Mechanical Properties and Wear of Five Commercial Fibre-Reinforced Filling Materials

Sufyan GAROUSHI[1], Pekka K. VALLITTU[1,2], Lippo LASSILA[1]

Objective: *To evaluate and compare certain mechanical properties and wear of five commercial short-fibre reinforced composites (Alert, EasyCore, Build-It, TI-Core, and everX Posterior), in relation to their microstructural characteristics.*
Methods: *Fracture toughness, work of fracture, and flexural strength were determined for each material following ISO standards. The specimens (n = 6) were dry stored (37 °C for 2 days) before they were tested. A wear test was conducted with 15,000 chewing cycles using a dual-axis chewing simulator. Wear pattern was analysed by a three-dimensional (3D) noncontact optical profilometer. Scanning electron microscopy (SEM) was used to evaluate the microstructure of each composite material. The results were statistically analysed using ANOVA followed by post hoc Tukey's test.*
Results: *everX Posterior exhibited the highest fracture toughness (2.4 MPa $m^{1/2}$) among the materials tested ($P < 0.05$). EasyCore presented the highest flexural strength (125.4 MPa), which was not significantly different ($P > 0.05$) from Alert (119 MPa) and everX Posterior (120 MPa). Lowest wear values were found for EasyCore and Build-It (19 and 22 µm). TI-Core showed significantly higher wear depth (45 µm) than all other materials ($P < 0.05$).*
Conclusion: *Significant differences between commercial short-fibre reinforced composites were found for fracture toughness and wear.*

Key words: *fracture toughness, short fibre composite, wear*
Chin J Dent Res 2017;20(3):137–143; doi: 10.3290/j.cjdr.a38768

The use of light-cured conventional particulate composite (PFC) resins for restoring cavities and build-up core foundations in stress-bearing areas has increased rapidly in recent years[1]. Besides the ability to bond to hard tooth tissues, mediated by adhesive systems, they have the advantage of natural shade and are cheaper compared with cast gold and ceramic restorations. However, insufficient material properties limit the success of composite restorations in high stress-bearing areas. Fracture within the body (bulk) and margins of restorations and secondary caries have been cited as major problems regarding the failure of posterior composites[2]. The fracture-related material properties, such as fracture resistance, deformation under occlusal load, and the marginal degradation of materials have usually been evaluated by the determination of the basic material parameters of fracture toughness and flexural strength[3].

Fracture toughness is a mechanical property that describes the resistance of brittle materials to the catastrophic propagation of flaws under an applied load and, thus, it describes the material's tolerance to damage[4]. Fracture toughness values depend on the physical properties and chemical composition of the individual component of restorative material. A material with high fracture toughness has the ability to better resist crack initiation and propagation. Consequently, the properties of fracture toughness and flexural strength become important criterions in the longevity of dental materials[3-5]. Fracture toughness of conventional PFC

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1 Department of Biomaterials Science and Turku Clinical Biomaterials Center –TCBC Institute of Dentistry, University of Turku, Turku, Finland.
2 City of Turku Welfare Division, Oral Health Care, Turku, Finland.

Corresponding author: Dr Sufyan GAROUSHI, Department of Biomaterials Science, Institute of Dentistry and TCBC, University of Turku, Itäinen Pitkäkatu 4 B, FI-20520, Turku, Finland. Tel: 358-233338379. Email: sufgar@utu.fi

This study belongs to the research activity of BioCity Turku Biomaterials Research Program (www.biomaterials.utu.fi).

Table 1 The SFRC resins investigated and their composition.

Brand	Manufacturer	Type	Composition
Alert	Jeneric/Pentron, Wallingford, CT, USA	LC packable	Filler (conventional and micro glass fiber) 84 wt%, 62 vol%
EasyCore	SpofaDental, Markova, Czech Republic	DC flowable	Bis-GMA, HDMA, glass fibre
Build-It	Jeneric/Pentron, Wallingford, CT, USA	DC flowable	Bis-GMA, UDMA, HDMA, 67.3 wt% Boroaluminosilicate glass and chopped glass fibre
TI-Core	Essential Dental Systems, Hackensack, NJ, USA.	AC packable	Bis-GMA, titanium and lanthanide reinforced 75 wt%
everX Posterior	GC Corp, Tokyo, Japan	LC packable	Bis-GMA, PMMA, TEGDMA, discontnuous E-glass fiber filler, Barium glass 74.2 wt%, 53.6 vol%

PMMA, polymethylmethacrylate; Bis-GMA, bisphenol-A-glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; HDMA, hexanediol dimethacrylate; LC, light cured; DC, dual cured; AC, auto cured; wt%, weight percentage; vol%, volume percentage.

resins is still lower than that of dentine[6]. Furthermore, the microstructure of PFC resin does not resemble that of dentine. Conventional PFC resin consists of filler particles embedded in a resin matrix, while dentine consists of collagen fibres embedded in a hydroxyapatite matrix. Therefore, dentine should be rather seen as a fibre-reinforced composite.

