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The shear bond strength of bidirectional and random-oriented fibre-reinforced composite to tooth structure

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KEYWORDS
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Summary *Objectives*. The objective of this study was to evaluate the bond strength and fracture pattern of fibre-reinforced composite (FRC) with two different fibre orientations and matrix compositions to dentine and enamel.

Materials and methods. Extracted human molars were used as substrates (enamel and dentine) with a standard acid-etch technique. Light-polymerizable FRC with two different interpenetrating polymer network matrices and random or bidirectional fibre orientations was applied to the substrate, together with the adhesive resin. As a control, particulate filler composite resin was bonded to the substrates. The substrate–composite specimens ($n=10$) were either stored in water for 24 h or additionally thermocycled for 6000 cycles. The shear bond strength of composite to substrate was measured and the fracture surfaces were evaluated visually and with SEM.

Results. Three-way factorial analysis of variance highlighted significant differences according to the substrate type, storage condition and composite material ($p<0.05$). Dentine specimens showed a significantly lower range of bond strength values (8.8–15.0 MPa), compared with enamel specimens (14.0–23.0 MPa). The highest mean bond strength in dentine was 15.0 MPa obtained with bidirectional FRC, whereas the highest bond strength in enamel was 23.0 MPa obtained with random-oriented FRC. Thermocycling did not identify a significant effect on the dentine bond strength, but did identify a significant decrease in enamel bond strength values ($p<0.05$). Several cohesive failures were observed in the tooth structure with the control material, whereas no cohesive bulk fracture of the tooth was observed when a thin layer of FRC was placed at the interface.

Conclusions. The addition of bidirectional or random continuous fibres did not show any significant improvement in bond strength values compared to control of particulate filler. However, the difference in the fracture patterns observed may have implications for clinical applications.

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Introduction

During the last decade, fibre-reinforced composite (FRC) was introduced as a new material for a treatment alternative in aesthetic, metal-free dentistry.[1–3] FRCs can be used for the fabrication of laboratory-made single crowns and partial or full coverage fixed partial dentures,[4] as well as chair-side periodontal splinting,[5] adhesive fixed partial dentures,[6] and post-core systems,[7] and in orthodontic applications.[8]

Resin-preimpregnated FRC has been shown to possess adequate flexural modulus and flexural strength to function successfully in the oral cavity.[9] The performance of the FRC system depends on the cohesive strength of the polymer matrix as well as fibre type, volume fraction, and the quality of the fibre–polymer matrix interface.[10] In addition to the mechanical performance, the composition of the polymer matrix and fibres also has a major role in the bonding ability and durability of FRC to the luting resin cements at the tooth-restoration interface. Therefore, various fibre and polymer matrix compositions have been developed.[11,12] It has been concluded that preimpregnation of the fibres with the light polymerizable resin system by the manufacturer is of importance to optimize the properties.[13] The preimpregnation is based on using either photopolymerizable dimethacrylate monomer resin only, or on using a combination of dimethacrylate monomer resin and linear polymer, which forms semi-interpenetrating polymer network (semi-IPN) after being polymerized.[13–15] The rationale for using a semi-IPN polymer matrix over the dimethacrylate system is in the highly viscous resin, which improves the handling properties of the FRC and the adhesive properties of the veneering composite and luting resin cements to the FRC. The FRC with a semi-IPN resin matrix can be adhered with resin composites by means of interdiffusion bonding.[15,16] Two different kinds of semi-IPNs are available for dental use; one requires further-impregnation by resin and the other is fully preimpregnated.[14–16]

The employment of fibres with different fibre orientations may also change the dynamics of the adhesive interface and result in a modification of the interfacial bond failures. Previous studies have evaluated the effect of the addition of continuous unidirectional FRC at the tooth-restoration interface and have shown that the bond strength of chair-side fabricated continuous unidirectional FRC did not differ from that of particulate filler composite.[17] However, besides the use of continuous unidirectional FRC, also woven (bidirectional) FRC and wool (random orientation) FRC have specific applications in dentistry. To the authors' knowledge, there are as yet no studies evaluating the effect of fibre orientations and matrix compositions on the bond strength of FRC to tooth structure.

