

Effect of Antibacterial Varnishes Applied to Root Dentin on Shear Bond Strength of Tooth-colored Restorative Materials

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Clinical Relevance

The application of antibacterial varnish affects the shear bond strength of tooth-colored restorative materials after six months.

SUMMARY

This study investigated the effect of certain varnishes on the bond strength of different tooth-colored restorative materials applied to root dentin. One-hundred and eighty tooth slabs, including mesial and distal surfaces, were attained through dividing the teeth, then embedding them in methylmethacrylate. The root surfaces were ground flat through cementum, exposing the dentin. The samples were then randomly divided into three main groups: Group 1: Cervitec; Group 2: Fluor Protector and Group 3: No applications (control). Cervitec and Fluor Protector were applied to the root dentin surfaces according to the manufacturer's instructions. All the samples were kept in artificial sali-

va for six months. Each main group was subdivided into five groups of 12 teeth each: Group A: Flowable Resin Composite (Grandio Flow); Group B: Microhybrid Resin Composite (Artemis); Group C: Polyacid Modified Resin Composite (Dyract Extra); Group D: Resin Modified Glass Ionomer Cement (Vitremer) and Group E: Conventional Glass-Ionomer Cement (Ionofil Molar). Restorative materials were applied to the root dentin surfaces using a cylindrical mold. After thermocycling (1000 cycles, 5°C/55°C, dwell time 30 seconds), the shear bond strength of the restored samples was determined by a universal testing machine (Zwick Test Machine, Zwick GmbH & Co, Ulm, Germany) at a 5 mm/minute crosshead speed. Failure mode was determined under a stereomicroscope. The data were evaluated statistically by using one-way Analysis of Variance and Duncan tests ($p \leq 0.05$). In the fluoride varnish group, all of the restorative materials except for Ionofil Molar, showed lower bond strengths when compared to the control group ($p < 0.05$). In the Cervitec group, Artemis and Dyract Extra showed lower bond strengths; whereas, Ionofil Molar showed a higher bond strength than the control group ($p < 0.05$).

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The highest percentage of cohesive fracture was observed in Artemis and Dyract Extra in the control group.

INTRODUCTION

Reforms in healthcare and health awareness have lengthened lifespans and reduced edentulism, while at the same time presenting significant challenges to the dental community in the form of root caries. Root caries is often more difficult to diagnose than coronal caries and restoration is challenging or even impossible.¹ The most desirable treatment for root caries is remineralization,² and several methods have been proposed to promote remineralization. These methods include daily use of a fluoride-containing mouthrinse, professional application of fluoridated gels and antibacterial varnish.³⁻⁵ Fluoride inhibits root caries, as it does coronal caries, even though the tissues initially affected are quite different.⁶ Several *in situ* and *in vivo* studies demonstrated the cariostatic effect of topically applied fluorides on initial root surface lesions.⁷⁻⁸ Chlorhexidine (CHX)-containing products are noteworthy, because of their ability to reduce pathogens from the immediate environment and possibly from carious dentin and soft tissues.⁹ CHX is effective against *Streptococcus mutans*.⁵ An antimicrobial varnish containing CHX has been reported as effective in reducing root caries activity.¹⁰ Varnishes ensure a slow release of CHX, thus bringing out constant levels of the agent at desired locations.¹¹

Restorative management of root caries is challenging in view of the difficulties, such as visible moisture control, access, proximity of the pulp, proximity of the gingival margin and the high organic content of dentin.¹² Many restorative materials, such as Resin Modified Glass Ionomer Cements (RMGIC),¹³ conventional Glass Ionomer Cements (GIC), Polyacid Modified Composite Resins (PMCR), resin composites and amalgam have been used to restore root carious lesions.^{12,14} Significant progress has been made in the area of adhesion and esthetic restorative materials, and tooth-colored restorative materials have become popular.¹⁵ After the introduction of GICs in 1972, they have gained popularity for their capacity for fluoride release.¹⁶ Chemically hybrid materials are between GICs and resin-based composites. RMGICs are similar to conventional GICs, while PMCRs are more similar to resin-based composites.¹⁷⁻¹⁹ For achieving maximum esthetics in restoring root caries, composites are the most suitable choice. Hybrid composites are currently available and combine improved strength with the superior esthetic quality previously seen only in microfill composites.²⁰ Flowable resin composites were developed in response to requests for easy handling properties in late

1996. These resin composites were created by reducing filler content.²¹

This study determined the effects of CHX and fluoride varnishes applied to root dentin on the shear bond strength (SBS) of a nanohybrid flowable resin composite (Grandio Flow), microhybrid resin composite (Artemis), PMCR (Dyract Extra), RMGIC (Vitremer) and conventional GIC (Ionofil Molar).

