Microshear bond strength of composite resin to demineralized dentin after remineralization with sodium fluoride, CPP-ACP and NovaMin containing dentifrices

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ABSTRACT

Background: Bonding composite to remineralized dentin is challenging. The aim of this study was to evaluate the microshear bond strength (μ SBS) of composite to demineralized dentin, which had been remineralized with sodium fluoride (NaF), CPP-ACP and NovaMin containing dentifrices.

Materials and methods: 108 extracted human premolars or molars were sectioned longitudinally into buccal and lingual halves ($n = 216$). Dentin (3×3 mm) was exposed on the cervical aspect of teeth and the samples were allotted randomly to six groups ($n = 36$) according to the remineralizing agent used namely, group 1 (sound dentin), group 2 (demineralized dentin), group 3 (NaF), group 4 (CPP-ACP), group 5 (NovaMin) and group 6 (non-fluoridated). The type of bonding system (total-etch or self-etch) formed the subgroups. Samples in groups 2–6 were submitted to an acid challenge for 3 days followed by remineralization in groups 3–6 for 90 days. Etching pattern ($n = 3$) was observed under SEM. μ SBS of the bonded samples ($n = 15$) were evaluated. The data were statistically analyzed using Kruskal Wallis and Dunn's Post Hoc tests ($p < 0.05$).

Results: SEM micrographs of remineralized samples showed varying degrees of partially occluded and exposed dentinal tubules. Under both the adhesives, the mean μ SBS of composite in groups 2–6 was lesser than that of group 1. Among self-etch subgroups, μ SBS of NaF group was not significantly different from that of normal dentin.

Conclusion: Composite forms a weaker bond to remineralized dentin. Self-etch bonding system is capable of achieving acceptable bond strength to dentin remineralized with NaF and NovaMin.

1. Introduction

Breakthrough in the field of medicine and preventive dentistry has prolonged the retention period of dentition in the oral cavity. This has also steadily increased the prevalence of non-carious cervical lesions (NCCLs). NCCLs involve loss of tooth structure due to several etiologies like chemical agents, occlusal stress and mechanical factors or a combination of all these. They are the result of increased speed and level of demineralization, which becomes dominant with time. Erosion, abrasion and abfraction occur on account of interplay of several such factors.^{1,2} Dentinal hypersensitivity, plaque retention, caries incidence, loss of structural integrity and pulp vitality further complicate NCCL treatment.³

Exposed and patent dentinal tubules are dominant in dentinal hypersensitivity, the remedy for which, over the years has mainly aimed at the concept of occlusion of dentinal tubules and formation of

remineralized zones which are resistant to dissolution.^{4,5} This situation has been managed by the topical application of various physical and chemical agents either chair-side by the clinician or at home by the patient.⁶

The loss of cervical tooth structure is often compensated by restoration of the defect to meet esthetic and functional demands. NCCLs are restored using glass ionomer cement (GIC) or composites resins. While taking esthetics under consideration, composites are usually preferred.⁷ But the durability of composite restoration depends on the substrate onto which they bond.⁸ Demineralization occurring in NCCLs and long term remineralizing treatment alter the structure of dentin. Thus, it is a unique challenge for the clinician to successfully restore such demineralized and partially remineralized NCCLs.⁹

However, literature regarding the bonding of composites to such partially remineralized NCCLs is sparse. Hence, this *in vitro* study aimed to evaluate the microshear bond strength (μ SBS) of composite resin

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Fig. 1. Schematic representation of methodology a. Extracted posterior teeth were collected b. Teeth sectioned horizontally, roots discarded c. Crowns sectioned longitudinally into buccal and lingual halves d. 3 × 3 mm dentin was exposed on the cervical one-third, rest of the sample was covered with nail varnish.

* Samples were immersed in 0.5% citric acid (CA) solution (pH 2.5) for 3 days.