Thus, this study was designed to obtain information about the behaviour of different fibre orientations, namely, bidirectional and random FRC, and matrix compositions at the adhesive interface to dentine and enamel.

Materials and methods

Specimen preparation

One hundred and sixty extracted, human third molars free of visible caries obtained from 18–25 year-old donors were used within one month of extraction. Upon collection, adhering soft tissues and blood were removed under running water and the teeth were stored in frozen form until used. During the specimen preparation, each tooth was embedded in autopolymerizable acrylic resin (Palapress, Heraus Kulzer, Wherheim, Germany) and stored in water at room temperature. Enamel bonding sites were prepared on the buccal surfaces, while dentine bonding sites were prepared on the occlusal surfaces. Bonding surfaces were ground flat using 1000 grit silicon carbide abrasive paper (SiC, Struers, Copenhagen, Denmark) at 300 rpm, under water-cooling, using an automatic grinding machine (Rotopol-11, Struers) at a force of 20 N within the limits of superficial dentine. At this stage, specimens that showed any visible pulp exposures or cracks were excluded from the study, and all materials to be tested were used according to the manufacturers' recommendations. Ten teeth were randomly assigned to each group.

Bonding procedure

The adhesive resin agent, the particulate filler resin and the FRCs used in this study are listed in Table 1. The bonding sites on each substrate surface were etched for 15 s using a 35% phosphoric acid gel (Ultra etch, Ultradent products, USA). Subsequently, the substrate surface was rinsed thoroughly for 15 s with water. Enamel substrates air-dried for 5 s and dentine substrates were gently blotted with absorbent paper, leaving a visibly moist dentine surface. Dentine primer was applied to the substrate surface and air-dried gently for 5 s, leaving the surface shiny. After primer application on the enamel or dentine, the adhesive resin was

Table 1 Materials used in this study.

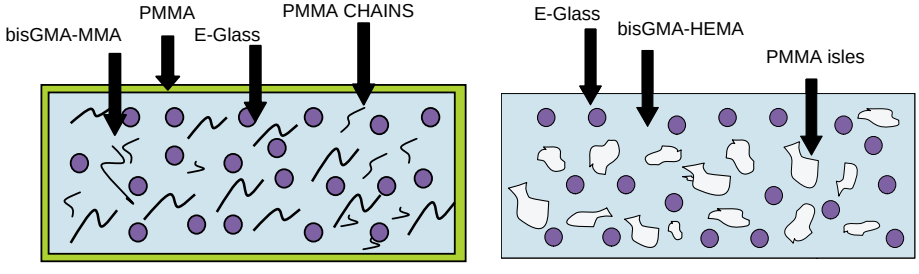
Materials	Code	Lot no	Manufacturer	Material composition
ScotchBond multi-purpose adhesive and primer	SB	Adhesive.20000907, primer: 20001120	3M, St. Paul, MN, USA	Adhesive: HEMA, bisGMA, primer: HEMA, water, vitrebond copolymer
StickNet (bidirectional FRC)	SN	2020218-W-0042	StickTech, Turku, Finland	Porous PMMA, E-glass fibers
Filtek Z250	Z250	20000523	3M, St. Paul, MN, USA	bisGMA, UDMA, bis-EMA 60% vol fillers
EverstickNet (bidirectional FRC)	EN	2040122-EN-061	StickTech, Turku, Finland	PMMA, bisGMA, E-glass fibres
Experimental random FRC	EW	-	StickTech, Turku, Finland	PMMA, bisGMA, E-glass fibres

bisGMA, bisphenol A-glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; bis-EMA, bisphenol A polyethylene glycol diether dimethacrylate; PMMA, polymethylmethacrylate; HEMA, hydroxyethylmethacrylate; FRC, fibre reinforced composite; E-glass, E-glass fibres, silanated.

