Effect of Storage Time on Microtensile Bond Strength of Short Glass Fibre-Reinforced Composite

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Objective: *To evaluate the microtensile bond strength (μ-TBS) of short glass fibre-reinforced composite (FC) to dentine and the reliability of two adhesive systems and influence of water storage on bond strength.*
Methods: *Experimental FC was prepared by mixing short glass fibres with a fraction of 22.5 wt% and IPN-resin 22.5 wt% with silane-treated particulate fillers 55 wt% using a high-speed mixing machine. FC composite and conventional restorative composite resins (PFC) (Grandio; control) were bonded incrementally to flat, midcoronal dentine from 40 human molars, using two different adhesives systems (total-etch [Scotchbond] and self-etch [Futurabond]). Teeth were sectioned both mesiodistally and buccolingually to obtain multiple bonded beam specimens. The specimens were stored in water at 37°C for 1 month or 6 months before being tested in tension at a crosshead speed of 0.5 mm/min until failure. The failure mode was analysed by SEM. The data were analysed using ANOVA.*
Results: *The bond strength of experimental FC did not differ ($p > 0.05$) from conventional PFC. Bond strength values were significantly higher ($p < 0.05$) with the total-etching system than with the self-etching system. Water storage decreased the bonding values.*
Conclusion: *Short-FC with semi-IPN polymer matrix revealed similar bonding capacity to conventional PFC.*
Key words: *fibre-reinforced composite, microtensile bond, restorative composite*

With the advances in dentine adhesives and the evolution of aesthetic dentistry in the 1990s, composites have become more widely used in posterior restorations. After many significant material improvements, restorative composite resins still suffer from two key shortcomings: mechanical strength deficiencies and polymerisation shrinkage. Thus, advanced research has been undertaken to improve composite resins in order to create a material with high strength and low polymerisation shrinkage, keeping in mind the requirements of aesthetic properties. Attempts have been made to change the type of fillers or filler size and their surface silanisation. By changing the polymerisation kinetics of resins, attempts have been made to influence the matrices and degree of monomer conversion[1,2]. Reinforcing the resin with short glass fibres[3], with fibre-reinforced composite (FC) substructure[4,5], whiskers[6], particulate ceramic fillers (dense and porous)[7] and optimisation of filler content are among the methods that have been studied[1]. However, further significant improvements are still needed. In the polymerisation process of composite resin, the chemical reaction that occurs in the organic phase of the composite converts monomers into polymers, with consequent shrinkage. The extent of this shrinkage influences the tension stage generated at the interface between composite and dental structure and commonly compromises the bond integrity in this region. To enhance the marginal integrity of composite resin restorations, bonding agents are used to withstand the polymerisation contraction forces. The generation of

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adhesive systems developed according to the total-etch concept are applied using a multi-step procedure. More recently, further types of adhesive systems have been developed in order to simplify and reduce the stages of application (etch and rinse adhesive in two steps or self-etching systems in either two or one steps, according to the classification of Van Meerbeek et al[8]). Several studies have demonstrated that the resin–dentine bond strength of adhesives decreases after water storage[9,10]. This decrease has been partly explained by the plasticisation effect of water at the hybrid layer or degradation of dentine collagen. From these viewpoints, microtensile bond strength (μ-TBS) tests are used as an *in vitro* indicator of the marginal seal.

Glass fibres have been investigated in reinforcing dental polymers for over 30 years[11]. They have documented reinforcing efficiency and good aesthetic qualities compared with carbon or aramid fibres[12]. The effectiveness of fibre reinforcement is dependent on many variables, including the resins used, the quantity of fibres in the resin matrix[13,14], length of fibres[13], form of fibres[15], orientation of fibres[16], adhesion of fibres to the polymer matrix[17], and impregnation of fibres with the resin[18]. Short random fibres provide an isotropic reinforcement effect in multidirections instead of one or two directions, as described by Krenchel[19].

