METHODS

Fifty four mandibulary third molars were obtained under a protocol approved by the Faculty of Dentistry's Ethics Committee at Universitas Indonesia. All specimens were sectioned to reach the dentin surface and embedded in resin with the dentin surfaces on the outside. The dentin surface was ground with a mechanical grinder (Struer LaboPol, Denmark) and polished with wet silicon carbide paper

#600 to obtain a flat surface. All specimens were observed under a light optical stereo microscope to ensure the dentin cleanness with no defect. A plastic sheet with 5 mm in diameter was applied on dentin surface and surrounding dentin surface was varnished with nail polish to border the tested area. Thereafter a gelatin ring with same diameter was applied encircle the tested area.⁶

Specimens were randomly divided into nine groups ($n = 6$). Self-adhering flowable composite (Table 1) was applied on dentin surface by scrubbing technique for 1, 2, and 3 grams of scrubbing pressure each for 15, 20, and 25 seconds. The scrubbing pressure was controlled by digital balance. According to the previews study, applying the brush applicator with an angle of 0 degree relative to dentin surface was equal to 3 grams, 30 degrees relative to dentin surface equal to 2 grams, and 60 degrees relative to dentin surface equal to 1 gram.

Polymerization of self-adhering flowable composite was done using LED MAX Hilux 450 (Benlioglu, Turkey) with intensity of 600 mWcm^{-2} for 20 seconds. Composite resin (Filtex Z350, 3M ESPE) was applied incrementally into the gelatin ring and polymerized for 20 seconds. All specimens were then immersed in saline solution at 37°C for 24 hours. Shear bond strength was tested for all specimens with an Universal Testing Machine (Shimadzu AG-5000E, Japan) at a crosshead speed of 1 mm/minute. The shear bond strength data were statistically analyzed by one-way ANOVA and Post Hoc Test Bonferonni. Thereafter by two-way ANOVA for analyze the interaction between groups.

The surface fracture area of specimens were sectioned and immersed in HCl 37% for 30 seconds to demineralized the dentin, then immersed in NaOCl 1% for 10 minutes to dissolve the demineralized matrix dentin.^{7,8} Thereafter the interface between self-adhering flowable composite and dentin was observed with a Scanning Electron Microscope (JEOL JSM 6510LA) at magnification of $1000\times$.

RESULTS

The mean value of shear bond strength of self-adhering flowable composite (Dyad Flow, Kerr) to dentin surface with scrubbing pressure 1, 2, and 3 grams each for 15, 20, and 25 seconds are shown in Table 2.

Table 1. Composition and application procedures of self-adhering flowable composite

Brand	Composition	pH	Procedures
Dyad Flow (Kerr)	GPDM (Glycerol Phosphate dimethacrylate), Barium glass filler, Colloidal Silica, Ytterbium Fluoride	1.9	Use provide brush to apply Dyad Flow with moderate pressure for 15–20 seconds to obtain a thin layer (< 0.5 mm). Light cure for 20 seconds

Source: Manual product of Dyad Flow, Kerr

Table 2. Mean shear bond strength of self-adhering flowable composite to dentine surface based on scrubbing pressure and duration (MPa)

Scrubbing Pressure	Scrubbing duration		
	15 sec	20 sec	25 sec
1 g	3.30 ± 0.92	3.70 ± 1.19	4.33 ± 1.66
2 g	3.77 ± 1.47	4.36 ± 1.44	5.23 ± 1.25
3 g	4.99 ± 0.82	5.25 ± 2.04	7.22 ± 0.68

It was showed that the shear bond strength increased simultaneously with scrubbing pressure and duration.

Statistically showed the normal data distribution, therefore the analysis continue by two-way ANOVA that showed the differences between scrubbing pressure groups and between scrubbing duration groups.

The statistic analysis was supported by the SEM images of morphology between dentin surface and self-adhering flowable composite with scrubbing pressure 1, 2, and 3 grams each for 15, 20, and 25 seconds. It was showed that the formation of hybrid layer gradually appears in line with increasing in scrubbing pressure and duration (Figure1, 2, 3).