Remineralized for 90 days with intermittent demineralization using 0.5% CA and storage in artificial saliva.

bonded to demineralized dentin which had been remineralized with fluoride, CPP-ACP and NovaMin containing dentifrices. The objectives of this *in vitro* study were:

1. To analyze the acid-etched patterns promoted by phosphoric acid and acidic monomers on dentin remineralized using three different dentifrices using scanning electron microscope (SEM).
2. To evaluate the microshear bond strength (μ SBS) of composite bonded with total-etch and self-etch adhesives to remineralized dentin under universal testing machine (UTM).

Null hypotheses tested were that neither the type of remineralizing regimen nor the adhesive will influence the μ SBS of the composite to dentin.

2. Materials and methods

The schematic representation of methodology is given in Fig. 1. 108 extracted human premolars or molars were thoroughly cleaned of debris and stored in 10% formalin until use. The crowns were separated

from the roots by sectioning the teeth 1 mm apical to the cemento-enamel junction using a diamond disc. The crown portions were sectioned longitudinally into buccal and lingual halves (n = 216). The sectioned halves were placed in Teflon rings filled with self-cure acrylic resin, until the resin set. Dentin was exposed on the cervical one-third of the buccal and lingual surfaces of the teeth and the surface was wet polished with 600-grit silicon carbide paper to obtain a flat surface. They were then inspected under a dental operating microscope to ensure that no remnants of enamel were present. Except for a window of 3 × 3 mm dentin surface, the rest of the sample surfaces were painted with nail polish.

Cutting debris was removed by ultrasonically cleaning the samples. They were then randomly allocated into 6 groups (n = 36) on the basis of surface treatment protocol. Samples in group 1 (sound dentin) did not receive any further treatment while those in the rest of the groups were immersed in 0.5% citric acid solution (pH 2.5) for 3 days. At the end of demineralization, the samples were washed with distilled water for 30 min. Samples in group 2 (demineralized dentin) did not receive any further treatment. The samples in groups 3–6 were subjected to a 90 days remineralization treatment regimen. Table 1 summarizes the

Table 1
Grouping, categorization, commercial name and composition of the experimental pastes used.

Group	Category	Commercial name	Composition
3	Sodium fluoride-based	Glister, Amway enterprises, Hong Kong.	Water, sorbitol, hydrated silica, glycerin, propylene glycol, sodium lauryl sulphate, xylitol, sodium carboxymethylcellulose, PEG-8, flavor, titanium dioxide (CI 77891), xanthan gum, sodium fluoride, sodium saccharin, methylparaben, propylparaben, FD&C Blue No. 1 (CI 42090).
4	CPP-ACP-based	GC Tooth Mousse, GC corporation, Japan.	Pure water, glycerol, CPP-ACP, D-sorbitol, CMC-Na, propylene glycol, silicon dioxide, titanium dioxide, xylitol, phosphoric acid, flavoring, zinc oxide, sodium saccharin, ethyl p-hydroxybenzoate, magnesium oxide, guar gum, propyl p-hydroxybenzoate, butyl p-hydroxybenzoate.
5	NovaMin-based	Sensodyne, GSK Group of companies, India.	Glycerin, PEG-8, silica, calcium sodium phosphosilicate (NovaMin), cocamidopropyl betaine, sodium methyl cocoyl taurate, flavor, titanium dioxide, carbopol, sodium monofluorophosphate, sodium saccharin.
6	Non-fluoridated	Meswak, Dabur, India.	Calcium carbonate, sorbitol, water, silica, sodium lauryl sulphate, flavor, miswak extract, cellulose gum, carrageenan, sodium silicate, PVM/MA copolymer, sodium saccharin, sodium benzoate, CI 77891, triclosan.

commercial name, categorization and composition of the experimental pastes used. Paste was made into slurry by dissolving dentifrice (5 g) in water (10 ml). The respective experimental pastes were applied on the exposed dentin and brushed with the help of an electric toothbrush (Oral-B, Procter & Gamble, USA) for 2 min. For the next 3 h, the samples were placed in artificial saliva (Aqwet, Cipla, Mumbai, India). The samples were then immersed in 15 ml of 0.5% citric acid 3 times a day for 2 min each, followed by immersion in 15 ml of artificial saliva for 3 h after every acid challenge. They were brushed with the paste for 2 min again. The samples remained in artificial saliva for the remaining 12 h.