Polymethyl methacrylate (PMMA)- and dimethacrylate-based semi-interpenetrating polymer network (semi-IPN) matrix has been established as a polymer matrix in denture base materials[20]. Also some products of FC use semi-IPN polymer in the matrix[21].

Recently, the use of short glass fibres in combination with semi-IPN matrix in restorative filling composite has been reported, with encouraging results[22,23]. In addition, the use of short-FC led to improvements in polymerisation shrinkage stress and marginal microleakage values compared with the conventional particulate filler restorative composite (PFC)[24]. However, μ-TBS of short fibre-reinforced dental composite resin with semi-IPN-polymer matrix has not been evaluated.

Thus, the aim of the present study was to measure the μ-TBS of composite resin of glass FC with semi-IPN polymer matrix. In addition, the effects of two different adhesive systems and water storage periods on bonding strength were evaluated.

Materials and methods

Materials

Dimethacrylate (BisGMA 67% [bisphenol A-glycidyl dimethacrylate] and TEGDMA 33% [triethyleneglycol dimethacrylate]) resin consisting of 50 wt% nanofillers (SiO_2, 20 nm in size) (Hanse Chemie, Germany) and E-glass fibres with BisGMA-PMMA (polymethylmethacrylate, molecular mass 22 x 10^4) resin matrix (everStick, StickTech Ltd, Turku, Finland). In addition, radio-opacity fillers of $BaAlSiO_2$ (3 ± 2 μm in size) (Specialty Glass, USA) were incorporated to the resin system. Before the $BaAlSiO_2$ filler particles were incorporated into the resin matrix, they were silane-treated using a previously defined technique[25]. Commercial particulate filler composite (Grandio Caps, VOCO, Germany) was used as a control group.

Methods

Experimental FC was prepared by mixing 22.5 wt% of short E-glass fibres (3 mm in length) with 22.5 wt% of resin matrix and then 55 wt% of $BaAlSiO_2$ radiopacity-fillers was added gradually to the mixture. The mixing was carried out using a high-speed mixing machine for 5 min (SpeedMixer, DAC, Germany; 3500 rpm). The dimethacrylate-based resin matrix consisting of PMMA forms a semi-IPN polymer matrix for the composite of FC.

Forty extracted, sound and caries-free human molar teeth with similar occlusal size were selected. Upon collection, adhering soft tissues and blood were removed under running water and the teeth were frozen in wet gauze for a period not exceeding 3 months.

The teeth were mounted into an acrylic block (diameter 2.5 cm) below the cementoenamel junction using autopolymerised acrylic resin (Palapress, Heraus Kulzer, Wehrheim, Germany). The occlusal surface was ground flat using 1000-grit (FEPA) silicon carbide abrasive paper (Struers, Copenhagen, Denmark) at 300 rpm under cooling water using an automatic grinding machine (Struers Rotopol-11). Grinding was visibly controlled until all enamel was ground away and the superficial dentine exposed. Teeth that showed any visible pulp exposures or cracks were excluded from the study. The teeth were divided into two groups (n = 20) according to the composite resins (FC or PFC). Each of the groups was further separated into two groups (n = 10) according to the adhesive systems: total-etching (Scotchbond, 3M ESPE, St Paul, MN, USA) and self-etching (FuturaBond NR, VOCO, Cuxhaven, Germany). Adhesives were applied in accordance with manufacturers' instructions. After the application of the adhesives to dentine, 6 mm high resin composite build-ups were constructed incrementally (2 mm) with FC or PFC. Each layer of composite was light-activated with a hand-light-curing unit (Optilux–501, Kerr, CT, USA) for 40 s