The requirement to strengthen dental composite has led to an ever-increasing research into reinforcement techniques. Several former approaches dealt with the incorporation of ceramic particle reinforced (random orientation), whisker (single or multi-layer) or fibre reinforced (continuous or discontinuous fibres in various orientations)[7-9]. A number of manufacturers have developed short fibre reinforced composite (SFRC) resins that claimed to be a solution for conventional PFC resins weakness. To the authors' knowledge, until now there are only five SFRC resins available on the market. These SFRC resins perhaps mimic structurally the fibrous structure of dentine and are recommended for use as bulk base or core build-up materials in large cavities of either vital or non-vital posterior teeth[10-14].

Earlier formulations of SFRC resin (Alert, Jeneric/Pentron) were already commercialised in the late 1990s as packable composites and a new type of SFRC resin (everX Posterior, GC Corporation, Tokyo, Japan) was launched globally in 2013. Although *in vitro* studies have shown good mechanical and physical performance of some SFRC resins (everX Posterior and Alert), compared with conventional PFC resins, the mechanical properties and wear of these SFRC materials have never been compared. Many of the properties of fibre-reinforced composites are strongly dependent on microstructural parameters, such as fibre diameter, fibre length, fibre orientation, fibre loading, and adhesion of fibres to the polymer matrix[15]. Because of the development of newer materials on the market, clinicians are often uncertain about choosing the best materials to achieve optimum results. A comparative evaluation of mechanical properties of available SFRC materials would help the clinician to select better products. Therefore, the aim of this study was to evaluate and compare certain important mechanical properties and wear of five commercial SFRC resins (Alert, EasyCore, Build-It, TI-Core, and everX Posterior), in relation to their microstructural characteristics.

Materials and methods

The short fibre-reinforced composite (SFRC) resins used in the study are listed in Table 1. All materials were manipulated according to the manufacturers' recommended directions.

Mechanical tests

Three-point bending test specimens ($2 \times 2 \times 25$ mm^3) were made from each tested composite. Bar-shaped specimens were made in a half-split stainless steel mould between transparent Mylar sheets. Polymerization of the light and dual-cured resin was done using a hand light-curing unit (Elipar S10, 3M ESPE, St Paul, MN, USA) for 20 s in five separate overlapping portions from both sides of the metal mould. The wavelength of the light was between 430 and 480 nm and light intensity was 1600 mW/cm^2. For auto-cured material, specimens were kept in their moulds for 10 min before being carefully removed.

The specimens from each material (n = 6) were stored dry at 37°C for 48 h before testing. The three-point bending test was conducted according to the ISO 4049 (test span: 20 mm, cross-head speed: 1 mm/min, indenter: 2 mm diameter). All specimens were loaded in a material testing machine (model LRX, Lloyd Instruments, Fareham, England, UK) and the load-deflection curves were recorded with PC-computer software (Nexygen 4.0, Lloyd Instruments).

Flexural strength (σ_f) was calculated from the following formula (ISO 1992):

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

Here, F_m is the applied load (N) at the highest point of a load-deflection curve, I is the span length (20 mm), b is the width of test specimens and h is the thickness of test specimens.

Single-edge-notched-beam specimens (2.5 × 5 × 25 mm^3) according to adapted ISO 20795-2 standard method (ASTM 2005) were prepared to determine the fracture toughness. A custom-made stainless steel split mould was used, which allowed the specimens to be removed without force. An accurately designed slot was fabricated centrally in the mould extending until its mid-height, which enabled central location of the notch and optimisation of the crack length (x) to be 0.5. The restorative material was inserted into the mould and placed over a Mylar-strip-covered glass slide in one increment. Before polymerisation, a sharp and central crack was produced by inserting a straight-edged steel blade into the prefabricated slot. Polymerization of the composite was carried out for 20 s in five separate overlapping portions. The upper side of the mould was covered with Mylar strip and glass slide from both sides of the blade, before being exposed to the polymerization light. Upon the removal from the mould, each specimen was polymerized, again on the opposite side. The specimens from each group (n = 6) were stored dry at 37°C for 48 h before testing. The specimens were tested in the three-point bending mode, in a universal material testing machine at a crosshead speed of 1.0 mm/min.