Since, two bonding systems were tested here, 36 samples under each group were randomly allocated into two subgroups ($n = 18$) corresponding to the bonding system used, namely, total-etch (TE, subgroup A) and self-etch systems (SE, subgroup B). Table 2 gives the type, constituents and method of use of the bonding agents and composite. Etching of the exposed dentin surfaces was done using 37% phosphoric acid for the total-etch subgroup and using the acidic primer provided with Clearfil SE bond for the self-etch subgroup. The etchant and the acidic primer were rinsed off with water.

Three samples from each subgroup were picked to get insight into the etching pattern. 2.5% glutaraldehyde in Phosphate Buffer (PB) (pH-7.2) enabled fixing of samples in 4 h. 30–100% alcohol was used to dry the samples. They were examined under a SEM (Hitachi S-3400, Japan) at 1500x.

The remaining 15 samples from each subgroup were used for evaluating the μ SBS of composite resin to dentin. Adhesives from the respective subgroups were applied on the dentin surface following manufacturers' instructions. Translucent polyethylene tubes (one tube per sample) with 0.75 mm inner diameter and 2 mm height were fixed on the dentin surface and the adhesives light cured (3M Unitek 2500 curing light, 3M ESPE, St.Paul, MN, USA). The tubes were filled with composite resin in 1 mm increments and light cured. The specimens remained in distilled water for a day. The polyethylene tubes were slit

open with a scalpel and the set up was then mounted on a universal testing machine (Instron, Canton, U.S.A) for μ SBS analysis. A loop measuring 3 mm was formed with a stainless steel wire. As the load cell secured the loop, the free end of the wire loop was kept in flush with the dentin-composite interface. Load application was done at the rate of 1 mm/min until the composite sheared off from the dentin surface. The load at failure divided by the surface area was taken as the μ SBS value of the sample (in MPa). The fractured samples were viewed under SEM and the failure mode determined as adhesive, cohesive (dentin/resin), or mixed.

2.1. Statistical analysis

The results were tabulated and statistically analyzed using SPSS Statistics 22. Shapiro-Wilk test and Levene test assessed normality and homogeneity of variances of the μ SBS data. Majority of data was found to be non-normal in distribution and variances were heterogeneous in nature. Hence, non-parametric Kruskal Wallis test followed by Dunn's Post Hoc test was applied. The significance was set at $p < 0.05$. Failure modes were analyzed using chi-square test.

3. Results

Fig. 2 shows the representative SEM images of all the groups. The mean μ SBS values (in MPa) of all the groups are given in Table 3. Sound dentin (Group 1) showed the highest mean μ SBS under both the adhesive systems ($p < 0.05$). Among the subgroups A (TE), group 1 (sound dentin, 19.56 ± 0.36) showed significantly higher μ SBS, compared to group 4 (CPP-ACP, 12.12 ± 0.57), group 6 (non-fluoridated, 11.94 ± 0.27), group 2 (demineralized dentin, 11.83 ± 0.43), group 5 (NovaMin, 11.66 ± 0.28) and group 3 (sodium fluoride, 11.56 ± 0.15) ($p < 0.05$).

Among the subgroups B (SE), group 1, (sound dentin, 19.25 ± 1.14) showed significantly higher μ SBS compared to group 4

Table 2
Type, composition and mode of application of the adhesive systems and composite used.