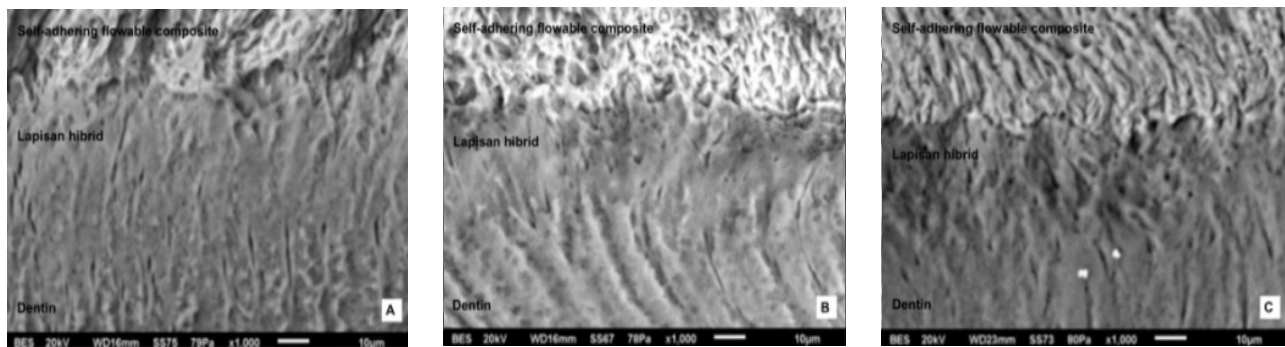


Figure 1. Morphology interface between self-adhering flowable composite and dentin surface. A) 1 g scrubbing pressure for 15 sec; B) 1 g scrubbing pressure for 20 sec; C) 1 g scrubbing pressure for 25 sec.

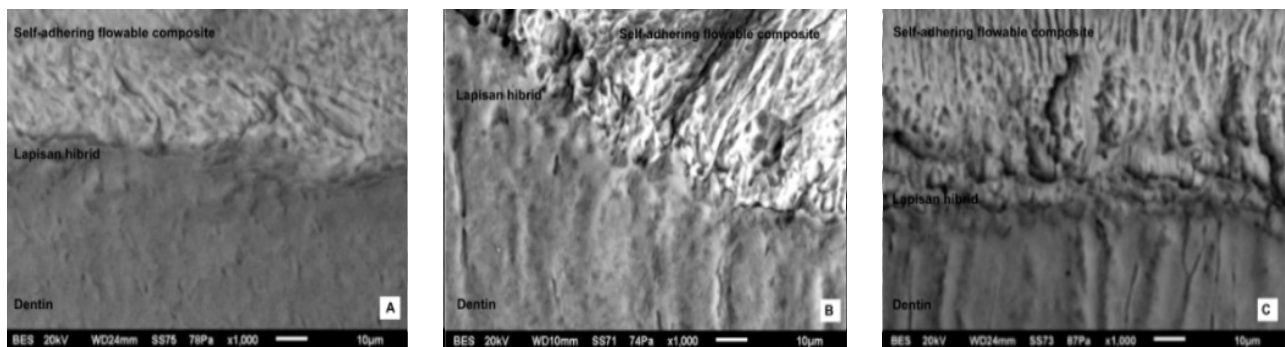


Figure 2. Morphology interface between self-adhering flowable composite and dentin surface. A) 2 g scrubbing pressure for 15 sec; B) 2 g scrubbing pressure for 20 sec; C) 2 g scrubbing pressure for 25 sec.

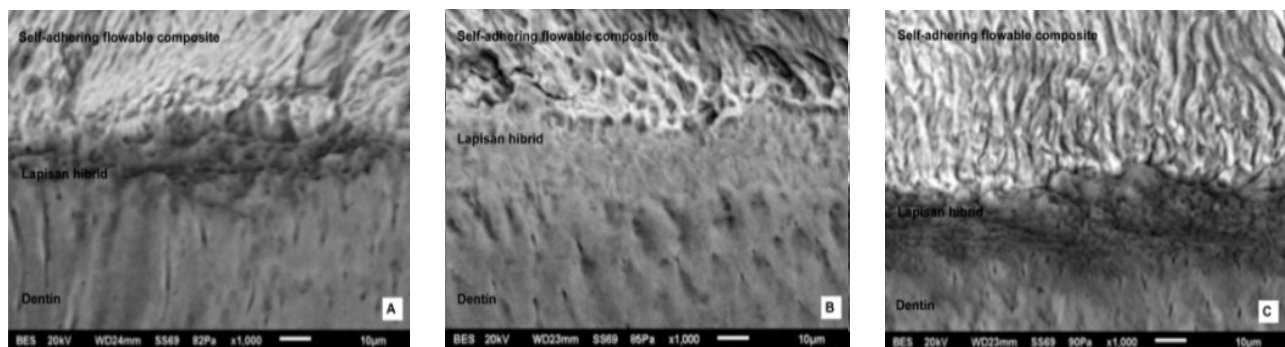


Figure 3. Morphology interface between self-adhering flowable composite and dentin surface. A) 3 g scrubbing pressure for 15 sec; B) 3 g scrubbing pressure for 20 sec; C) 3 g scrubbing pressure for 25 sec.