The fracture toughness was calculated using the equation:

$$K_{max} = [P\ L / B\ W^{3/2}]\ f(x),$$

$$\text{where: } f(x) = 3/2x1/2\ [1.99\text{-}x\ (1\text{-}x)\ (2.15\text{-}3.93x\text{+}2.7x2)] / 2(1+2x)\ (1\text{-}x)3/2$$

$$\text{and } 0 < x < 1 \text{ with } x = a/W.$$

Here P is the maximum load in kilonewtons (kN), L is the span length (20 cm), B is the specimen thickness in centimetres (cm), W is the specimen width (depth) in cm, x is a geometrical function dependent on a/W and a is the crack length in cm.

Work of fracture (the energy required to fracture the specimen) was calculated from the area under the load-displacement curve of single-edge-notched-beam specimens and reported in units of Ncm.

Wear test

Two specimens of each commercial SFRC resin was prepared in an acrylic resin block for localised wear testing. Longitudinal cavities (20 mm length × 10 mm width × 3 mm depth) were prepared and then SFRC materials were placed in one increment into the prepared cavities and covered with the Mylar strips and glass slides before being light irradiated for 40 s in five separate overlapping portions. The surfaces were then polished flat using a sequence of #1200- to #4000-grit silicon carbide papers. For the control group, flat human enamel specimens (n = 4) were produced as previously described[16] by abrading the buccal aspect of caries-free human 3rd molars, collected as approved by the local ethics committee.

After 1 day of water storage (37°C), a 2-body wear test was conducted using the chewing simulator CS- 4.2 (SD Mechatronik, Feldkirchen-Westerham, Germany), which has two chambers simulating the vertical and horizontal movements simultaneously with water. Each of the chambers consists of an upper sample holder that can fasten the loading tip with a screw and a lower plastic sample holder in which the SFRC specimen can be embedded. The specimens were embedded in acrylic resin in the lower sample holder, for use as antagonistic wear materials. The manufacturer's standard loading balls were embedded in acrylic resins in the upper sample holders, and were then fixed with a fastening screw. A weight of 2 kg, which is comparable to 20 N of chewing force and 15,000 loading cycles with a frequency of 1.5 Hz, were used.

The wear patterns (n = 4) on the surface of each specimen were profiled with 3D optical microscope (Bruker Nano GmbH, Berlin, Germany) using Vision64

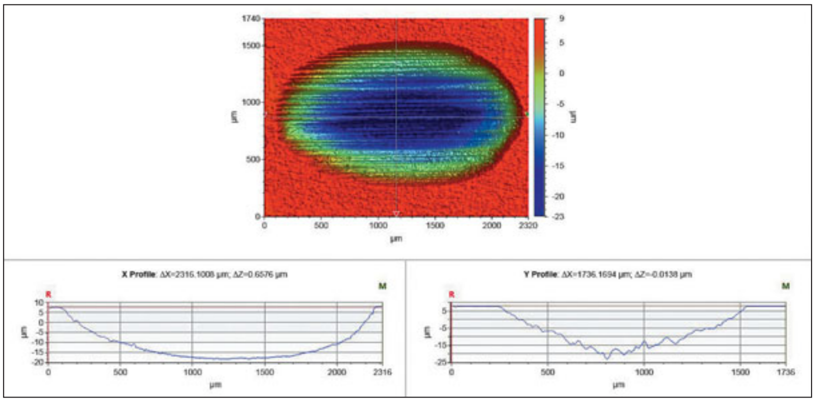

Fig 1 Typical 3D surface profile of the wear pattern where wear depth was measured.

