

EFFECT OF DIMETHYL SULFOXIDE AS DENTIN SURFACE PRETREATMENT ON MICROTENSILE BOND STRENGTH OF SELF-ADHESIVE VERSUS CONVENTIONAL FLOWABLE COMPOSITE

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ABSTRACT

Objective: Evaluation of the microtensile bond strength of self-adhesive flowable composite in comparison with conventional flowable composite/self-etch adhesive system with and without dimethyl sulfoxide surface pretreatment.

Materials and methods: Based on the type of composite, 60 recently extracted, intact human permanent molars were randomly divided into two main groups, self-adhesive flowable composite (SAFC) (n = 30) was used in the first group, and conventional flowable composite (CFC) (n = 30) was employed in the second. Afterwards, two subgroups were created for each group according to the surface pretreatment (n = 15). A test of microtensile bond strength (μ TBS) was conducted on a sample of 15 specimens. Using a stereomicroscope, the mode of failure was examined. The multiple comparison post hoc test was utilized for statistical analysis of the μ TBS data.

Results: The multiple comparison test showed that the microtensile bond strength (μ TBS) of self-adhesive flowable composite (SAFC) Fusio Liquid Dentine with dimethyl sulfoxide (DMSO) as surface pretreatment was significantly greater than that of any group ($p > 0.05$). A comparison test showed that the self-adhesive flowable composite with and without dimethyl sulfoxide (DMSO) surface pretreatment differed significantly upon examination of μ TBS ($p < 0.05$). But when comparing conventional flowable composite with and without dimethyl sulfoxide (DMSO) surface pretreatment, there was no significant difference in μ TBS ($p > 0.05$).

Conclusion: The application of dimethyl sulfoxide (DMSO) as a surface pretreatment enhances Fusio Liquid Dentin self-adhesive flowable composite microtensile bond strength to dentin, but it barely affects the Flow-It conventional flowable composite strength.

KEYWORDS: self-adhesive composite; DMSO; dentin; bond strength; solvent-free adhesive.

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INTRODUCTION

As the demand for tooth-colored restorations increased, dental composites became the most popular restorative material. Compared to amalgam, composites have great aesthetics, biocompatibility, physical, and mechanical properties.¹ Flowable composite is a class of composite resins that provide favorable handling properties and good adaptation to cavity walls. Flowable composites can perform the role of stress absorption because of their low modulus of elasticity.²

Dental composite restorations use bonding agents to adhere to the tooth structure. Over the last four decades, dental adhesives have evolved at a rapid pace. This evolution has increasingly sought to simplify procedures, moving from three-step, two-step, and single-step adhesive systems,³ for further simplification, “self-adhering composite resins” were recently introduced, eliminating the necessity for a separate step for bonding.⁴

There have been multiple published in vitro investigations on the physical characteristics, binding strength, and marginal sealing potential of these self-adhesive flowable composites,^{5,6} these lab experiments showed that there are still a lot of unanswered questions regarding the mechanical qualities and efficacy of bonds, and some research indicated that self-adhesive composites have little interaction with dentin or enamel.^{7,8}

For both permanent and primary teeth, self-adhesive flowable composites have lower mean microtensile bond strength than conventional flowable resin composites. The integration of a bonding agent into the resin material may have contributed to the lower bond strength of self-adhesive flowable composites. This resulted in incomplete adhesive infiltration into demineralized dentin, ineffective dentin tubule sealing, deterioration of exposed collagen, and degradation of the resin material,⁹ that is why, prior to the restoration, we need to treat the dentine surface in order to strengthen the dentin bond.

Excessive residual water on the dentin surface is assumed to have a major impact on the limited durability of the resin-dentin bond, through three potential mechanisms: water first permits unprotected dentin collagen fibrils to be broken down by endogenous matrix metalloproteinases (MMPs) and cysteine cathepsins. This, in turn, prevents adequate resin bonding impregnation into dentin, thereby weakening the bond strength of the resin composite to dentin.¹⁰ Water also speeds up the hydrolysis of the polymers having ester linkages. Third, in adhesive components, water causes phase separation, which ultimately causes the adhesive resin to hydrolyze and break down.¹¹

Reduced bond strength can lead to long-term problems like leaks, recurring decay, and progressive loss of tooth structure and restoration,¹² the goal of ethanol wet bonding is to remove water from the exposed dentinal collagen and replace it with resin components that are more hydrophobic. As a result, numerous approaches have been put forth to get around these problems and enhance the resin-dentin bond's limited durability.