Materials	Type	Composition	Application Mode
Adper Single Bond 2/Plus, 3M ESPE, St.Paul, MN, USA. (Subgroup A)	Two step-etch and rinse adhesive	Bis-GMA, HEMA, silica nanofillers, dimethacrylates, water, ethanol, methacrylate functional copolymer of polyacrylic and polyitaconic acids, novel photoinitiator system	Apply etchant for 15 s. Rinse for 10 s and blot dry excess water with a cotton pellet. Apply two consecutive coats of adhesive for 15 s with gentle agitation. Gently air thin for 5 s. Light cure for 10 s. Apply primer for 20 s. Dry gently. Apply adhesive, light cure for 10 s.
Clearfil SE Bond, Kuraray Co. Ltd., Osaka, Japan. (Subgroup B)	Two step self-etch adhesive	Primer: 2-HEMA, 10-MDP, hydrophilic aliphatic dimethacrylate, camphoroquinone, water, accelerators, dyes. Bond: Bis-GMA, 2-HEMA, 10-MDP, hydrophobic aliphatic dimethacrylate, colloidal silica, camphoroquinone, initiators.	Place the composite resin in increments and light cure each increment for 40 s each.
Filtek Z350, 3M ESPE, St.Paul, MN, USA.	Nanocomposite	Matrix: bis-GMA, UDMA, TEGDMA, bis-EMA Filler: 78.5% combination of agglomerated zirconia/silica cluster filler with primary particle size of 5–20 nm, and non-agglomerated/non-aggregated 20-nm silica.	

Bis-EMA –Ethoxylated bisphenol A dimethacrylate, Bis-GMA–Bisphenol A glycidyl methacrylate, HEMA–2-Hydroxyethyl methacrylate, MDP–10-methacryloyloxydecyl dihydrogenphosphate, TEGDMA–Triethylene Glycol Dimethacrylate, UDMA–Urethane Dimethacrylate.

Fig. 2. Representative SEM images of all the groups. SEM micrographs of 1A (sound dentin/TE) showed complete removal of the smear layer and open dentinal tubules, whereas 1B (sound dentin/SE) had a surface smeared with smear layer. Few partially occluded tubules were seen. Group 2 (demineralized dentin) showed no trace of smear layer. Both the subgroups had deeply demineralized wide-open and funnel-shaped dentinal tubules with exposed collagen fibrils. Loss of peritubular dentin is seen 2B (demineralized dentin/SE), whereas loss of both peritubular and intertubular dentin is appreciable in 2A (demineralized dentin/TE). In groups 3 (NaF) and 4 (CPP-ACP), while subgroup A (TE) showed predominantly open tubules, subgroup B (SE) showed partial occlusion of most of the dentinal tubules with narrowing of the tubular orifices. The surface was free of film or precipitate in group 3, while visible precipitates were seen in group 4. Partial occlusion of most of the dentinal tubules was seen in 5A (NovaMin/TE), while in 5B the tubules remained predominantly occluded. Open tubules were discernible in both 6A (non-fluoridated/TE) and 6B (non-fluoridated/SE), but the surface remained smeared with debris in subgroup B.

Table 3

Mean μ SBS values (in MPa) of all the groups.

Groups	Total-etch (n = 15)	Self-etch (n = 15)
1 (Sound dentin)	19.56 $\pm$ 0.36 ^a	19.25 $\pm$ 1.14 ^a
2 (Demineralized dentin)	11.83 $\pm$ 0.43 ^b	11.80 $\pm$ 0.52 ^b
3 (Sodium fluoride)	11.56 $\pm$ 0.15 ^b	12.40 $\pm$ 0.18 ^{a,b}
4 (CPP-ACP)	12.12 $\pm$ 0.57 ^b	11.97 $\pm$ 0.39 ^b
5 (NovaMin)	11.66 $\pm$ 0.28 ^b	11.97 $\pm$ 0.17 ^{a,b}
6 (non-fluoridated)	11.94 $\pm$ 0.27 ^{a,b}	10.62 $\pm$ 0.11 ^c

*Denotes significant difference among the subgroups ($p < 0.05$). Under each subgroup, different alphabets denote significant difference among the groups ($p < 0.05$).