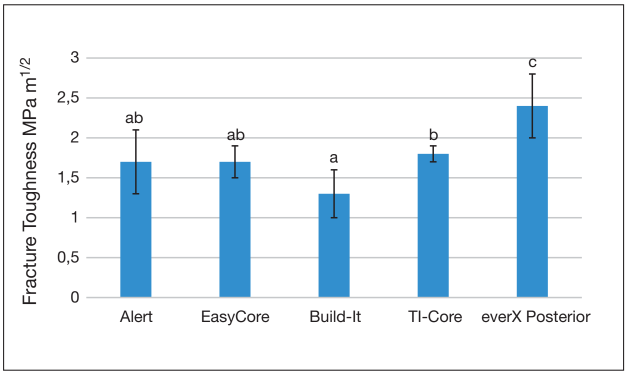

Fig 2 Bar graph illustrating means fracture toughness (KIC) and standard deviation (SD) of investigated SFRC materials. The same letters above the bars represent non-statistically significances ($P > 0.05$) among the groups.

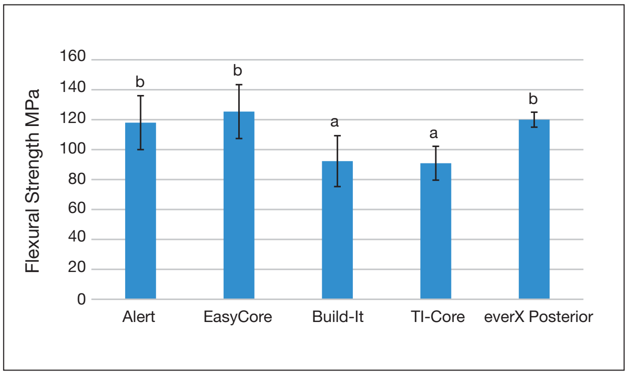

Fig 3 Bar graph illustrating means flexural strength (MPa) and standard deviation (SD) of investigated SFRC materials. The same letters above the bars represent non-statistically significances ($P > 0.05$) among the groups.

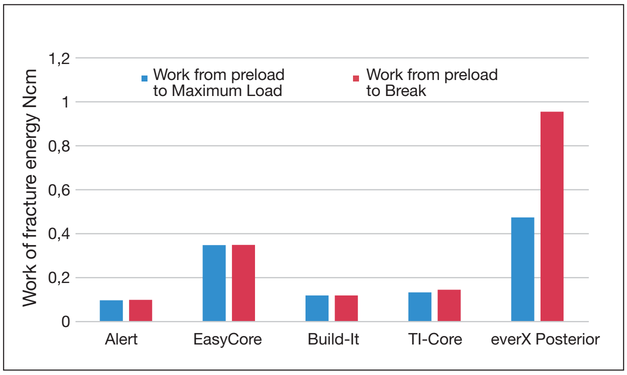

Fig 4 Bar graph illustrating work of fracture energy (Ncm) from preload to maximum load and extension of investigated SFRC materials.

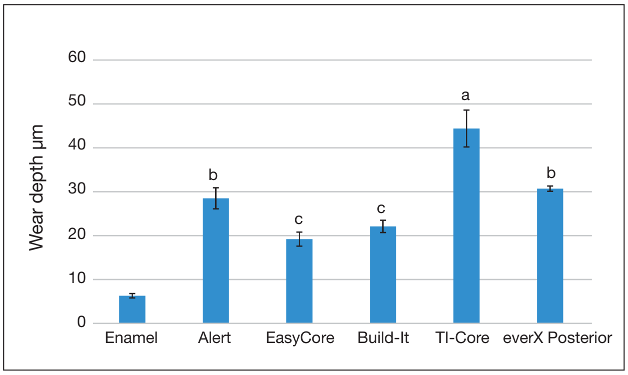

Fig 5 Bar graph illustrating wear depth (micron) of investigated SFRC materials and enamel (reference) after 15,000 cycles of 2-body wear test. The same letters above the bars represent non-statistically significances ($P > 0.05$) among the groups.

software. The maximum wear depth values (µm), representing the average of lowest or deepest points of all profile scans, were calculated from different points (Fig 1).

Microscopic analysis

Scanning electron microscopy (SEM, JSM 5500, Jeol Ltd, Tokyo, Japan) provided the characterisation of the microstructure of the investigated SFRC materials. The specimens (n = 3) from each group were gold sputter coated before the SEM examination.