The hydrolytic destruction of dentinal collagen fibers and resin components present in the resin-dentin hybrid layer may be inhibited by excluding water using high concentrations of ethanol. Previous research has shown that when combined with hydrophobic adhesives, the ethanol-wet bonding approach achieved good results.¹³⁻¹⁵ Nevertheless, due to technique sensitivity and time consumption, this procedure is not clinically practical despite its benefits.

Dimethyl sulfoxide surface pretreatment is an additional strategy that has been suggested for addressing the problem of adhesive resin and collagen fiber hydrolytic breakdown occurring simultaneously. Water self-association can be broken down by dimethyl sulfoxide (DMSO), which also prevents the water layer from forming around triple-helical collagen molecules.^{16,17} Dentin bonding has been proposed to be improved by

dimethyl sulfoxide (DMSO) on the basis of these characteristics.

The null hypothesis tested is that there will be no significant difference in μ TBS between self-adhesive and conventional flowable composite with and without surface pretreatment using dimethyl sulfoxide (DMSO).

Materials used in the study:

The protocol of the study was approved by the Mansoura University ethical committee (Approval No. A04403023CD).

One type of commercially available self-adhesive flowable composite (Fusio Liquid Dentin, Pentron, Avenue Orange, CA, USA), one type of conventional flowable composite (Flow-It ALC, Pentron, Avenue Orange, CA, USA) with its corresponding self-etch adhesive system (Bond-1 SF, Pentron, Avenue Orange, CA, USA), and Dimethyl Sulfoxide Primer (S100 OT, Oy, Finland) were used in the study. Full details are shown in Table 1.

MATERIALS AND METHODS

Sample size calculation:

The microtensile bond strength between various composites that was collected from earlier research (Yuan et al., 2015), sample size was determined using the G Power program version 3.1.9.7, which computed the sample size based on an effect size of 0.52, a 2-tailed test, α error = 0.05, and power = 90.0%. The total sample size that was determined was 15 teeth in each group.

Specimens preparation and restoration placement protocol:

60 newly extracted non-carious and crack free human permanent molars that were extracted due to periodontal diseases from healthy patients (50-60 years) or from patients with a complete denture schedule, collected from the oral surgery clinic at the Faculty of Dentistry at Mansoura University.

TABLE (1)

Brand Name	Adhesion Mode	Manufacturer	Composition	Batch No
Fusio Liquid Dentin	SA	Pentron, Avenue Orange, CA, USA	Resin matrix: 4 - META, Bis-GMA, UDMA and HEMA Fillers: Barium glass and silica	8877224
Flow-It ALC		Pentron, Avenue Orange, CA, USA	Resin matrix: Bis-EMA, Bis-GMA and TEGDMA Fillers: Barium glass, silica, zirconium oxide and glass ionmer	8802317
Bond-1 SF	SE	Pentron, Avenue Orange, CA, USA	Resin matrix: 4-META, UDMA, TGDMA, HEMA Fillers: treated barium glass - silane and silica (amorphous)	9291479
OT Primer S100		OT, Oy, Finland	DMSO	3527K

Abbreviations: SA: Self Adhesive, SE: Self-Etch, DMSO: Dimethyl Sulfoxide, 4 - META: 4-Methacryloxyethyl trimellitic anhydride, Bis-GMA: Bisphenol A-glycidyl methacrylate, UDMA: Urethane-dimethacrylate, Hema: Hydroxyethylmethacrylate, TGDMA: Triethylene glycol dimethacrylate, Bis-EMA: Bisphenylglycidyl dimethacrylate.

Occlusal enamel and superficial dentin were removed perpendicular to the tooth's long axis using a low-speed diamond saw (Isomet 4000, Buehler Ltd., LakeBluff, IL, USA) equipped with a water coolant. It was confirmed that there was no more enamel on the dentin surfaces using a stereomicroscope (Olympus model SZ-PT, Tokyo, Japan) at x75 magnification.¹⁸ Next, polishing was applied to the flat dentin surfaces with wet 600-grit silicon carbide papers (SIA Brand Switzerland) in a rotating manner for 30 seconds to create a standardized smear layer.

Grouping of specimens:

The selected teeth (n = 60) were randomly assigned into two major groups (n = 30). The self-adhesive flowable composite (Fusio Liquid Dentin, Pentron, Avenue Orange, CA, USA) was used for the restoration of one group, while the conventional flowable composite (Flow-It ALC, Pentron, Avenue Orange, CA, USA) with self-etch adhesive system (Bond-1 SF, Pentron, Avenue Orange, CA, USA) was used for the restoration of the second group. Based on the surface pretreatment procedure using dimethyl sulfoxide (DMSO), each group was furthermore separated into two subgroups (n = 15). Each subgroup underwent a microtensile bond strength test (μ TBS) (n = 15).

