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Short Glass Fiber-reinforced Composite with a Semi-interpenetrating Polymer Network Matrix for Temporary Crowns and Bridges

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Abstract

Aims: The purpose of this study was to investigate the reinforcement effect of short E-glass fiber fillers on some mechanical properties of temporary crown and bridge (TCB) composite resin with a semi-interpenetrating polymer network (semi-IPN).

Methods and Materials: Experimental temporary fiber reinforced (TFC) composite resin was prepared by mixing 15 wt% of short E-glass fibers (3 mm in length) with a 35 wt% of semi-IPN-resin (dual or chemical cure) with 50 wt% of silane treated particulate silica fillers using a high speed mixing device. Temporary crowns (n=6) and test specimens (2 x 2 x 25 mm^3) (n=6) were made from the experimental TFC and conventional TCB composite (control, Protemp Garant, 3M-ESPE, St. Paul, MN, USA). A three-point bending test was done according to ISO standard 10477, and a compression loading test was carried out using a steel ball (Ø 3.0 mm) with a speed of 1.0 mm/min until fracture occurred. The degree of monomer conversion (DC%) of both composites was determined by Fourier transfer infrared (FTIR) spectrometry.

Results: The analysis of variance (ANOVA) revealed both dual and chemical cure experimental TFC composite resins had statistically significant ($p<0.05$) higher flexural strengths (117 and 99 MPa, respectively) and compressive load-bearing capacity (730 and 623 N, respectively) compared to the control TCB composite resin (72 MPa, 549 N).

Conclusion: The use of short fiber fillers with semi-IPN polymer matrix yielded an improved mechanical performance compared to a conventional TCB composite resin.

Keywords: Fiber composite, temporary crown and bridge composite, fiber reinforce composite

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Introduction

Quality temporary crown and bridge (TCB) restorations are an important part of prosthetic treatment. Both direct and indirect methods of fabrication of these temporary restorations can be used. The indirect method has been associated with a better fit and mechanical strength, but time constraints and inadequate laboratory support have led to the use of the direct method.[1] Ideal TCB materials should have a high mechanical strength, dimensional stability, biocompatibility, adequate esthetic properties, favorable marginal adaptation, and easy handling characteristics.[2]

Resin composites have been developed for the fabrication of temporary restorations. Advantages of the resin composite include: ease of manipulation, even directly into the oral cavity with some monomer systems; a low exothermic setting reaction; and decreased shrinkage during polymerization. However, one of the main shortcomings of temporary composite resins is frequent fractures of the restoration under masticatory loads[3] and a poor surface gloss of the material.[4]

Fracture strength and marginal adaptation are two material related parameters that are important for clinical performance. Fracture strength provides an estimate of the performance of a restoration under masticatory forces.[5] Marginal adaptation is a measure of the polymerization shrinkage of a material when a TCB restoration is cemented on a tooth for a long period of time.[6]

Glass fibers have been investigated as a reinforcement component of dental polymers for more than 30 years.[7] Studies have documented reinforcement efficiency and esthetic qualities compared to carbon or aramid fibers.[8-10] The effectiveness of fiber reinforcement is dependent on many variables including the following:

- The resins used
- The quantity of fibers in the resin matrix[11,12]
- Fiber length[12]
- The form of the fibers[13]
- Orientation of the fibers[14]
- The adhesion of fibers to the polymer matrix[15]
- The degree of impregnation of fibers with the resin[16]

Short random fibers provide an isotropic reinforcement effect in multiple directions instead of one or two directions as described by Krenchel.[17]

Polymethyl methacrylate-based (PMMA) semi-interpenetrating polymer network (semi-IPN) matrix has been established as a polymer matrix in denture base materials.[18] Also, some fiber reinforced composite (FRC) products use a semi-IPN-polymer matrix.[19]

Recently the use of a semi-IPN matrix in combination with short glass fibers in restorative filling composites has shown encouraging results.[20,21] However, TCB composite resin with semi-IPN-polymer matrix in combination with glass fibers has not been evaluated to the authors' knowledge. Thus, the aim of this study was to investigate the reinforcing effect of short E-glass fiber fillers on some mechanical properties of TCB composite resin with semi-IPN-polymer matrix.

Methods and Materials

The materials used in the study are listed in Table 1. Experimental temporary crown and bridge fiber composite (TFC) was prepared by mixing 15 wt% of short E-glass fibers (3 mm in length) and 35 wt% of semi-IPN formatting resin

Table 1. Materials used in the study.

Brand	Manufacturer	Lot no.	Composition
Protemp Garant	3M ESPE, St. Paul, MN, USA	B 258254	Dimethacrylate-based material
everStick	StickTeck Ltd, Turku, Finland	2050426-ES-125	PMMA, Bis-GMA
Stick Resin	StickTeck Ltd, Turku, Finland	540 1042	60% Bis-GMA-40% TEGDMA

PMMA, poly methyl methacrylate, M_w 220.000
Bis-GMA, bisphenol A-glycidyl dimethacrylate.
TEGDMA, triethylenglycol dimethacrylate.

system (dimethacrylate-PMMA) either with both chemical and photo activated resin matrix (dual cure) or with just a chemical activated resin matrix. After these mixtures were created, then 50 wt% of filler particles of SiO2 (3±2 µm in size) were gradually added and mixed using a high speed mixing device for 15 minutes (SpeedMixer, DAC, Germany, 3000 rpm). Before the silica filler particles were incorporated into the resin matrix, they were silane treated, according to the technique defined in a previous study.[22] During polymerization, the resin matrix of dimethacrylate-PMMA formed semi-IPN polymer matrix for the experimental TFC composite.