(11.97 $\pm$ 0.39), group 5 (11.97 $\pm$ 0.17), group 2 (11.80 $\pm$ 0.52) and group 6 (10.62 $\pm$ 0.11) ($p < 0.05$). Group 6 (non-fluoridated) showed the least μ SBS value compared to all the other groups ($p < 0.05$). No significant difference was found between the μ SBS values of groups 1 and 3 (sodium fluoride, 12.40 $\pm$ 0.18) ($p > 0.05$).

Intragroup comparisons showed that group 6 showed significantly higher bond strength under subgroup A, while groups 3 and 5 had significantly higher bond strength under subgroup B ($p < 0.05$). No significant difference was found among the subgroups in groups 1, 2 and 4 ($p > 0.05$).

Among the subgroups A, the fracture pattern was predominantly mixed (50%), followed by adhesive (33.3%) and cohesive in dentin (16.7%). Among the subgroups B, the fracture pattern was equally distributed between mixed (50%) and adhesive (43.3%) types, with the least percentage of cohesive failures in dentin (6.7%). No cohesive fractures were seen in composite. Intragroup and intergroup comparisons of failure patterns revealed no significant difference ($p > 0.05$).

4. Discussion

In the current study, dentin was exposed on the cervical aspect of the buccal and lingual surfaces of the teeth so that it closely relates to

the condition of NCCL's. The demineralization-remineralization cycle was adopted from a previous study.¹⁰ This closely mimics an erosive intraoral environment, in which cycles of demineralization and remineralization occur. In the present study, remineralization was followed for 3 months, since studies have proven that long-term remineralization therapies offer relief from hypersensitivity.^{11,12}

In the present study, Filtek Z350, a methacrylate-based nanofill composite resin designed for use in anterior and posterior restorations was chosen. Qin et al. (2013), showed that Filtek Z350 can be used as a restorative material for an effective clinical performance in NCCL's.¹³

Microshear bond strength testing was chosen, as it is a distinctive test to assess the interfacial strength between tooth and polymeric composite restorative materials. The smaller bonded surface area would allow a more uniform stress distribution, which provides an accurate assessment when compared to the conventional shear test.¹⁴

Sound dentin showed the highest bond strength under both the bonding systems. The polyalkeneic acid copolymer present in Single Bond 2, partially demineralizes the dentin surface and forms complexes of calcium-polyalkeneate on the surface of hybrid layer (HL) that extends 3 μ m into the tubules. This enables hydrolytic stability and absorbs interfacial stresses, thus providing improved bond strength.¹⁵

Self-etch adhesives simultaneously demineralize dentin and subsequently allow permeation of resin monomers into it. Thereafter, apatite crystals remain scattered in the HL. 10-MDP, the acidic monomer in the primer of Clearfil SE Bond, interacts with apatitic calcium forming insoluble salts, which strengthens its bond to dentin.¹⁶

In this study, the μ SBS of composite to demineralized dentin was significantly lesser than to sound dentin. This could be attributed to the loss of minerals from the peritubular and intertubular dentin that compromises the bonding of adhesive monomers to demineralized dentin. The remaining unsupported collagen network serves as water filled channels. This hydrophilic substrate further lowers the bonding ability of adhesive resins.¹⁷

SEM micrographs of CPP-ACP group (4A) showed predominantly open dentinal tubules following phosphoric acid treatment. Ishikawa

et al. (1994) showed that following CPP-ACP application, calcium phosphate (Ca-P) precipitation occurs on the superficial dentin and that the precipitates did not penetrate deep enough into the dentinal tubules in order to effectively occlude them. Also this precipitation occurred so rapidly, that it prevented continuous Ca-P interaction in solution thus, resulting in precipitate formation only on the surface of dentin.¹⁸ The surface debris was sparse as the precipitates formed due to CPP-ACP were dissolved by the use of phosphoric acid as etchant. The low pH (< 1) of phosphoric acid could be attributed to this. Further, the rinsing step might have led to the washing away of the dissolved products, and the superficially deposited minerals, thus resulting in predominantly open dentinal tubules as seen under SEM. This relatively precipitate-free dentin surface could also be the reason behind the higher bond strength of this group under TE system, compared to SE system, though not statistically significant.