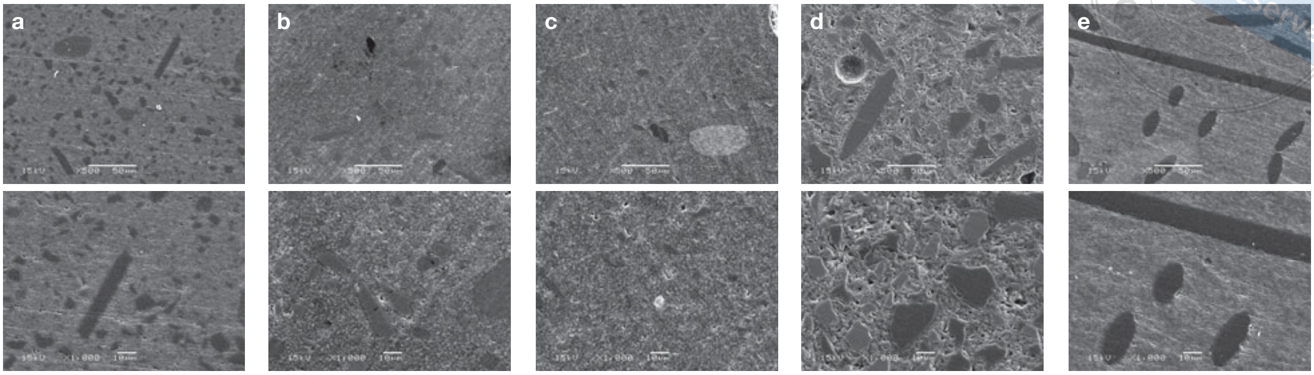

Fig 6 SEM photomicrographs of non-fracture polished surface of investigated SFRC materials. a) Alert; b) EasyCore; c) Build-It; d) TI-core; e) everX Posterior.

Statistical analysis

The data were statistically analysed with SPSS version 23 (SPSS, IBM Corp) using analysis of variance (ANOVA) at the $P < 0.05$ significance level followed by a Tukey HSD post hoc test to determine the differences between the groups.

Results

The mean values of fracture toughness and flexural strength for tested SFRC materials with standard deviations (SD) are summarised in Figures 2 and 3. ANOVA revealed that everX Posterior had statistically significantly higher fracture toughness (2.4 MPa $m^{1/2}$) than all other tested composite materials ($P < 0.05$). EasyCore presented the highest flexural strength (125.4 MPa), which was not significantly different ($P > 0.05$) from Alert (119 MPa) and everX Posterior (120 MPa). A pronounced increase in work to fracture energy was found for everX Posterior over the other tested materials (Fig 4). The mean values for wear depth recorded for each material after 15,000 chewing simulation cycles are show in Figure 5. Lowest wear values were found for EasyCore and Build-It (19 and 22 µm). Alert and everX Posterior showed similar wear values (28 to 30 µm). Only TI-Core showed significantly higher wear (45 µm) than all other materials ($P < 0.05$). SEM analysis showed typical microstructure of each tested material with different fillers (particle/fibre) size, loading and orientation in polymer matrix (Fig 6). This suggested an explanation for different toughening capability between tested materials.

Discussion

Five different commercially available short fibre reinforced composite (SFRC) resins were evaluated in this study. All of them were manufactured to be used in high stress-bearing areas and were presented in order to enhance the fracture resistance of posterior composite restorations and build-up foundations. A large variation in the loading and constitution of fillers can be seen (Table 1) in the different commercial SFRC resins tested.

The fracture toughness of a material is a measure of how well that material hinders the progress of a crack or flaw under load. Fibre impedes the extension of a crack and develops interlocking bridges behind the progressing crack dissipating energy by fibre pullout resulting in graceful rather than catastrophic failure[11]. Short fibres enhanced the ability of the material to resist the fracture propagation, as well as to reduce the stress intensity at the crack tip from which a crack propagates in an unstable manner. As a consequence, an increase work of fracture energy and fracture toughness can be expected. A recent systemic review by Heintz et al showed that fracture toughness is mostly correlated with clinical fracture of composite resins, and no correlations were observed between clinical outcomes and flexural modulus or flexural strength of these materials[3].

In the present study, millimetre scale discontinuous or short fibre reinforced composite (everX Posterior) resin showed relatively high fracture toughness (2.4 MPa $m^{1/2}$) compared with all other materials. This finding is in agreement with several studies, which reported superior fracture toughness values of everX Posterior in comparison with many commercial hybrid and bulk-fill composite resins[10,11,17]. To our knowledge there are no other dental composites with fracture toughness values above 2.4 MPa $m^{1/2}$. Data available in the literature[18,19] regarding fracture toughness values of different restorative materials such as ceramic and amalgam are in range of 1.1 to 1.9 MPa $m^{1/2}$. On the other hand, Alert, EasyCore, Build-It and TI-Core had significant lower fracture toughness values and work of fracture energy than everX Posterior, which was an expected finding (Figs 2 and 4). In order for a fibre to act as an effective reinforcement for polymers, stress transfer from the polymer matrix to the fibres is essential[15]. This is achieved by having a fibre length equal to or greater than the critical fibre length and the given fibre aspect ratio in range of 30 to 94[11,15]. Aspect ratio, critical fibre length, fibre loading and fibre orientation are the main factors that could improve or impair the mechanical properties of fibre-reinforced composites[17]. Aspect ratio is the fibre length to fibre diameter ratio (l/d). It affects the tensile strength and the reinforcing efficiency of the fibre reinforced material[15].