A metal die was fabricated simulating an upper premolar with 2.0 mm of axial and occlusal reduction. A transparent template matrix of an ideally contoured crown was used to facilitate standardized crown construction. The crowns (n=6/group) were fabricated from both the dual and chemical cure experimental TFC composite and commercial TCB composite. The commercial TCB composite is a dual cure material mixed according to the manufacturer's instructions using a distributor tip. The experimental dual cure TFC composite and commercial TCB crowns needed additional curing with a hand-light curing unit (Optilux–501, Kerr, CT, USA) for 40 seconds (wavelength: 380 and 520 nm with maximal intensity at 470 nm, light irradiance 800 mW/cm^2).

A static compressive fracture test was performed using a Model LRX universal testing machine (Lloyd Instruments Ltd, Fareham, UK) at a speed of 1 mm/min, and data were recorded using PC software (Nexygen Lloyd Instruments Ltd). After polymerization, the metal die with the crown was fixed to an inclined metal base to provide a 45º

Figure 1. Position of loading tip on the temporary crown and the compressive load test setup.

angle between the occlusal surface of the tooth and the loading tip (spherical Ø 3 mm) (Figure 1). The loading event was registered until the crown fractured.

Three-point bending test specimens (2 x 2 x 25 mm^3) were made from experimental TFC composite and conventional TCB composite resins. Specimens were made in a half-split stainless steel mold between transparent Mylar sheets. Specimens from each group (n=6) were stored dry at room temperature for 24 hours before testing. The three-point bending test was conducted according to the ISO 10477 standard (test span: 20 mm; cross-head speed: 1.0 mm/min; and indenter: 2 mm diameter). The test was

performed using the same universal testing machine and recording software that was used for the static compressive test.

Flexural strength (σf) and flexural modulus (Ef) were calculated from the following formula:[23]

$$\sigma_f = 3F_m I /(2bh^2)$$

$$E_f = SI^3 /(4bh^3)$$

Where,
F_m is the applied load (N) at the highest point of load-deflection curve
I is the span length (20.0mm)
b is the width of test specimens and
h is the thickness of test specimens.
S is the stiffness (N/m) S=F/d and
d is the deflection corresponding to load F at a point in the straight-line portion of the trace.

Toughness was calculated as the integral of the area under the stress/strain curve and reported in units of MPa.

The degree of monomer conversion (DC%) of both composites during and after polymerization was monitored using Fourier transform infrared spectroscopy (FTIR) (Spectrum One, Perkin Elmer, Beaconsfield Bucks, UK) with an attenuated total reflectance (ATR) sampling accessory. Materials were inserted into 1.8 mm-thick disc molds with a diameter of 6.5 mm and then placed on the ATR-sensor (ZnSe-crystal). The upper surface of the specimen (n=3) was covered with a Mylar sheet and a glass slide of 1 mm thickness and slightly pressed against the ATR sensor to ensure a good contact of the specimen.

In case of dual cured composite, the substrate was light-polymerized with a hand-held light–curing unit (Optilux–501, Kerr, CT, USA) for 40 seconds. The spectra during the polymerization process was recorded every 30 seconds for 15 minutes. The DC% was calculated from the aliphatic C=C peak at 1638 cm^{-1} and normalized against the aromatic C=C peak at 1608 cm^{-1} according to the following formula:

$$DC\% = \left[1 - \frac{C_{aliphatic} / C_{aromatic}}{U_{aliphatic} / U_{aromatic}}\right] 100\% \quad (1)$$

Where,
- **$C_{aliphatic}$** is at an absorption peak at 1638 cm^{-1} of the cured specimen
- **$C_{aromtic}$** is at an absorption peak at 1608 cm^{-1} of the cured specimen
- **$U_{aliphatic}$** is at an absorption peak at 1638 cm^{-1} of the uncured specimen
- **$U_{aromtic}$** is at an absorption peak at 1608 cm^{-1} of the uncured specimen

The fraction of remaining double bonds for each spectrum was determined by standard base line techniques using the comparison of maximum heights of aliphatic and reference peaks for the calculations.

Mean values of load-bearing capacity and flexural strength were statistically analyzed 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) to determine differences between the groups.

Results

The mean flexural strength, load-bearing capacity, and degree of conversion of all tested specimens along with standard deviations (SD) are summarized in Table 2. An ANOVA revealed dual and chemical cure experimental TFC composite resin had statistically significantly higher flexural strength (117 and 99 MPa) and compressive load-bearing capacity (730 and 623 N), respectively, compared to the control TCB composite resin (72 MPa, 549 N) ($p<0.05$). The ANOVA also showed the experimental dual cure TFC composite had a statistical significantly higher flexural strength and load-bearing capacity compared to chemical cure experimental TFC composite ($p<0.05$).