The tested groups were classified as follows:

Group 1: Every tooth dentin surface was pretreated with dimethyl sulfoxide (DMSO), followed by the application of Fusio Liquid Dentin self-adhesive flowable composite.

Group 2: Every tooth dentin surface was restored by the application of Fusio Liquid Dentin self-adhesive flowable composite directly without surface pretreatment.

Group 3: Every tooth dentin surface was pretreated with dimethyl sulfoxide (DMSO), followed by the application of Flow It conventional flowable composite using Bond 1 SF adhesive in SE mode.

Group 4: Every tooth dentin surface was restored by the application of Flow It conventional flowable composite using Bond 1 SF adhesive in SE mode without surface pretreatment.

Restoration Procedure:

All materials used in this study were applied according to the manufacturer's instructions:

- I- Dimethyl sulfoxide (DMSO) (OT, Oy, Finland) was applied by using a microbrush and rubbed for 60 seconds.
- II- Self etch adhesive (Bond-1 SF, Pentron, Avenue Orange, CA, USA) was applied using a syringe needle of its own, and it was rubbed for 20 seconds. The adhesive was then light cured for 10 seconds using a visible light curing unit with an output density of 450 mW/cm² (LED Bluephase C5, Ivoclar, Vivadent, Amherst, NY, USA).

III- Application of composite resin:

Both types of composites were incrementally applied to build up 4 mm high blocks. Specimens were prepared by using acrylic split cylinders with one 1.0 and two 1.5 mm thickness totaling 4 mm in height and 4 mm in inner diameter. A customized holding acrylic ring was used to fix the acrylic cylinder.

For self-adhesive flowable composite (SAFC), Fusio liquid dentin, Pentron, Avenue Orange, CA, USA, the first layer was applied using a 1.0 mm thick cylinder on the top of the dentin surface. It was agitated for 20 seconds using a micro-brush and light-cured for 10 seconds. A 1.5 mm thick cylinder was then positioned on top of the first layer, and the second layer was applied and light cured for 10 seconds. Subsequently, the third layer was placed using the same cylinder and light-cured for 10 seconds.

Regarding conventional flowable composite (CFC), Pentron, Avenue Orange, CA, USA, following the initial 1.5 mm thick cylinder placement on the dentin surface, the first layer is applied as one increment up to the cylinder's top. The second layer was placed in the same manner; both layers were light cured for 3 seconds each.

The third layer was then placed using the 1.0 mm thick cylinder and light cured for 6 seconds. After that, specimens were kept at room temperature for 24 hours in distilled water.

Microtensile bond strength (μ TBS) test:

After mounting the acrylic cylinder with the teeth, a 0.3 mm thick diamond coated disc (Buehler, IL, USA) was used to serially section restored teeth at a speed of 2050 rpm and a feeding rate of 8.8 mm/min while plenty of water cooling was used. After serial sectioning in the buccolingual direction, the teeth were rotated 90 degrees clockwise and sectioned in the mesio-distal direction. To obtain beams, a final horizontal cut was made at the cemento-enamel connection level. Out of the cut beams in the middle of each group, only four beams were chosen. The resulting beams had a thickness of 1 ± 0.1 mm and a length of 7 ± 1 mm. Every beam's thickness and length were measured using a digital caliper (Mitutoyo, Tokyo, Japan).

Determination of μ TBS:

For each tested subgroup, 60 beams were tested. On a universal testing machine (Instron, MA, USA) using Geraldeli's jig, beams were placed. Then, beams were glued by their ends to the central groove of Geraldeli's jig using cyanoacrylate-based glue (Zapit, DVA Inc., USA). A cross-head speed of 0.5 mm/min was used to provide tensile load until the bond failed through the specimen. Then, using MegaPascal (Bluehill Lite software, Instron, MA, USA), μ TBS was computed. Using a scalpel, carefully remove the specimen fragments from

Geraldeli's jig. Store them in the matching plastic cones labeled with their names until the failure mode is examined.

Following debonding, the fractured sites were examined under an x15 magnification stereomicroscope (Olympus model SZ-PT, Tokyo, Japan) in order to determine the mode of failure. Failure modes are categorized as adhesive failure (failure at the interface between the resin and dentin), cohesive failure (failure only within the dentin or resin composite), and mixed failure (failure at the interface between the resin and dentin combined with cohesive failure of the adjacent substrates). A statistical analysis was conducted to determine the most common mode of failure.

RESULTS

Microtensile Bond Strength:

The mean μ TBS values for all tested groups are shown in Table 2.

