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Dental Materials Journal 26(4): 589−597, 2007

Twenty-four Hour Flexural and Shear Bond Strengths of Flowable Light-cured Composites: A comparison Analysis Using Weibull Statistics

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Received January 25, 2007/Accepted February 26, 2007

By means of Weibull analysis, this study evaluated and compared the flexural strength and shear bond strength of flowable light-cured composites against those of conventional ones. Twenty specimens of each material were prepared for flexural and shear bond strength measurements. Specimens were measured after water storage at 37℃ for 24 hours. Three of four flowable composites showed significantly higher flexural strength than conventional ones, with Weibull moduli ranging between 6 and 14. With the presence of a bonding agent, the shear bond strength to enamel of both types was not different significantly (p=0.28), with Weibull moduli ranging between 4 and 9. In the selection of an excellent resin composite material, results of this study showed that a high, stable Weibull modulus value could be a sound indicator.

Keywords: Flowable composites, Mechanical strength, Weibull analysis

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INTRODUCTION

Presently, resin composites have replaced amalgam as the universal restorative material. Following the success of conventional composites (CCRs), demand — in tandem with continuous research development — led some manufacturers to launch flowable composites (FCRs). As such, the first generation of FCRs was introduced in late 1996, just before condensable composites[1)].

FCRs are materials of low viscosity. Hence, they flow easily into all the nooks and crannies, adapt readily to the tooth structure, and are able to fill irregular, internal surfaces. By virtue of these advantages, a prominent application of FCRs is inevitably as liners in areas of difficult access, such as the gingival margin of Class II preparations. The conventional wisdom is that the use of flowable composites would result in less microleakage, internal voids, and postoperative sensitivity[1,2)].

To date, FCRs have been explored and evaluated in numerous experiments — including as a base material in indirect Class II composite restorations[3)]. In terms of the occurrence rate of enamel microcracks in restored teeth, it was lower with FCRs than with conventional hybrid composites[4)]. As for Class IV restorations, FCRs were perceived as an inexpedient choice because they were acceptable only as filling materials in low-stress applications[1)]. With due consideration to the strengths and weaknesses of FCRs, most dentists would unreservedly use FCR as a lining material while completely reserved in using FCR as a restorative material. On the other hand, other dentists would use FCRs for a wide plethora of applications — as liners and pit and fissure sealants to repair marginal cracks and voids, as well as for Class I and V restorations[5)].

Fractures in the body and at the margins of restorations can lead to the failure of composite fillings. To prevent such fractures and failures, clinical studies would be the best and most effective means to characterize composite filling materials. However, clinical trials are more expensive and time-consuming than laboratory tests. Against this background, an *in vitro* determination of material parameters — such as flexural strength and shear bond strength — is a very important and helpful means of predicting clinical performance[6,7)].

Flexural strength measurement determines the real strength of a restorative material *per se*, while shear bond strength represents the strength of the material in correlation with tooth structure with the presence of a bonding agent. In both cases, Weibull statistical analysis is recommended because it allows for skewed data — predicting values within and outside the data set, thereby giving an “overall” analysis of the performance of a material[7-12)]. However, no reports are available that directly compare the mechanical properties of FCRs against their CCR counterparts of a similar brand using the Weibull statistical approach. In characterizing the performance of FCRs, it is very important that they be compared directly against CCRs of a similar or same brand, so as to have a more meaningful and comprehensive understanding of their mechanical characteristics.

Polymerization is in progress even after light curing[13)]. As such, this investigation was carried out after 24-hour water storage to evaluate the following properties: (a) flexural strength; (b) shear bond strength; and (c) Weibull modulus. The hypothesis was that the (a), (b), and (c) properties of FCRs were correlated to each other and would be significantly different compared with the CCRs.

MATERIALS AND METHODS

Four FCRs (Metafil Flo, Filtek Flow, Point 4 Flowable and Unifil Flow) and four CCRs (Metafil C, Filtek A110, Point 4, and Unifil F) of Shade A3, paired from the same manufacturers, were used in this study. Details of the materials used are listed in Table 1. Then, as listed in Table 2, bonding agents were chosen from the same manufacturers.