applied together with one layer of bidirectional (woven) or random-oriented (wool) FRCs, pressed to the etched and primed surface of the tooth with a silicon instrument (Refix D, StickTech, Turku, Finland), and light-polymerized for 10 s with a hand-held light-curing unit (Elipar, ESPE, Seefeld, Germany). The wavelength of the unit was between 380 and 520 nm. Light intensity was 650 mW/cm^2 according to the radiometer (Optilux Radiometer Model-100 SDS Kerr, Danbury, CT, USA). Before application of fibres, StickNet (SN) fibres were further-impregnated with an adhesive resin (SB) for 10 h in a dark container, whereas everStickNet (EN) fibres were ready to be used without further impregnation. In the further-impregnated SN FRC, there were larger polymethylmethacrylate (PMMA) isles in the cross-linked matrix than in the EN FRC (Fig. 1). Following the surface treatment, each of the 2 mm increments of particulate filler composite Z250 (Table 1) was built up onto the FRC layer to a height of 4.0 mm using a polyethylene mould with an inner diameter of 3.6 mm, and polymerized with a hand-held light-curing unit (Elipar, ESPE) for 40 s. Subsequently, every surface treatment group was divided into two subgroups according to the storage conditions, with 10 specimens in each subgroup. These subgroups were stored either in water (Grade 3) at 37 °C for 24 h or, following the water storage, additionally thermocycled for 6000 cycles between (5 ± 2) and (55 ± 2) °C with a dwell time of 30 s and a transfer time of 5 s, according to ISO TR 11405. The thermocycling process was completed in four and a half days.

Shear bond test

Twenty-four hours after thermocycling, a shear bond strength test was performed using a testing machine (model LRX, Lloyd Instruments, Fareham, England) at room temperature (23 ± 2) °C. The specimens were secured in a mounting jig (Bencor Multi-T shear assembly, Danville Engineering Inc., San Ramon, CA) with the shearing rod against and parallel to the flat prepared bonding sites (Fig. 2). The bonded adherend was loaded at a crosshead speed of 1.0 mm/min until fracture, and shear bond



A: Ready-to-use FRC (EN)

B: Further-impregnated FRC having PMMA isles in the polymer matrix

Figure 1 Schematic illustration of two different types of polymer matrix composition of FRCs used in this study namely A: the polymer matrix composition of EN FRC and B: the matrix composition of SN FRC.

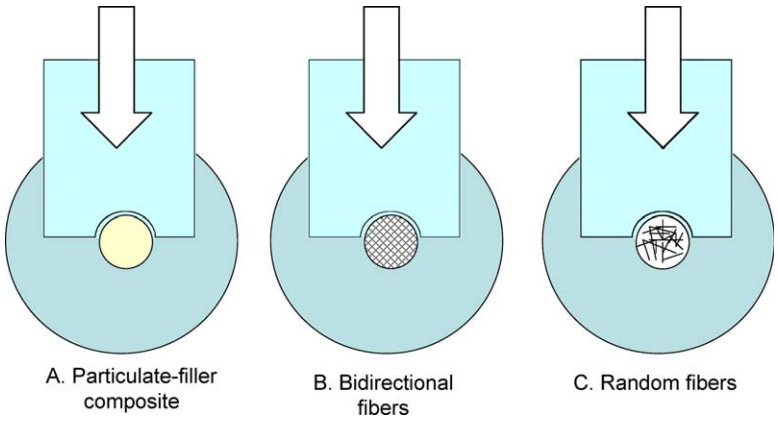


Figure 2 Schematic illustration showing adhesive materials on substrate for the shear bond strength test. Arrow shows the direction of the load.

strengths were calculated by dividing the highest fracture force (N) with the area of the adherend (diameter 3.6 mm) and recorded in MegaPascals (MPa) using PC software (Nexygen, Lloyd Instruments Ltd, Fareham, England). The specimens were stored in water, except for the period of testing. Debonded areas were examined visually for their failure region and were also gold-sputtered at a vacuum of 0.05 mbar under argon atmosphere, and observed under a scanning electron microscope (JSM 5500, Jeol Ltd, Tokyo, Japan).

Statistical analysis

The data for all the groups were analysed statistically with SPSS 11.0 (Statistical Package for Statistical Science, SPSS Inc., Chicago, IL, USA).

Three-way factorial analysis of variance (ANOVA) was used to investigate the influence of substrate (2 levels), adherent (4 levels) and the storage condition (2 levels) on the shear bond strength. Post-hoc testing was accomplished with the Tukey test. The level of significance was set at $\alpha = 0.05$.