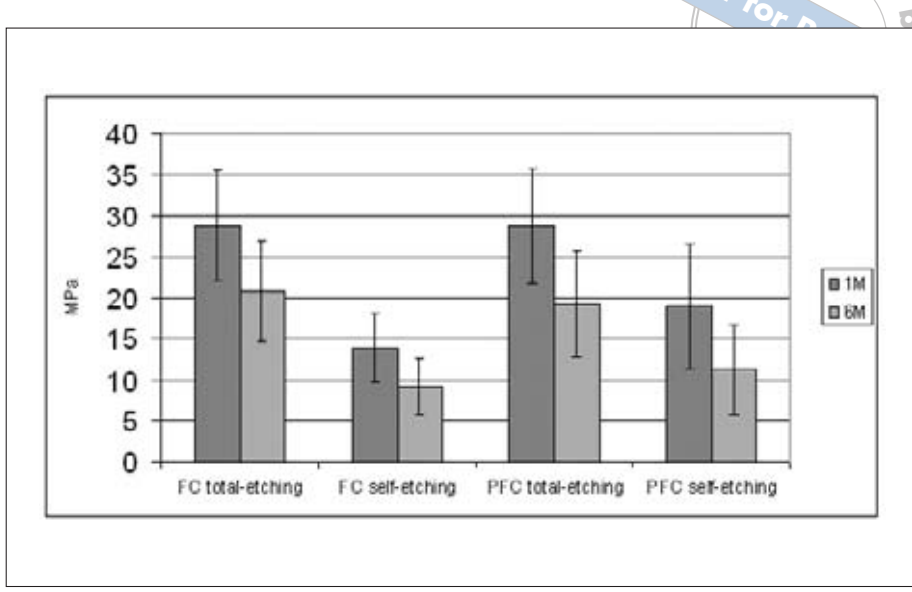

Fig 1 Microtensile bond strength of experimental fibre-reinforced composite and conventional restorative composite using two different adhesive systems. Vertical lines represent standard deviations. M refers to water storage time (months) at 37°C.

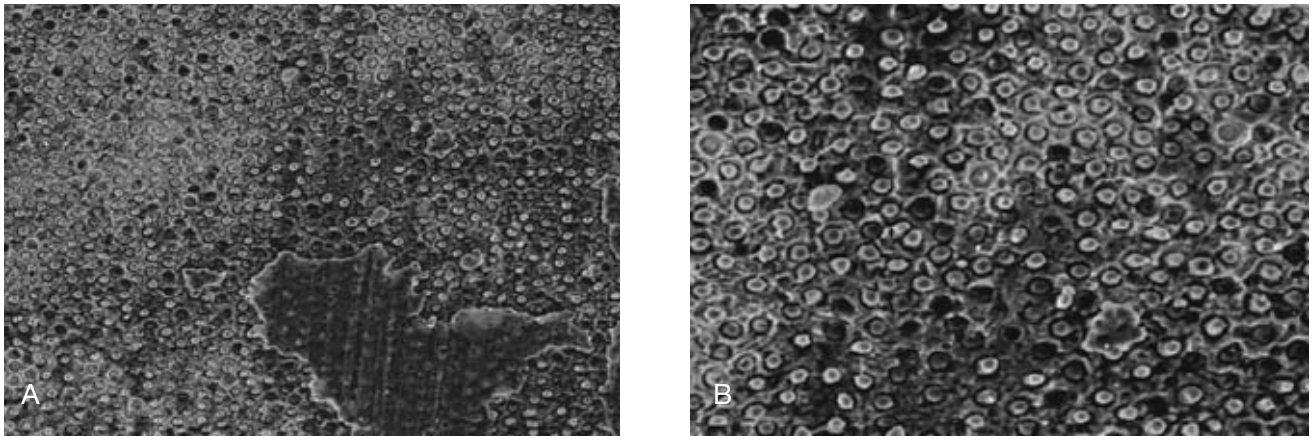

Fig 2 SEM observations of the fractured surface along the dentine side of one specimen.
(A) Mixed failure is observed. Most of the surface is covered by an adhesive layer although some dentine is exposed.
(B) At a higher magnification of the exposed dentine, resin tags in the tubule entrances are observed.