A visible light curing unit (New Light VL-II, GC; irradiated diameter: 10 mm) was used for activating the specimens, and close contact was ensured between exit window of the lamp and matrix. Before each application, light intensity was checked using a radiometer (Demetron, Kerr). During the experiment, light intensity was maintained at 450 mW/cm^2.

All procedures, except for mechanical testing, were performed in a thermohygrostatic room maintained at 23±0.5℃ and 50±2% relative humidity.

Flexural strength test

Teflon molds of 2.0 mm depth, 2.0 mm width, and 25.0 mm length were used to fabricate specimens for flexural strength measurement.

Twenty specimens were prepared for each material. Each mold was filled with the material, covered with a celluloid strip and a glass plate, then clamped. After 15 seconds, the glass plate was removed and the specimen light-cured in three sections with an overlap of 2 mm and an irradiation time of 20, 30, or 40 seconds according to manufacturer's recommendations. After light curing, the material was removed from the mold while excess filling material was removed with a silicon carbide bur. Then, the specimens were polished with a wet sandpaper (#600) to achieve a flat, even surface. After immersion in distilled water in a 37℃ incubator for 24 hours, the specimens were measured using a digital micrometer (No. 293– 421– 20, Mitutoyo) and tested.

Flexural strength was measured using the three-point bending method with a 20-mm span at a crosshead speed of 0.5 mm/min, by mounting the apparatus on a universal testing machine (Autograph DCS-2000, Shimadzu) as outlined in ISO 4049. A maximum external force of 10 kgf (98 N) was applied to the midpoint of test beam. Then, the stress at failure was calculated and recorded as the flexural strength of each materia[6,12,13].

Shear bond strength test to enamel

Random samples of human premolar teeth, extracted

Table 1 Materials investigated in this study. Information as provided by the manufacturers

Material	Manufacturer	Batch no.	Filler	Monomer	Curing time(s)
Metafil Flo* (microfilled)	Sun Medical, Moriyama, Japan	VV10, VV12, EK1, EK2	Barium silica glass, colloidal silica, and TMPT Filler content: 44 vol% (65 wt%) Filler particle size: 0.01– 10 μm	UDMA	40
Metafil C** (microfilled)	Sun Medical, Moriyama, Japan	TE1	TMPT and colloidal silica Filler content 54 vol% (66 wt%) Filler particle size: 0.01– 10 μm	UDMA	40
Filtek™ Flow* (microfilled)	3M, St. Paul, MN, USA	OBK, 20010104	Silica and silica zirconia Filler content: 47 vol% (68 wt%) Filler particle size: 1.50 μm	Bis-GMA, TEGDMA	20
Filtek A110** (microfilled)	3M, St. Paul, MN, USA	1AP	Inorganic silica Filler content: 40 vol% (56 wt%) Filler particle size: 0.04 μm	Bis-GMA	40
Point 4 Flowable* (microhybrid)	Kerr, Orange, CA, USA	206B43	Barium silica glass Filler content: 48 vol% (70 wt%)	TEGDMA, EBPADM	40
Point 4** (microhybrid)	Kerr, Orange, CA, USA	205553	Barium aluminoborosilicate glass Filler content: 57 vol% (76 wt%) Filler particle size: 0.4 μm	BISGMA, TEGDMA, EBPADM	40
Unifil Flow* (microhybrid)	GC Corp., Tokyo, Japan	0107201	Fluoroaluminosilicate glass, silica Filler content: 67 wt% (vol%: not Available) Filler particle size: 0.7 μm	UDMA (26%) Dimethacylate (7%)	40
Unifil F** (microhybrid)	GC Corp., Tokyo, Japan	161181, 0204031	Fluoroaluminosilicate glass, silica Filler content: 77 wt% (vol%: not Available) Filler particle size: 0.8– 0.9 μm	UDMA (16%) Dimethacylate (7%)	20

*: Flowable type (FCR); **: Conventional type (CCR); Bis-GMA: Bisphenol A diglycidylmethacrylate; EBPADM: Ethoxylated bisphenol A dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; TMPT: Trimethylol ropane trimethacrylate; UDMA: Urethane dimethacrylate