METHODS AND MATERIALS

Ninety recently extracted, unerupted human third molars were collected and stored in distilled water. One-hundred and eighty mesial and distal surfaces were attained by sectioning the teeth buccolingually. The specimens were embedded in methylmethacrylate in a plastic ring. The underlying superficial root dentin was exposed by mounting the specimens on a metallurgical polishing wheel and ground flat with 240, 400 and 600-grit SiC paper. The specimens were inspected with a stereomicroscope (Leica MS5, Singapore, Singapore) to ensure that no cement was left and no pulpal exposure occurred. The specimens were randomly assigned to three main groups, containing 60 specimens each. The groups were prepared to receive the following treatments:

Group 1: CHX varnish (Cervitec)

Group 2: Fluoride varnish (Fluor Protector)

Group 3: No treatment (control)

Cervitec and Fluor Protector were applied to the root surfaces of the samples, following manufacturer's instructions, using a disposable brush (Groups 1 and 2). For the control group, no application was done (Group 3).

All the samples were stored in artificial saliva for six months. The composition of the artificial saliva was as follows (g/l): methylp-hydroxybenzoate, 2.00; sodium carboxymethyl cellulose, 10.0; KCL, 0.625; $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, 0.059; $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, 0.166; K_2HPO_4 , 0.804; KH_2PO_4 , 0.326; Sorbo, 42.75. The PH of the artificial saliva was 6.75 (Radiometer MKII Blood Microsystem).

After six months, the three main groups were subdivided into five equal subgroups (n=12).

Group A (Flowable Composite Group): The root dentin surfaces were etched with 34.5% phosphoric acid gel (Vocacid Gel; Voco) for 15 seconds and rinsed for 20 seconds. One coat of Solobond M (Voco) acetone-based one-bottle adhesive was applied homogeneously to the visibly moist root dentin surfaces with a disposable brush and left undisturbed for 30 seconds. Solobond M was spread with a faint air jet and light-cured for 20 seconds. A nanohybrid flowable composite Grandio Flow (Voco) was applied using a syringe and light cured for 40 seconds.

Table 1: Antibacterial Varnishes, Restorative Materials and Bonding Agents Used in This Study

Materials	Type	Manufacturer
Fluor Protector	Fluoride Varnish	Ivoclar-Vivadent, Schaan, Liechtenstein
Cervitec	Chlorhexidine Varnish	Ivoclar-Vivadent, Schaan, Liechtenstein
Solo Bond M	Bonding Agent	Voco, Cuxhaven Germany
Grandio Flow	Flowable Resin Composite	Voco, Cuxhaven Germany
Excite	Bonding Agent	Ivoclar-Vivadent, Schaan, Liechtenstein
Artemis	Microhybrid Resin Composite	Ivoclar-Vivadent, Schaan, Liechtenstein
Prime&Bond 2.1	Bonding Agent	De Trey Dentsply, Konstanz, Germany
Dyract Extra	Polyacid Modified Resin Composite	De Trey Dentsply, Konstanz, Germany
Vitremer Primer	Primer	3M Dental Products, St Paul, MN, USA
Vitremer	Resin Modified Glass Ionomer Cement	3M Dental Products, St Paul, MN, USA
Ionofil Molar	Traditional Glass Ionomer Cement	Voco, Cuxhaven, Germany

Group B (Composite Group): The root dentin surfaces were etched for 15 seconds with 37% phosphoric acid gel (Total Etch, Vivadent) and rinsed with water spray for 20 seconds. One coat of an ethanol-based adhesive (Excite, Vivadent) was applied to the visibly moist root dentin surfaces and brushed gently for 10 seconds. Following evaporation of the solvent, the adhesive was light-cured for 20 seconds. The microhybrid resin composite (Artemis, Vivadent) was applied and cured for 40 seconds.