Results

The means and standard deviations of shear bond strength of the groups are shown in Fig. 3. Three-factor ANOVA showed significant differences according to the substrate type, storage condition and composite material ($p < 0.05$). Some interactions between the factors were also significant ($p < 0.05$).

In particular, as regards to substrate, dentine specimens showed significantly lower values of bond strength (8.8–15.0 MPa), compared with enamel (14.0–23.0 MPa).

When the substrate was dentine, the storage condition did not show any significant effect on the bond strength values ($p > 0.05$), whereas the material had a significant effect ($p < 0.05$). On the dentine substrate, the lowest shear bond strength was obtained with ready-to-use bidirectional FRC (EN) at a range of 10 MPa, whereas the highest bond strength was obtained with the further-impregnated bidirectional FRC (SN) at a range of 15 MPa.

In contrast to dentine, both the storage condition and the material used had a significant effect on the bond strength to the enamel substrate ($p < 0.05$). The bond strength values ranged from the highest values of 23 MPa for the random-oriented FRC (EW) after water storage, to the lowest value of 9.8 MPa for the further-impregnated group (SN) following the thermocycling.

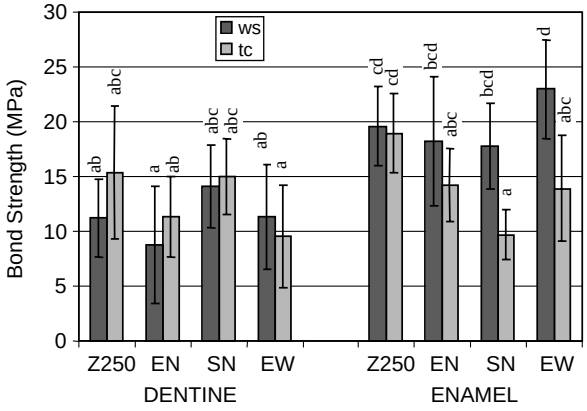


Figure 3 The means and standard deviations of shear bond strengths according to storage conditions to dentine and enamel with different material combinations. (The vertical lines represent standard deviations.) For abbreviations, see Table 1. Values designated by the same superscript letter are not statistically different at $p = 0.05$.

Table 2 Results of the analysis of fracture mode for the shear bond test ($n=20$/group).

Substrate	Material	Adhesive		Cohesive		
		Tooth/resin	Resin/FRC	Within FRC	Within tooth	In resin
Dentine	Z250	2	0	0	6	12
	EN	0	0	15	0	5
	SN	0	0	20	0	0
	EW	0	0	18	0	2
Enamel	Z250	0	0	0	5	15
	EN	0	0	16	0	4
	SN	0	0	0	20	0
	EW	0	0	17	0	3

For abbreviations, see Table 1.

The shear bond strength values obtained following bonding to enamel after water storage with the use of random FRC (EW) were higher compared with other groups (23 MPa). However, after thermocycling, the shear bond strengths had a tendency to decrease in all groups for the enamel substrate. For the dentine substrate, the bond strength values for the FRC did not show any significant difference compared to the particulate filler composite control group ($p>0.05$). At the enamel, only the SN group showed significantly lower bond strength compared with other groups. The bond strength of EN, EW and the control did not show a significant difference ($p>0.05$). There was a significant difference in the bond strengths between the two bidirectional FRC materials depending on the substrate type ($p<0.05$). EN showed better bonding properties to enamel compared with SN, which attached better to dentine.

The evaluation of the fracture patterns of the specimens is shown in Table 2. The control group of particulate filler composite resin showed a mixed type of failures, including fractures cohesively inside the tooth substrate, cohesively within the resin close to the enamel or dentine surface, or adhesively, at the enamel/dentine interface. After failure, most of the bidirectional FRC (SN, EN) test specimens remained attached to the tooth surface, whereas the random-oriented groups fractured totally and were torn away from the surface. SEM evaluation of the fracture surfaces confirmed the visual analysis (Fig. 4A and B) whereby bidirectional (SN) and random-oriented FRC (EW) displayed mostly cohesive fractures within the FRC.