Multiple comparative tests between the various materials revealed that the μ TBS of self-adhesive flowable composite (SAFC) Fusio Liquid Dentine with dimethyl sulfoxide (DMSO) as surface pretreatment was significantly higher than all groups ($p > 0.05$). The comparison test showed that there was a significant difference in μ TBS between self-adhesive flowable composite with and without dimethyl sulfoxide (DMSO) surface pretreatment ($p < 0.05$); however, there was no significant difference in μ TBS between conventional flowable composite with and without dimethyl sulfoxide (DMSO) surface pretreatment ($p > 0.05$), as shown in Table 3.

TABLE (2) Descriptive statistics of microtensile bond strength

Micro tensile Strength	Group (1) SAFC+DMSO	Group (2) SAFC	Group (3)CFC+DMSO	Group (4) CFC
Mean \pm SD	31.56 \pm 5.75	7.23 \pm 4.43	17.18 \pm 4.94	14.83 \pm 2.65
Median (min-max)	31.45 (21.32-41.75)	6.06 (1.54-14.43)	17.88 (8.75-27.89)	15.02 (9.21-17.96)

Means are values \pm standard deviation

The current study's experimental unit is the tooth.

Abbreviations: SAFC: Fusio Liquid Dentin Self-Adhesive Flowable Composite; CFC: Flow It Conventional Flowable Composite; DMSO: Dimethyl sulfoxide primer.

TABLE (3) Comparison of microtensile bond strength

Micro tensile Strength	Group 1 SAFC+DMSO	Group 2 SAFC	Group 3 CFC+DMSO	Group 4 CFC	Test of significance
Mean \pm SD	31.56 \pm 5.75	7.23 \pm 4.43	17.18 \pm 4.94 ^a	14.83 \pm 2.65 ^a	F=3.65 P<0.001*

*F: One Way ANOVA test; *statistically significant*

Non-significant differences between the study groups are indicated by similar letters in the same row.

Abbreviations :SAFC: Fusio liquid dentin Self-Adhesive flowable composite; CFC: Flow It conventional flowable composite; DMSO: Dimethyl sulfoxide primer.

Failure mode evaluation:

As shown in Table 4, in all groups, the most common failure modes were adhesive and mixed failure. However, the cohesive failure mode percentage was the lowest when compared to other failure modes. The adhesive failure mode

was commonly observed in self-adhesive flowable composite (SAFC) with and without dimethyl sulfoxide (DMSO) surface pretreatment, while the mixed failure mode was more commonly observed in conventional flowable composite (CFC) with and without dimethyl sulfoxide (DMSO) surface pretreatment.

TABLE (4) Comparison of Mode of Failure

Mode of failure	Group 1 N(%)	Group 2 N(%)	Group 3 N(%)	Group 4 N(%)	test of significance
AD	15(100)	15(100)	15(100)	13(86.7)	MC=6.21 P=0.102
M	0	0	0	2(13.3)	
MC Nemar test	P=1.0	...	P=0.50	P=1.0	

DISCUSSION

Fusio Liquid Dentin is a flowable composite with glass fillers and nano-sized amorphous silica that is based on a 4-MET monomer. The special formulation of Fusio Liquid Dentin is hydrophilic and acidic. When the methacrylate monomers come into contact with the tooth surface, their negatively charged carboxylic acid groups bond to mineral ions found within the tooth structure. Dentin bonding and sealing capabilities are improved as the monomers

are integrated into the dentin surface through the polymerization and neutralization of the carboxylic acid groups.¹⁹

In general, a number of variables may be responsible for dental adhesives' reduced dentin bond strength: to begin with, dentin is more water-rich and less mineralized than enamel. Furthermore, the smear layer is present. Third, the stability and endurance of the composite resin-dentin bond are reduced by fluid-filled dentin channels that

are subject to a minimal but continuous outward pressure from the pulp.²⁰

Results of FLD (Fusio Liquid Dentin) groups reported decreased μ TBS values, and it was determined that this difference was statistically significant, which is in agreement with Altunsoy M. et al.²¹ who claimed that in their investigation, which evaluated the μ TBS values of FLD and VF (Vertise Flow) on permanent dentin without adhesive and with various surface pretreatments, FLD had lower values, and this difference was found to be statistically significant. Brueckner C. et al.²² believed that the varied functional monomers and composition of SACs were the cause of this difference.