DISCUSSION

The scrubbing pressure guideline to obtain the optimal shear bond strength of self-adhering flowable composite to dentin surface was found in this study. Applying the brush applicator with an angle of 0 degree relative to dentin surface was equal to 3 grams scrubbing pressure. The highest shear bond strength of self-adhering flowable composite to dentin was 7.22 MPa. This is value lower than shear bond strength of self-etch adhesive to dentin that studied by Al Qahtani⁹ with mean value of 12.61 MPa. In self-etch adhesive system, the etching process and the infiltration of resin occur in the same time.

The mechanism of self-adhering flowable composite bonds to dentin through the chemical bond between the phosphate functional group of GPDM monomer and calcium ion of the tooth structure. Furthermore, through a micromechanical bond as a result of an interpenetrating network formed between the polymerized monomers and collagen fibers as well as the smear layer of dentin, it was known as hybrid layer.^{2,10} It was expected that both self-adhering flowable composite and self-etch adhesive have a similar shear bond strength to dentin, however it was not found in this study.

It can be showed that there was the different demineralization potential between self-adhering flowable composite and self-etch adhesive materials. Factors that influence the demineralization potential of adhesive materials were pH, duration of application process, wettability, viscosity, and water content in materials.¹¹ Self-adhering flowable composite has pH of 1.9² or moderate acidity (pH 1.5–2.0),⁸ which can only remove part of smear layer. The remaining smear layer will prevent the adhesive to infiltrate to the dentinal tubuli and decrease the bond strength. The bond strength to dentin significantly decreased on dentin with thick smear layer.¹² On the other hand, the thickness of hybrid layer is not influenced by smear layer thickness.¹³

The bonding mechanism of adhesive material on dentin surface obtained through the hybrid layer. During demineralization process the hybrid layer formed by infiltration of resin into collagen fibrils. The scrubbing process modified the smear layer into thicker hybrid layer that will increase the bond strength of adhesive.¹⁴ In this study application of self-adhering flowable composite was done by scrubbing technique to form the hybrid layer. This condition simultaneously increase the shear bond strength of self-adhering flowable composite to dentin surface based on scrubbing pressure and duration. Chan *et al.*,¹⁵ stated that bond strength of self-etch adhesive on dentin surface significantly increased after scrubbing process on thick smear layer.

The scanning electron microscopy analysis showed that the formation of hybrid layer gradually appears in line with the scrubbing pressure and duration increment. It was expected that increasing in scrubbing pressure and duration

would modified the smear layer into more qualified hybrid layer. The bond strength to dentin is not dependent on the thickness of hybrid layer but it depends on the quality of the hybrid layer.¹⁶

The important thing in the application of self-adhering flowable composite is the scrubbing process. This process will accelerate the evaporation of solvent contained in adhesive material and increase the diffusion potential of monomer into the dentinal tissue. It will also increase the bond strength of adhesive materials to dentin surface.^{17,18}

This study showed that higher scrubbing pressure will increase the shear bond strength of self-adhering flowable composite to dentin surface. Scrubbing pressure will increase the kinetic energy so that increase the monomer diffusion. Scrubbing pressure caused the collagen tissues to collapse but when the pressure was released, the collagen will relapse and at that time the monomer will penetrate between the collagen tissues and form the hybrid layer.¹⁹ This study also showed that longer scrubbing process increased the shear bond strength of self-adhering flowable composite to dentin surface. Longer in scrubbing duration will increase the demineralization potential of phosphate group contained in GPDM and increase the penetration of monomer into the dentinal tissue. Zohairy⁸ explained that longer contact duration of adhesive materials will increase the bond strength to dentin.

Self-adhering flowable composite has higher viscosity than self-etch adhesive because of filler content. The higher filler content will increase viscosity which can reduce wettability and penetration into dentinal collagen. Adhesive materials should have good wettability and low molecular weight to obtain optimal bond strength to dentin surface,²⁰ but self-adhering flowable composite does not have these properties although the scrubbing technique has been used. Therefore, self-adhering flowable composite as an adhesive material or a restorative material without separate bonding agent need to be considered.

It can be concluded that increasing the scrubbing pressure and duration will increase the shear bond strength of self adhering flowable composite resin to dentin surface. Meanwhile the highest shear bond strength was obtained when the applicator in 0° relative to dentin surface during scrubbing process.

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