Table 2 Bonding agents used in this study. Information as provided by the manufacturers

Bonding product	Manufacturer	Batch no.	Restorative material	Components and procedure
AQ Bond	Sun Medical, Moriyama, Japan	FF1	Metafil Flo, Metafil C	One-bottle bonding agent: 4-META AQ sponge: bonding promoter AQ sponge + bond, coat 20 s, air-blow 3– 5 s, 2nd coat, air blow 5–10 s, light cure 10 s.
Single Bond	3M, St. Paul, MN, USA	20020603	Filtek Flow, Filtek A110	Etchant: 35% Hp_3PO_4 Primer and adhesive: H_2O, ethanol, HEMA, BisGMA, dimethacrylates, polyalkenoic acid copolymer, photoinitiator Etching 15 s, rinse 10 s, air-blow 1 s, adhesive 2 times, air-blow 2–5 s, light cure 10 s.
OptiBond Solo	Kerr, Orange, CA, USA	110B16	Point 4 Flowable, Point 4	Etchant: 37.5% H_3PO_4 Primer and adhesive: BHT, CQ, EtOH, HEMA, GPDM, fumed silica, NaSiF, barium aluminoborosilicate, BisGMA, ODMAB Etching 15 s, rinse 10 s, air blow 1 s, adhesive 15 s, air blow 3 s, light cure 20 s.
Unifil Bond	GC Corp., Tokyo, Japan	0206101	Unifil Flow, Unifil F	Monomer: carbonic acid, HEMA Self-etching primer 20 s, air blow 5 s, bonding agent, light cure 10 s.

BHT: 2.6-Di-tert-butyl-4-methylphenol (inhibitor); BisGMA: Bisphenol A diglycidylmethacrylate; CQ: Camphorquinone; EtOH: Ethanol; GPDM: Glycerol phosphate dimethacrylate;
H_3PO_4: Phosphoric acid; HEMA: Hydroxyethyl methacrylate; NaSiF: Sodium hexafluorosilicate; ODMAB: 2-ethylhexyl 4-dimethylamino benzoate

for orthodontic reasons, were used to measure the shear bond strength to enamel. After extraction and cleaning, the teeth were stored immediately in distilled water at about 4°C within three months before use. Flat, proximal enamel surfaces were used in this study. The teeth were embedded in a slow-setting epoxy resin (Epofix Resin, Struers) in cylindrical rubber molds (25 mm in diameter), with the proximal site for bonding facing the bottom of the mold. Embedded specimens were ground flat on a wet SiC abrasive paper up to 1000-grit, until an area of at least 4 mm in diameter was exposed in enamel.

A split Teflon mold with a cylindrical hole (diameter of 3.6 mm; height of 2.0 mm) was clamped to the prepared enamel surface. Etching and bonding agents were applied to the enamel surface according to manufacturers' instructions. The bonding materials used in this study are listed in Table 2. The Teflon mold was filled with the restorative material, covered with a celluloid strip and a glass plate, then clamped. After 15 seconds, the glass plate was removed. Then, the specimen was light-cured with an irradiation time of 20, 30, or 40 seconds according to manufacturer's recommendations before it was removed from the mold.

Of each material, 20 specimens were prepared. The prepared specimens were immersed in distilled water in a 37°C incubator. After 24 hours, they were measured using a digital micrometer and then secured in a mounting jig. Shear force was transmitted by a flat, blunt, 1-mm-broad shearing edge at a 90° angle to the direction of the load and at the back of the loading plate. Shear bond strength test was conducted using a universal testing machine, where shear force was applied at a crosshead speed of 0.5 mm/min with a maximum external force of 50 kgf (490 N). Stress at failure was calculated and recorded as the shear bond strength[14].

After shear bond strength measurement, fracture surfaces of the samples were ultrasonically washed in distilled water for two minutes and then lightly air-dried. To prepare for examination using a field emission scanning electron microscope (FE-SEM; DS-720, Topcon), the surfaces were coated with gold (Ion Coater IB-3, Eiko Engineering Co. Ltd.) at 7 mA for 240 seconds. Representative areas of test sites were photographed at ×1000 magnification.