Sufficient adhesion between fibre and matrix provides good load transfer between the two ingredients, which ensures that the load is transferred to the stronger fibre and this is how the fibre actually works as reinforcement. However, if the adhesion is not strong and if any voids appear between the fibre and the polymer matrix, these voids may act as initial fracture sites in the matrix and facilitate the breakdown of the material. everX Posterior had fibre length distribution between 0.3 and 1.5 mm, which is in the range of the reported critical fibre length and desired aspect ratio[11,17]. Therefore, it is unsurprising that short fibre fillers inclusion with semi-IPN resin matrix revealed improvements in fracture toughness.

Alert has a fibre length in micrometer scale (20 to 60 µm) and diameter of 7 µm (Fig 6a), which is well below the critical fibre length[14]. This explained the difference in fracture toughness values between the two commercial SFRC resins. Unfortunately, no literature on the short fibre fillers of other tested products exists for comparison. However, differences were seen by SEM analysis, which prove that materials with different microstructure characteristic (fillers {particulate/fibre} loading, length and diameter) could differ with regard to mechanical properties and wear. SEM pictures (Fig 6c) showed that dual-cured SFRC resin (Build-It) had relatively fine fillers and few micro fibres, and this might explain the lower reinforcing efficiency (Figs 2 to 4) and wear depth (Fig 5). The auto-cure SFRC resin (TI-Core) showed significantly higher wear (45 µm) than all other materials. This is explained partially by the large fillers, which might be abraded or broken out of the surface (Fig 6d). Moreover, the quantity of in-mixed porosities or air bubbles contributes to the polishability of this hand-mix packable material[20]. This is in accordance with Schmage et al, who demonstrated low values of surface hardness for TI-Core resin and showed that its titanium filler particles spread out easily[21]. On the other hand, light-cured SFRC resins (Alert and everX Posterior) with a normal packable consistence ranged in the same level of wear resistance, but differed widely in fracture toughness and work of fracture energy (Figs 2, 4 and 5). SFRC resin (EasyCore) showed beneficial wear-resistance and flexural strength (125.4 MPa), which was not significantly different from Alert (119 MPa) and everX Posterior (120 MPa). This is most likely due to fine fillers in combination with the dual-curing mode of the material (Fig 6b). It should be taken into account that it is instructed that SFRC resins (except for Alert) be used as bulk base or core foundation and should not be used as final fillings, but sometimes this procedure is unavoidable in clinical conditions[12,22]. They require an additional surface layer of conventional hybrid composite resin for giving the appearance of a natural tooth, and good wear resistance.

The difference in mechanical properties and wear values among the tested SFRC resins may be due to factors other than filler loading (particulate/fibre). Stress transfer from the polymer matrix to filler particles is one of the important factors to effect fracture toughness and wear. There may be a difference in the adhesion between fillers and matrix among these SFRC resins. Besides the filler system, monomer structures of the resin matrix also influence the mechanical properties and wear.

Methodologically, limitations such as sample size and the ageing process, such as alternate thermal stress, and water storage, should be taken into consideration. Despite the importance of laboratory studies to answer questions in a short time, the real performance of restorations can only be determined by long-term clinical trials.

Conclusion

Within the limits of this *in vitro* study, it can be concluded that commercial short fibre reinforced composites have different properties, which should be taken into account when optimum reinforcing results are to be achieved. everX Posterior has superior fracture toughness and EasyCore has good wear resistance.

Conflicts of interest

The authors reported no conflicts of interest related to this study.

Author contribution

Dr Sufyan Garoushi designed the study, prepared and evaluated the materials, and wrote the manuscripts; Dr Lippo Lassila designed the study and did the statistical work; Dr Pekka Vallittu contributed to the writing of the manuscript.

(Received Feb 22, 2017; accepted May 11, 2017)

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