Group C (Polyacid Modified Resin Composite Group): The primer Prime&Bond 2.1 (Dentsply) was applied with a brush (Microbrush) to the surfaces of the root dentin, maintaining contact with the root dentin for 30 seconds, then light cured for 20 seconds. Dyract Extra (Dentsply) was applied and cured for 40 seconds.

Group D (Resin Modified Glass Ionomer Cement Group): The primer (Vitremer Primer, 3M ESPE) was applied with a brush to the surfaces of the root dentin, maintaining contact with the root dentin for 30 seconds, then light cured for 20 seconds. Vitremer was mixed at a powder/liquid ratio of 1:1 on a glass plate, then inserted with a spatula.

Group E (Conventional Glass Ionomer Cement Group): Ionofil Molar (Voco) was mixed in an amalgamator and applied with a cement applicator.

A halogen curing unit (Hilux Expert, Benlioglu Dental, Ankara, Turkey) was used for the materials requiring curing. The adhesive systems and restorative materials are shown in Table 1. All adhesive systems were applied to the center of the specimens. The restorative materials were applied by packing them into cylindrical-shaped plastic tubes with an internal diameter of 3 mm and a height of 4 mm, following the manufacturers' directions. All the samples were subjected to 1000 cycles of thermocycling between 5°C and 55°C, with a dwell time of 30 seconds. To test shear bond strength, the specimens were held in jaws that had been clamped to the base plate of a universal test-

ing machine (Zwick Test Machine, Zwick GmbH & Co, Ulm, Germany). A shear load was applied vertically using a knife-edged rod from the load cell to the base of the cylindrical mold. The crosshead speed was 5 mm/minute. The maximum load that the specimen could withstand until failure was determined and the shear bond strengths were calculated by dividing the load of failure to the surface area of the mold. After testing the shear bond strength, the fracture sites were then viewed by a stereomicroscope (Leica MS5, Singapore, Singapore), under 16x magnification, to determine if the mode of failure was either adhesive or cohesive.

The bond strength data were statistically analyzed by one-way ANOVA and Duncan's test at a significance level of 0.05.

RESULTS

The results of the SBS tests with various tooth-colored restorative materials applied to varnish-treated and control-root dentin surfaces are shown in Table 2.

In the Fluor Protector group, Dyract Extra showed significantly higher SBS than Grandio Flow, Vitremer and Ionofil Molar ($p < 0.05$) but was not statistically different from Artemis ($p > 0.05$). Ionofil Molar exhibited significantly lower SBS than all the other restorative materials ($p < 0.05$). For the Cervitec group, Grandio Flow showed significantly higher SBS values than all the other restorative materials tested ($p < 0.05$). In the control group, Dyract Extra exhibited statistically significant high SBS values ($p < 0.05$); whereas, Ionofil Molar showed the statistically significant lowest SBS values compared to all the other restorative materials ($p < 0.05$).

For Artemis and Dyract Extra, both Fluor Protector and Cervitec showed statistically lower SBS values than the control group ($p < 0.05$). For Grandio Flow and Vitremer, only Fluor Protector had statistically lower SBS values than the control group ($p < 0.05$). For Ionofil Molar, Cervitec exhibited higher SBS values than Fluor Protector and the control ($p < 0.05$).

Table 2: Mean Shear Bond Strength Values of Five Different Restorative Materials to Root Dentin Treated with Fluor Protector, Cervitec and Control (no treatment) Groups (MPa)

Restorative Materials	Fluor Protector (Mean \pm SD)	Cervitec (Mean \pm SD)	Control (Mean \pm SD)
Grandio Flow	10.68 \pm 2.63	23.11 \pm 6.88	20.22 \pm 4.00
Artemis	13.29 \pm 1.72	11.18 \pm 3.52	21.10 \pm 9.32
Dyract Extra	15.27 \pm 5.81	14.80 \pm 2.93	30.22 \pm 11.22
Vitremer	11.06 \pm 2.23	16.22 \pm 9.75	22.96 \pm 9.52
Ionofil Molar	5.82 \pm 2.27	10.41 \pm 3.34	7.54 \pm 2.35