Statistical analysis

Results of flexural and shear bond strength measurements were analyzed using Weibull statistics with the following equation:

$$Pf = 1 - \exp[-(\sigma/\sigma_0)^m]$$

where Pf is the probability of failure, σ is the strength at a given Pf, σ_0 is the characteristic strength, and m is the Weibull modulus — a constant factor related to the dispersion of failure strength data[8,9].

All measurements were also analyzed using one-way ANOVA and Tukey's test. Strengths between pairs were distinguished using t-test[15]. Possible correlations between parameters were analyzed using a SigmaPlot 8.0 program (SPSS, Chicago, Illinois, USA).

RESULTS

Flexural strength test

Table 3 lists the Weibull analysis of the 24-hour flexural strength values. All FCRs, except Point 4 Flowable, showed a higher 24-hour flexural strength value than their CCR counterparts. Further, all composite pairs showed significant differences between the mean values of FCR and CCR, except with the Unifil pair. The mean flexural strength value of Metafil Flo was 1.3-fold of Metafil C, that of Filtek™ Flow was 1.9-fold of Filtek A110, and that

Table 3 Weibull analysis of 24-hour flexural strengths

Material	Mean (SD)$^{\#}$ (MPa)		Characteristic strength (σ_0) (MPa)	Weibull modulus (m)	Correlation coefficient (r)	Stress for 10% chance of failure $\sigma_{0.10}$ (MPa)	Stress for 90% chance of failure $\sigma_{0.90}$ (MPa)
Metafil Flo*	128.1	(13.6) ^{b,c}	134.5	9.68	0.97	106.6	146.6
Metafil C**	98.0	(8.4) ^{e}	101.9	12.39	0.97	85.0	109.0
Filtek™ Flow*	133.1	(9.7) ^{a,b}	137.7	14.30	0.96	117.7	146.0
Filtek A110**	71.1	(6.5) ^{f}	74.2	11.20	0.97	60.7	80.0
Point 4 Flowable*	100.7	(14.6) ^{e}	107.1	7.31	0.97	78.7	120.1
Point 4**	141.4	(15.6) ^{a}	148.5	9.52	0.95	117.3	162.1
Unifil Flow*	118.4	(13.1) ^{c,d}	124.8	8.84	0.97	96.7	137.1
Unifil F**	109.1	(18.5) ^{d,e}	117.1	6.17	0.99	81.3	134.0

n=20; *: Flowable light-cured composite resin; **: Conventional light-cured composite resin; #: Identical letters indicate no significant differences according to Tukey's test ($p>0.05$).