In the other groups under TE (NovaMin, sodium fluoride and non-fluoridated), SEM micrographs following etching showed that dentinal tubules were partially occluded, with narrowing of the tubular orifice. Studies have shown that application of remineralizing pastes (NovaMin and NaF) on dentin results in the formation of an acid-resistant, hypermineralized layer.^{19,20} Deposition of minerals from artificial saliva along with the calcium and sparse fluoride content in natural miswak extract could have resulted in this effect in the non-fluoridated group.²¹ This layer might have been a deterrent in obtaining optimal bond strength to remineralized dentin. This could be the reason behind the decreased bond strength seen in the above mentioned groups compared to untreated dentin.

The sodium fluoride group, under SE showed significantly superior bond strength compared to the rest of the groups. Acid conditioning leads to the release and activation of alkaline phosphatases and metalloproteases in dentin. These enzymes degrade the resin-dentin bond. Fluoride released from NaF protects HL from enzymatic degradation.²² This could be the reason behind the improved bond strength seen in this group.

NovaMin, in an aqueous environment, crystallizes into hydroxycarbonate apatite, a chemical and structural equivalent of hydroxyapatite (HA). Thus the residual HA in the hybrid layer enables chemical bonding between its calcium ions and SE monomers. Furthermore, they protect collagen from hydrolytic degradation.¹⁶ This could explain the significantly improved results of this group under SE compared to TE.

Meswak paste was chosen as a non-flouridated toothpaste in this study as the paste does not contain any added fluoride ingredient. Meswak-treated dentin showed the least bond strength in the self-etch subgroup. The reduced bond strength in this group could be attributed to the presence of certain essential oils in miswak extract, which would have interfered with the bonding process.²¹ But, better bond strength was observed in total-etch subgroup, as the residual oil gets washed away in the etch-and-rinse process.

Intergroup comparisons revealed that bond strength to remineralized dentin did not differ significantly from each other under the TE sub group, whereas under the SE subgroup, significantly higher bond strength was seen in samples treated with sodium fluoride and NovaMin compared to those treated with CPP-ACP. Similar findings were reported by Zimmerli et al. (2012), who observed that bonding to eroded dentin is better with SE adhesive system compared to TE system.²³ An *in vitro* study done by Elhassan et al. (2008), assessed the microshear bond strength of a SE adhesive to desensitized dentin. They observed that the μ SBS of control and test groups were similar, irrespective of the use of desensitizing agents.²⁴ This might be attributed to the shorter duration of remineralizing treatment (14 days), which was much lesser than that of the present study. Hence, the null hypotheses of this study stands rejected, as both, the type of remineralizing paste as well as the type of adhesive system used influenced the μ SBS bond strength of composite to dentin.

This study was done to clinically simulate adverse bonding

conditions that prevail in eroded dentin. The results clearly show that bond strength values obtained in demineralized specimens and those treated with remineralizing agents were lesser than those obtained with normal dentin. This study did not reproduce the natural sclerotic dentin encountered in NCCLs, which is a difficult substrate to bond, when compared to normal cervical dentin.²⁵

The interaction of dentin and desensitizers could be affected by numerous aspects like age of source of specimen, smear layer, dentinal tubule orientation, their density, branching, diameter and direction, and existing or absent peritubular dentin. Insufficient bonding could have also resulted from dehydration associated with the use of extracted teeth. Pulpal pressure, which was not simulated here, might also have significant consequence on bonding. Hence, further long-term clinical studies are mandatory to validate the results of this research.

Hence, it can be summarized that bonding to remineralized dentin is compromised owing to the chemical and morphological changes that occur in this substrate. The type of adhesive system used also influences this altered bond strength. While no significant difference was seen among the remineralizing agents under the total-etch category, dentin remineralized using sodium fluoride and NovaMin showed significantly higher bond strength under the self-etch subgroup.

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Declaration of competing interest

None.

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