There are currently very few papers assessing the μ TBS of SACs in the literature. Vertise Flow and Fusio Liquid Dentin's μ TBS were compared to dentin and other self-etch adhesive systems. According to the findings of Poitevin A. et al.⁵ their μ TBS were 11 MPa and 13 MPa, respectively. These results are more than what we found in our study, where Fusio Liquid Dentin showed 7.23 MPa μ TBS values without dentin surface pretreatment. This study describes the first-ever in vitro μ TBS of SACs to dentin with dimethyl sulfoxide (DMSO) pretreatment; consequently, the results of this study were compared to those of earlier investigations employing different surface pretreatment methods and with microshear bond strength data in an attempt to prove the given findings.

The fracture beam analysis confirmed the bond strength statistics by demonstrating a reduced proportion of cohesive failure in dentin and composite in all groups and a higher percentage of adhesive/mixed failure in all groups. Powers JM. et al.²³ stated that the degree of substrate fracturing is frequently a good indicator of the adhesive's retentive strength.

The results of group 1 showed the highest results, maybe because of the use of dimethyl sulfoxide (DMSO) primer as a surface pretreatment, but the results of group 2 showed a lower bond

strength than the results of group 1 due to the direct application of SAC without any surface pretreatment. These results are compatible with those proven by Tuloglu N. et al.,⁹ who contrasted the shear bond strength (SBS) values in primary and permanent teeth using the Optibond All-In-One one-step SE adhesive system and a conventional flowable composition by applying VF (Vertise Flow) without adhesive. They discovered that the group receiving Optibond+Ultimate Flow had the greatest SBS values for both primary and permanent teeth, while the group receiving SAC application alone had the lowest values.

Dentin bonding is affected by dimethyl sulfoxide (DMSO) pretreatments, although the exact mechanism is still unknown. But dimethyl sulfoxide (DMSO) increases adhesive infiltration into demineralized dentin²⁴, probably because it biomodifies the collagen matrix through the following mechanisms: (i) increased collagen microfibril spacing;^{25,26} (ii) improved dentin wettability;²⁷ (iii) decreased water's self-associative tendency;²⁸ (iv) DMSO's penetration enhancer properties.²⁹

The potential of dimethyl sulfoxide (DMSO) to enhance dentin-resin bonding has been assessed in a number of studies. The outcomes, however, have been mixed; numerous investigations have noted an improvement in the adhesive's bonding strength to dentin^{12,27,30} although only a few investigations found that the administration of dimethyl sulfoxide (DMSO) had no immediate impact on bond strength values.^{12,27,31}

Although Bond-1SF self-etch adhesive, which is solvent-free and doesn't include acetone, ethanol, or water in its chemical composition, was utilized in this investigation, phase separation can still occur in one-component adhesives when HEMA is present.^{32,33}

The results of groups 3 and 4 showed that dimethyl sulfoxide (DMSO) surface pretreatment had no significant effect on μ TBS, which may be affected by using of a separate adhesive step. It seemed fair

to anticipate some chemical interaction, keeping in mind that Bond 1 SF consists of the 4-MET monomer, which has been shown to form ionic interactions with hydroxyapatite¹⁹. Furthermore, Bond 1 SF has a high pH value (pH=3-4), which could decrease the dentin demineralization depth and, among other factors, cause a lack of formation and lower bond strength due to the material's composition and infiltration capacity.

Furthermore, the present findings are consistent with a study's findings by Khoroushi M. et al.³⁴ that assessed this innovative solvent-free adhesive system's bonding efficacy.³⁴ This result may be explained by the possibility that removing the solvent from a self-etch adhesive system will lessen the adhesive components' ability to penetrate the microstructures of dental tissue, hinder the formation of hybrid zones, and ultimately weaken the bond between the adhesive and the dentin.

Numerous factors could contribute to differences across all the groups, but the composition, rheological potential, and types of functional monomers are likely to have the most effects,³⁵ in addition to the presence or absence of surface pretreatment.

Since the interactions between the tested "dentin pretreatment" using self-adhesive flowable composite (SAFC) with and without dimethyl sulfoxide (DMSO) ($p < 0.005$) had a significant impact on dentin bonding strength, and there was no significant difference between the tested "dentin pretreatment" using conventional flowable composite (CFC) with and without dimethyl sulfoxide (DMSO) ($p = 0.005$), so the null hypothesis was partially rejected.

CONCLUSIONS

Under the limitations of this study, the results of the μ TBS test, and the circumstances of the current investigation, we can draw the conclusion that using dimethyl sulfoxide (DMSO) as a surface pretreatment with self-adhesive flowable composite (SAFC) showed satisfactory μ TBS results compared to a non-treated dentin surface, while application

of dimethyl sulfoxide (DMSO) prior to Bond 1 SF (solvent free adhesive) did not significantly affect the mean μ TBS of conventional flowable composite (CFC).

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