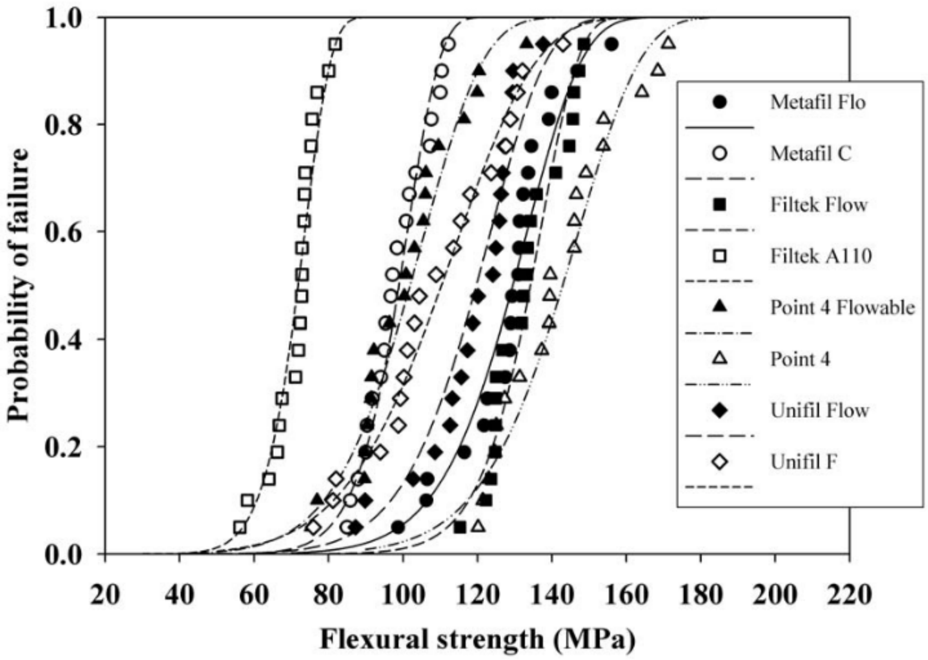

Fig. 1 Weibull distribution analysis of flexural strength data. Lines indicate the fitting curves of $Pf = 1 - \exp[-(\sigma/\sigma_0)^m]$.

Table 4 Weibull analysis of 24-hour shear bond strengths to enamel

Material	Mean (SD)# (MPa)		Characteristic strength (σ_0) (MPa)	Weibull modulus (m)	Correlation coefficient (r)	Stress for 10% chance of failure $\sigma_{0.10}$ (MPa)	Stress for 90% chance of failure $\sigma_{0.90}$ (MPa)
Metafil Flo*	15.5	(1.5) ^{c}	13.6	4.09	0.98	7.8	16.7
Metafil C**	15.8	(1.2) ^{c}	14.3	5.81	0.98	9.7	16.5
Filtek™ Flow*	24.1	(4.0) ^{a,b}	25.8	6.31	0.97	18.1	29.5
Filtek A110**	23.5	(4.3) ^{a,b}	25.3	5.77	0.99	17.1	29.2
Point 4 Flowable*	25.9	(3.9) ^{a}	27.6	7.05	0.99	20.1	31.1
Point 4**	27.7	(3.2) ^{a}	29.2	9.17	0.99	22.8	31.9
Unifil Flow*	16.7	(4.4) ^{c}	18.4	4.11	0.97	10.6	22.5
Unifil F**	18.2	(4.8) ^{b,c}	20.0	4.02	0.99	11.4	24.6

n=20; *: Flowable light-cured composite resin; **: Conventional light-cured composite resin; #: Identical letters indicate no significant differences according to Tukey's test (p>0.05).