Table 3: Frequency of Failure Mode of Five Different Restorative Materials to Root Dentin after Fluor Protector, Cervitec and Control (no treatment) Groups

Restorative Materials	Fluor Protector		Cervitec		No Treatment	
	Adhesive	Cohesive	Adhesive	Cohesive	Adhesive	Cohesive
Grandio Flow (n=12)	75%	25%	75%	25%	50%	50%
Artemis (n=10)	70%	30%	75%	25%	40%	60%
Dyract Extra (n=11)	63.6%	36.4%	58.3%	41.7%	40%	60%
Vitremer (n=10)	100%	0%	90%	10%	80%	20%
Ionofil Molar (n=9)	88.9%	11.1%	90%	10%	90%	10%

The frequency of failure for Fluor Protector, Cervitec and the Control is shown in Table 3.

DISCUSSION

Root caries is a serious oral health problem, because of its high prevalence and difficulty in treatment.⁶ Fluoride- and CHX-containing antibacterial agents can be promising for the remineralization of initial root caries. However, such varnishes can penetrate demineralized root dentin only up to 40% of the total lesion depth, due to their hydrophobic nature.¹¹ The root dentin could act as a CHX depot, slowly releasing CHX for as long as six months after a single application of Cervitec. This could be due to varnish penetration into the dentinal tubules and subsequent active agent release.¹¹ Besides these antibacterial varnish applications, it is sometimes impossible to arrest initial root-surface caries, and restorative procedures are needed.

The restorative materials in this study were selected from materials routinely used in clinical practice to restore root caries lesions. The tested materials were used as recommended by the manufacturer. Successful adhesion to dentin is one of the requirements when choosing tooth-colored materials. Different factors can affect the shear bond strength of tooth-colored materials to dentin. In this study, Fluoride- and CHX-containing antibacterial varnish applications were tested to determine their effect.

Microfilled resin composites are often advocated for the restoration of root surface lesions, because they have a lower elasticity modulus than hybrid resin composites. The argument for this type of resin composite restoration is because the tooth flexes during mastication and flexible restorative materials flex with the

teeth.¹⁴ Flowable composites, initially introduced to restore Class V defects, may also be preferred for restoring root caries.²² Arising from the progressive improvements of composite materials, flowable resin composites containing nanoparticles were introduced to the dental market.

Bond strengths exceeding 20 MPa eliminate gap formation produced during polymerization shrinkage.²³ It has been hypothesized that a minimum bond strength of 17 to 20 MPa to dentin is required to withstand contraction forces to resin composite materials.²⁴ In the current study, the microhybrid resin composite Artemis showed a mean SBS of 21.10 MPa only in the control group, and the nanohybrid flowable composite Grandio Flow exhibited a mean SBS of 20.22 and 23.11 MPa for the control and Cervitec groups, respectively.

In this study, 1% CHX varnish Cervitec significantly decreased the SBS of Artemis, which was used with an acetone-based bonding agent; whereas, it did not affect the SBS of Grandio Flow, which was used with the ethanol-based bonding agent. In two other studies, 2% CHX disinfectant treatment decreased the SBS of composite to dentin.²⁵⁻²⁶ On the other hand, Say and others²⁷ showed that the use of 2% CHX solutions as cavity disinfectants after etching dentin did not affect the shear and tensile bond strengths of One Step and Optibond Solo. A scanning electron microscopy study revealed that CHX solution deposited debris on the surface and within the tubules of etched dentin but CHX had no significant effect on the shear bond strength of composite to dentin.²⁸

The results of an *in vitro* study suggest that the application of topical fluoride agents to enamel surfaces seven days before bonding orthodontic attach-

ments does not have an adverse effect on bond strength.²⁹ In this study, Fluoride varnish Fluor Protector significantly decreased the SBS of Artemis, Grandio Flow, Dyract Extra and Vitremer. This difference was attributed to the fact that root dentin was used.