The degree of monomer conversion after 15 minutes of dual cure experimental TFC composite was 49.6% (1.9), chemical cure experimental TFC composite 43.7% (1.3), and TCB composite 53.5% (1.5).

Discussion

This pilot study evaluated some of the mechanical properties, which are important parameters related to the clinical performance for TCB

Table 2. Mean values with standard deviations (SD) of test specimens.

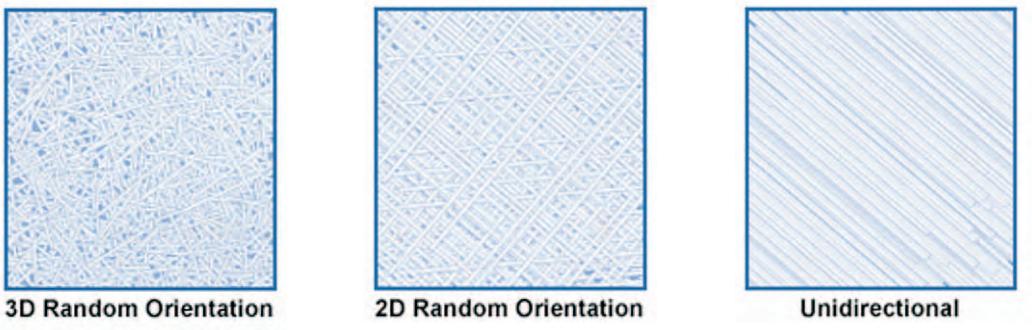
Groups	Flexural strength (MPa)	Compressive load bearing capacity (N)	Degree of monomer conversion (DC%)
Experimental dual cure TFC	117 (11)	730 (168)	49.6 (1.9)
Experimental chemical cure TFC	99 (20)	623 (160)	43.7 (1.3)
TCB (Control)	72 (10)	549 (90)	53.5 (1.5)

TFC = Experimental temporary fiber reinforced composite resin.
TCB = Commercial temporary crown and bridge composite resin.

composite resins. The results of the mechanical test revealed substantial improvements in load bearing capacity and flexural strength of TCB composite reinforced with short E-glass fiber filler in comparison with conventional TCB composite resin.

FRCs from a group of materials with high toughness and strength were of interest in this study. Currently, the interest in using FRC is growing, and acceptable success rates have been reported when they were used to reinforce definitive and long-term temporary restorations like fixed partial dentures.[24,25] In order for a fiber to act as an effective reinforcement for polymers the transfer of stress from the polymer matrix to the fibers is essential.[26,27] This can be achieved if the fibers have a length equal or greater than the critical fiber length.[26,28] Critical fiber length has been measured using a fiber fragmentation test to determine the critical lengths of E-glass with Bis-GMA polymer matrix vary between 0.5 and 1.6 mm.[29] Deteriorated or initially poor adhesion between the fibers and polymer matrix increase the critical fiber length. In this case the mechanical friction of fibers to polymer matrix at the interface can compensate for poor adhesion.[30] Based on this phenomenon, the present study used fibers 3.0 mm in length as fillers. According to Krenchel short fibers randomly orientated in three dimensions provide a strengthening factor of 0.2, whereas a two-dimensional orientation yielded a 0.38 factor and unidirectional fibers yielded a strengthening factor of 1.0. Strength values of a unidirectional FRC in a 3-point bending test are reported to be between 800-1200 MPa.[31]

The flexural test has been widely used to characterize the mechanical properties of dental materials.[27,32] In the present study the specimens of the experimental TFC composite resin had a flexural strength and load-bearing capacity higher when compared to conventional TCB composite resin. The reinforcing effect of fiber fillers is based on stress transfer from the polymer matrix to fibers and on the function of individual fibers as “crack stoppers.” Random fiber orientation and lower cross-linking density of the polymer matrix by the semi-IPN structure may have played a role in the improved mechanical properties.



The FTIR has proved to be a useful technique for the analysis of the degree of monomer conversion in dental composites.[33] Dual cure commercial TCB showed a higher degree of conversion than experimental TFC composite. This could be explained by the lower filler content and type of filler used. An increase in the filler content can limit the mobility of free radicals in the polymer matrix.[34] However, some of the difference could also be explained by differences between the polymer matrices of pure thermoset and semi-IPN.

Polymerization shrinkage of TCB restorations causes a marginal gap between the temporary crowns and teeth.[35] Efforts for reducing the polymerization shrinkage have included modification of resin compositions using higher filler contents to reduce the amount of polymerizable resin.[36] The authors hypothesize the use of fiber fillers could reduce the polymerization shrinkage of the composite, and this will have to be evaluated in further studies.

Conclusions

Short glass fiber reinforced composite resin with semi-IPN polymer matrix for use as TCBs demonstrated an improvement in mechanical properties compared with conventional temporary composite resin. This suggests the possibility of a better performance of short glass fiber reinforced composite resin in high stress-bearing application areas.

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