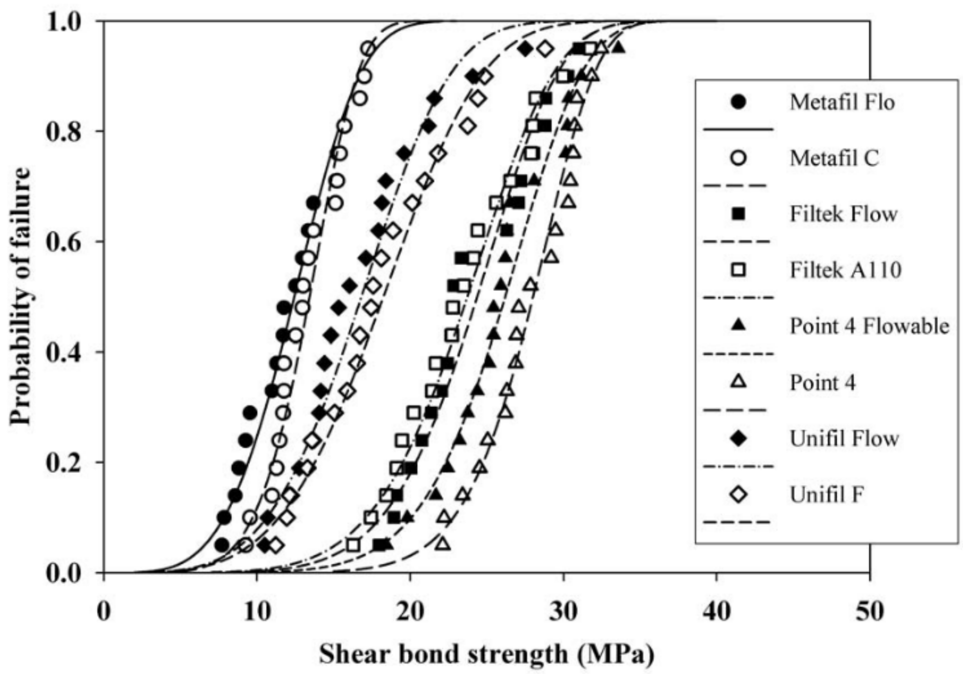

Fig. 2 Weibull distribution analysis of shear bond strength data. Lines indicate the fitting curves of $Pf = 1 - exp[-(\sigma/\sigma_0)^m]$.