Discussion

This study was designed to evaluate the effect of different fibre orientations and two variations of polymer matrix composition on the shear bond strength of FRC to dentine and enamel. The rationale for comparing bidirectional and random-oriented FRC with a simulated direct application came from their potential to be used in adhesive restorations by increasing the bonding surface area on enamel and dentine. Moreover, it

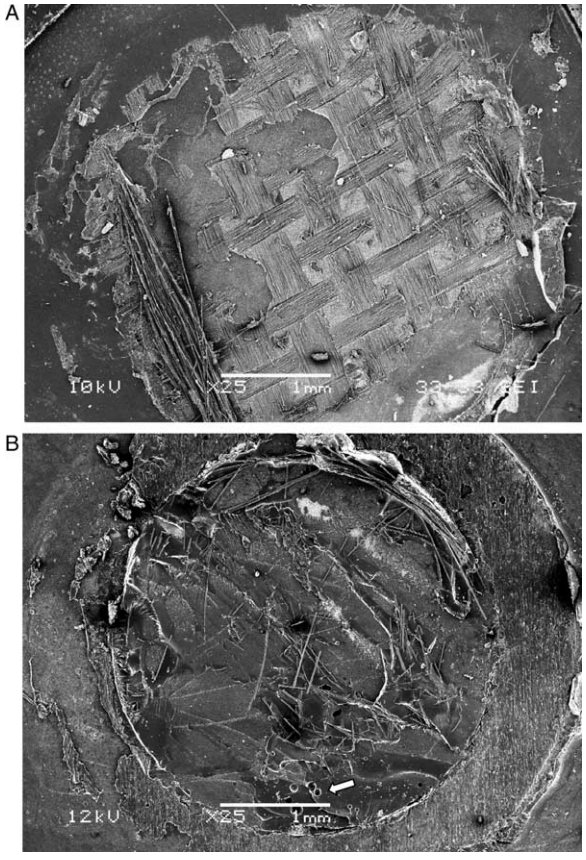


Figure 4 SEM photomicrographs of the dentine substrate after shear bond strength test. (A) The bidirectional FRC showed cohesive failures within the FRC layer resulting in splitting of the fibres. 90% of the specimens showed this mode of failure. (B) The random FRC showed cohesive failures within FRC. Arrow shows the air bubbles trapped inside the FRC matrix. 85% of the specimens showed this mode of failure.

could be that the properties of FRCs can possibly mimic the biomechanics of tooth structure better than unidirectional FRCs.

For the direct application of the FRC on the substrate surface, an adhesive system (SB) containing hydroxyethylmethacrylate (HEMA) was selected for this study. Previous studies in the literature have shown that low molecular weight HEMA can effectively penetrate to the linear phases of the semi-IPN matrix and thus enhance the bonding.[14,15,18] Furthermore, as the bonding system does not contain ethanol or acetone, the possible solvent action of the adhesive system on the FRC material, which might compromise the bonding, could be avoided.

Despite its well known limitations,[19] the shear bond test set up has been the most commonly employed laboratory technique for evaluating the bond strength of adhesives and resin-bonded restorations. A notable feature of the studies evaluating shear bond strength tests is the observation that the failure mode is predominantly cohesive within the substrate,[20] and this was attributed to the nature of the stresses generated and their distribution within the adherence zone.[19–21] This study demonstrated that the use of bidirectional FRC close to the adhesive interface did not show a significant improvement in bond strength to enamel or dentine substrate, but did change the path of crack propagation at the interface. As there were several cohesive failures observed in the tooth structure with the control material, no cohesive bulk fractures of the tooth were observed when a thin layer of FRC was placed at the interface.

There was a significant difference in the bond strength values where the substrate was concerned. It should be noted that as a substrate, enamel and the dentine have different elastic moduli. Previously, it was suggested that the elastic modulus of the substrate has an effect on the associated bond strength values, and that the differences in elastic moduli can result in changes in the stress distribution with a shear test set up.[22] This is particularly important since dentine with a lower elastic modulus compared with enamel might be expected to show relatively higher bond strength values. This means that differences according to the substrate might be even higher than obtained in the present study. On the other hand, the relatively low bond strengths obtained in this study might be explained by differences in material combinations, test set up and operator factors, all of which are known to affect the results.[23,24]

The bond strength of further-impregnated bidirectional FRC group SN was higher in combination with dentine. The visual and SEM evaluation of the bonding surfaces showed that loading produced cohesive failures within the FRC layer. When the shear load was applied to the interface, the composite block stayed attached to the FRC, resulting in splitting of the fibre layer. This finding is consistent with the previous studies showing good adhesion between FRC and the particulate filler composite.[14,16,18] On the other hand, the other half of the fibres also remained attached on the tooth surface.