(wavelength: 380 and 520 nm with maximal intensity at 470 nm, light irradiance 800 Mw/cm^2). After the preparation of resin-bonded specimens, each tooth was serially sectioned into rectangular beams with cross-sectioned areas of 1 ± 0.2 mm^2 using a slow-speed diamond saw (Ernst Leitz GMBH, Wetzlar 1600, Germany). These resin-bonded beams were stored in water at 37°C either for 4 weeks or 6 months before testing. After that the specimens were attached to a micro-tensile tester (Microtensile Tester, Bisco) with cyanoacrylate glue (Zapit; DVA, AnaHeim, Calif, USA) and subjected to microtensile testing at a crosshead speed of 5 mm/min until they fractured. The fractured beams were removed from the testing apparatus and the cross-sectional area at the site of failure was measured to the nearest 0.01 mm with digital calipers. μ-TBS values were expressed in MPa.

Scanning electron microscopy (SEM, model 5500, Jeol Ltd., Tokyo, Japan) was used to evaluate the failure mode.

Statistical analysis

The results were statistically analysed with analysis of variance (ANOVA) at the $p < 0.05$ significance level with SPSS version 13 (Statistical Package for Social Science, SPSS Inc, Chicago, IL, USA).

Results

The mean μ-TBS values with standard deviations (SD) of the tested groups are summarised in Fig 1. The experimental FC showed no statistically significant difference ($p > 0.05$) in μ-TBS compared with the conventional composite resin (control). The μ-TBS values were significantly higher ($p < 0.05$) with the total-etching system than with the self-etching system. Water storage decreased the μ-TBS values in all specimens ($p < 0.05$). Most of the failures were adhesional in nature at the composite–dentine interface for both adhesives and composites used.

Discussion

Currently, the performance of biomaterials is most often evaluated using laboratory tests. The shear bond test is an important technique selected by the International Standard Organization (ISO) for screening the bonding of resin-based restorative materials to tooth structure[26]. However, it is likely that bond strength of composite to dentine is dictated by the properties of the adhesive system rather than the composite used. Early laboratory experiments on the use of semi-IPN matrix in combination with short E-glass fibres in restorative filling composite showed enhancement in flexural strength, load-bearing capacity, and polymerisation shrinkage[22-24]. Therefore, it was important to evaluate the microtensile bond strength of experimental FC. The results of this study showed that microtensile bond strength and failure modes of experimental FC were similar to the conventional filling composite (Fig 1). Based on the results of present study and our previously published data of short FC, it is suggested that experimental FC could be used successfully to fulfil the requirements for the ideal posterior restoration. However, it should be emphasised that clinical trials are necessary in order to evaluate the usefulness of FC resin in dental restorations.

There have been several studies on the effectiveness of the self-etching adhesive systems and their adhesion to dentine, and controversial results have been reported about the bonding performance of these systems[27,28]. Our results were in agreement with previous studies[27,29], which showed that specimens made with the self-etching adhesive system suffered lower bond strength values than specimens with the total-etching adhesive system. This is probably because self-etching adhesive has limited demineralisation and impregnation depths due to wet dentine and ionic effects of high calcium and phosphate concentrations, which limit the apatite crystal dissolution[30]. Another factor that may contribute to the success of PFC restorations is water sorption. Direct exposure of the specimen beams to water storage for 6 months resulted in a significant reduction in the microtensile bond strength compared with specimen beams stored for 1 month. In polymer matrix, water acts as a plasticiser, increasing free volume and decreasing glass transition temperature of the polymer matrix in a manner that ultimately weakens resin–dentine bonds over time[31]. Also, it has previously been reported that the negative effects of water storage on bond strengths of the resin–dentine interface are due to hydrolytic degradation of the collagen fibres in the hybrid layer[9,10]. However, many studies reported that water sorption could cause gap reduction by hygroscopic expansion over time[32].

The failures in the specimens restored with both adhesive systems occurred predominately within the hybrid layer (Fig 2), which in turn may be considered the weakest portion of the bonded interface. Further studies are needed to evaluate the factors that may affect the mechanical properties of the hybrid layer.

Conclusion

E-glass fibre-reinforced composite resin with semi-IPN-polymer matrix has similar dentine bond strength values compared with the conventional particulate filler restorative composite.

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