Glass Ionomer Cements (GIC) are able to bond to dentin and enamel through adhesive bonding, for this reason, they have found a wider range of application.³⁰ GIC of all types continue to combine fluoride release, adhesion, good marginal seal and reasonable esthetics. All glass ionomer restorative materials contain water and are supplied as powder and liquid.³¹ These materials bond best to moist tooth structures, while drying reduces bond strength.¹⁴ Even with these limitations, properly placed glass ionomer restorations are durable and clinically successful.¹⁴ Conventional and Resin Modified Glass Ionomer Cements (RMGIC) have similar setting reactions. Both begin with an acid-base reaction, which releases fluoride as a byproduct of the setting reaction.¹⁴ RMGIC also requires a surface pretreatment.³² In addition to chemical bonding by ionic exchange between RMGIC and the dentin substrate, the penetration and further light-curing of RMGIC through the smear layer into dentinal tubules provides an additional mechanical interlocking of the polymer to dentin.³² As a result, RMGICs have been shown to yield higher bond strength to dentin than conventional GICs.³² In the current study, conventional GIC showed statistically lower SBS to root dentin than RMGIC in all groups. In a recent study, Vitremer bond strength values were found to be 5.58 ± 2.09 MPa for dentin;³³ whereas, the results of the current study showed Vitremer to be 11.06, 16.22 and 22.96 MPa, respectively, for Fluor Protector, Vitremer and the control groups. The high SBS values of Vitremer are attributed to the fact that, in the current study, root dentin was used. Similar to the results of the current study, Cunningham and Meiers³⁴ showed that a chlorhexidine solution did not significantly affect the shear bond strength of Vitremer. In the current study, the application of a varnish containing 1% CHX did not affect the SBS of Vitremer.

The chemical reaction allows for the establishment of bonding to dental hard tissues when the carboxylic components of the cement and the calcium present in enamel and dentin substrates react.³⁵⁻³⁶ However, such an adhesion mechanism is weak, leading to low bond strengths of the GIC to dentin.³⁵ In part, this weakness is due to the presence of a smear layer, which may interfere with the adhesion mechanism, because of its susceptibility to fail cohesively.³⁶ In order to overcome these drawbacks, and thus enhance bonding, different surface treatment agents have been suggested to remove the smear layer before placement of the GIC.^{35,37} Polyacrylic acid is the most commonly used

agent for conventional glass ionomer cements.^{35,37} In the current study, no surface treatment was applied. Two different antibacterial agents were applied to the root dentin surfaces six months prior to conducting the restorative procedure. CHX varnish applied to root dentin surfaces showed statistically higher SBS values for Conventional GIC than Fluor Varnish and the control groups. Chlorhexide varnish may have a chemical reaction that positively affected the SBS of Conventional GIC.

It was found that the compomer SBS to enamel was significantly affected by the method of treatment to enamel.³⁸ García-Godoy and others³⁹ revealed that Dyract's SBS value was 15.33 ± 6.96 for dentin. In the current study, Dyract Extra's SBS values were found to be 15.27, 14.80 and 30.22 for root dentin to Fluor Protector, Cervitec and the control groups, respectively.

Awliya and others⁴⁰ reported that fluorosis reduces the SBS of glass ionomer-based restorative materials to dentin. Also, in the current study, Fluor Protector applied to root dentin surfaces exhibited lower SBS values than the control for RMGIC and PMCR.

Evaluation of the failure mode after SBS testing showed a predominance of adhesive failure. The highest cohesive failure percentage was observed in the control group for Dyract Extra and Artemis. Cohesive failures of adhesive materials are related to the high values of bond strength, predicting an effective bonding.⁴¹

The SBS test is a simple procedure for the experimental evaluation of adhesive systems and is widely used.⁴² Other bond strength tests, which include tensile and fracture toughness, have been suggested.⁴³⁻⁴⁵ Whether any test with results in the fracture and removal of dentin truly measures the strength of the dentin substrate, is moot.⁴⁶ Bonding tests are necessary and useful in predicting the performance of adhesive systems, but clinical success can not be obtained by *in vitro* investigations.⁴⁷

CONCLUSIONS

Under the conditions of this *in vitro* study, it can be concluded that the varnishes Fluor Protector and Cervitec decreased the shear bond strength of microhybrid composite and Polyacid Modified Resin Composite. For Resin Modified Glass Ionomer Cement and flowable composite, only Fluor Protector decreased shear bond strength; whereas, Cervitec had a positive effect on the shear bond strength of conventional Glass Ionomer Cement. A Polyacid Modified Resin Composite statistically exhibits higher shear bond strength values than other restorative materials to root dentin without any varnish application.

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