It is known that the failure behaviour of FRCs is very complex because of the anisotropy of the material. The results of the present study are in good agreement with a previous report[25] which suggested that the fibres in the adhesive area may not necessarily increase or can even reduce the static adhesive strength. Moreover, the internal discontinuities at the interface can cause a redistribution of the stress fields and act as an energy-absorbing mechanism. As the crack propagates from one fibre to another, the micro-cracks and fibre breakages can absorb energy and thus decrease the energy of the final fracture. In contrast to the control group which produced some cohesive failures in the substrate, and irrespective of the bond strength obtained between the groups, the FRC groups did not show cohesive fractures continuing inside the substrate, indicating that the addition of the fibres at the interface and changing the path of crack propagation, may be of importance especially at the unsupported tooth structures, and may be used to reinforce and prevent catastrophic failures of the tooth structure.

Another important point to consider is the fibre volume fraction of the FRC materials. The fibre volume fraction of the random-oriented FRC group was lower compared to the bidirectional FRC group. Therefore, the authors hypothesized that the randomly orientated FRC group with a rough surface and a higher fraction of polymer matrix might ease the penetration of the monomers inside the material, thereby offering a better bonding site for the substrate and particulate filler composite. However, this hypothesis was not completely confirmed within the limitations of this study. The bond strength values using the random FRC group were high only at the enamel surface after water storage; however, after thermocycling, the decrease in bond strength values was notable.

Among the bidirectional FRCs, the bond strength of EN was lower compared with SN for the dentine substrate. However, EN showed higher bond strength in enamel groups. This might be due to the different matrix compositions of EN and SN.

Because of the further-impregnation with SB adhesive, SN also contains HEMA in the matrix composition which is known to attach well to the dentine structure.[26,27]

The visual evaluation of the bonding surfaces showed less splitting of the fibres when EN was used, and this may be due to the different polymer matrix structure of EN. Before polymerization, EN has a more homogeneous matrix, containing PMMA polymer chains in bisphenol A-glycidyl dimethacrylate resin, which might advantageously increase the cohesion of the matrix, as well as increasing the toughness. The preimpregnation polymer matrix of the SN group has PMMA isles, and with the further-impregnation by dimethacrylate monomers, a multiphasic polymer matrix is formed. Even though the multiphase semi-IPN polymer matrix is known to be beneficial for interdiffusion bonding,[15] the cohesive strength of this matrix combination may be lower than that of EN FRC. Also, possible air voids[17] in the manually further-impregnated FRC might cause stress concentration points that could result in a weakening of the polymer matrix.

There was a slight increase in dentine bond strength of some groups after thermocycling, however this increase was not statistically significant. This finding was in accordance with previous studies. This might be due to the mechanical interlocking to the dentine tubules because of the high water uptake of the HEMA-containing adhesive layer. Furthermore, water uptake of the adhesive layer might lead to a plasticizing effect, which might act as a stress breaker at the bonding interface.

In contrast to dentine, thermocycling did show significant reduction in enamel bond strength. This was contradictory compared to the results of a previous study which showed an around 10% increase in enamel shear bond strength after thermocycling when unidirectional FRC was used.[17] This effect was even clearer when the FRC had a ready-to-use matrix. However, with the same test set up and polymer matrix composition, the difference in the behaviour of unidirectional and bidirectional or random FRCs may be attributed to their anisotropic, orthotropic or isotropic behaviour with regard to thermal changes[28] rather than their matrix structures. However, this hypothesis needs further investigation.

Within the limitations of the present study it can be concluded that the addition of bidirectional or random continuous fibres did not show any significant improvement in bond strength values compared to control of particulate filler. But, the variation in fibre orientation did change the crack propagation at the interface.

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