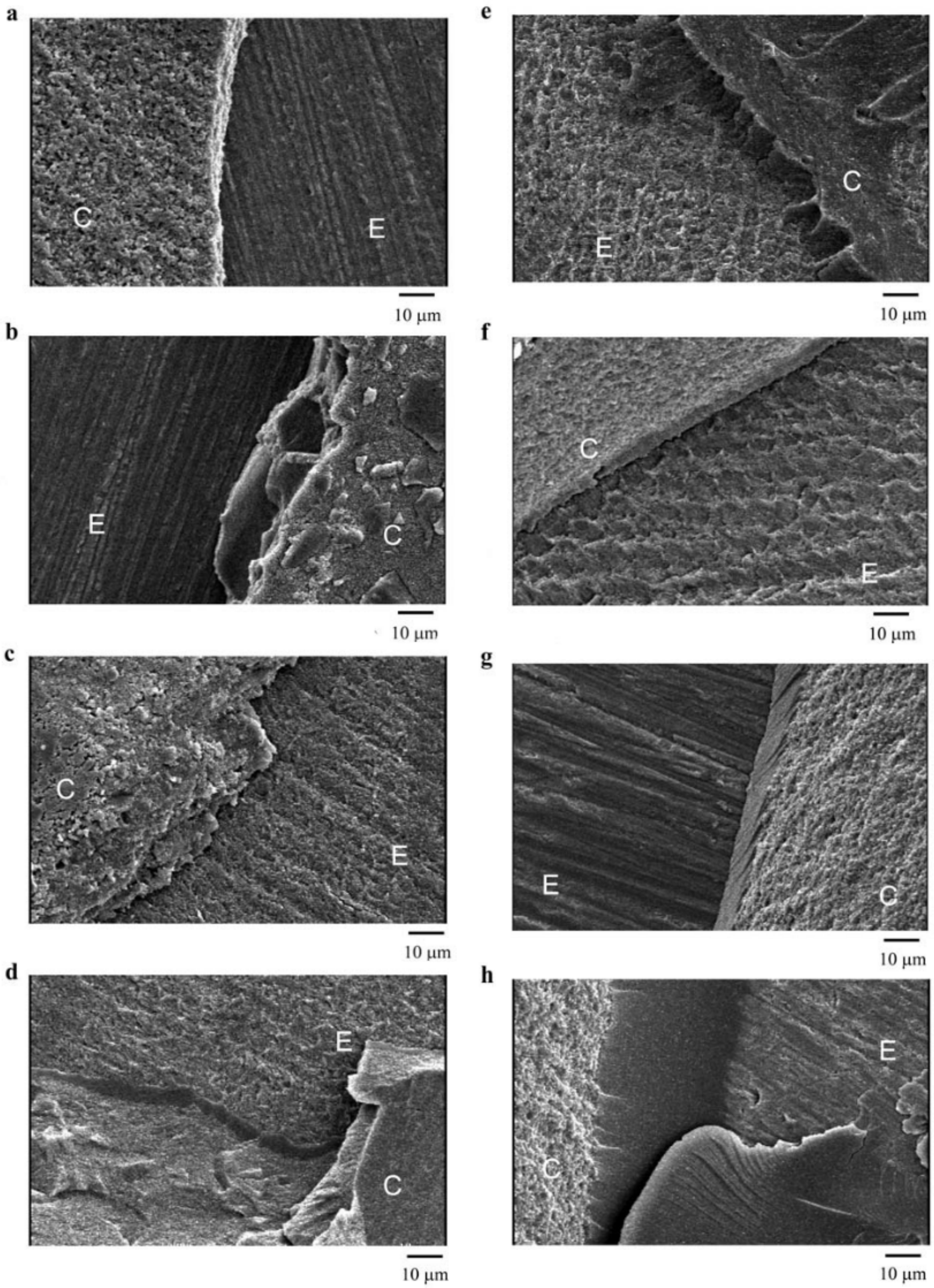

Fig. 3 Fracture modes of the specimens. C = Composite resin, E = Enamel. a: Metafil Flo, b: Metafil C, c: Filtek Flow, d: Filtek A110, e: Point 4 Flowable, f: Point 4, g: Unifil Flow, h: Unifil F.

of Unifil Flow was 1.1-fold of Unifil F. However, the mean flexural strength value of Point 4 Flowable was lower than that of Point 4 (0.7-fold).

Apart from the mean flexural strength values, Weibull analysis further showed that Point 4 had the highest characteristic strength (148.5 MPa), while Filtek A110 had the lowest characteristic strength (74.2 MPa). The Weibull moduli of materials were in the range of 6–14, and the correlation coefficients were in the range of 0.95–0.99. The Weibull curves for flexural strength are presented in Fig. 1.

Shear bond strength test to enamel

Table 4 lists the 24-hour shear bond strength values of the composites to enamel. The shear bond strengths of FCRs did not significantly differ from those of their CCR counterparts.

By means of Weibull analysis, Point 4 demonstrated the highest characteristic strength (29.2 MPa) while Metafil Flo showed the lowest (13.6 MPa). The Weibull moduli of materials were in the range of 4–9, and the correlation coefficients were in the range of 0.97–0.99. The Weibull curves for shear bond strength are presented in Fig. 2.

Figure 3 shows the SEM images of the fracture surfaces. All materials showed a mixed fracture mode, as indicated by the retention of composite resin on the enamel surfaces.

Statistical analysis

Application of t-test to each pair produced significant differences in flexural strength ($p<0.001$). However, for shear bond strength, each pair showed no significant differences (P4 pairs, $p=0.11$; Metafil pairs, $p=0.30$; Filtek pairs, $p=0.64$; and Unifil pairs, $p=0.33$). The analysis of possible correlations between flexural and shear bond strength values ($r^2=0.0003$) and between the Weibull moduli of flexural and shear bond strengths ($r^2=0.1801$) gave no significant correlation ($p>0.05$).

DISCUSSION

Results of flexural strength measurement of the present study agreed with those of previous studies — whereby Weibull moduli ranged between 6 and 9 for composite resins[16], and between 8.52 and 14.19 for FCRs[12]. As for the Weibull moduli of FCRs and CCRs in the present study, there seemed to be no clear significant difference pattern between them. In terms of flexural strength, there were significant differences between FCRs and CCRs. However, for shear bond strength, there were no significant differences between the pairs, as further indicated by the Weibull moduli.

In the fractured specimens of all composites, Fig. 3 showed that some composite resin was retained on all enamel surfaces. These SEM images clearly showed that the application of bonding agent on tooth substrate played an important role in influencing shear bond strength. At this juncture, it must be highlighted that the HEMA content — which enhances monomer diffusion in dentin substrates[17,18] — in the composition of Single Bond, Optibond Solo, and Unifil Bond did not show particular effect on shear bond strength to enamel. In contrast, a total etching procedure with phosphoric acid before applying Single Bond or Optibond Solo significantly resulted in an obvious pattern of enamel prisms on the fracture surfaces, which was not shown in the other two pairs. This etching procedure might also be a reason for the higher shear bond strengths of both Filtek and Point 4 pairs, despite the low flexural strength value of Filtek A110.

In agreement with another previous study[19], the shear bond strength values were lower than the flexural strength values. Compared to a previous study on tensile bond strength[7], the results of this study also showed a similar tendency, *i.e.*, disparity between bond strength and mechanical properties of composite resins. In that study[7], it was shown that the tensile strengths of restorative resins bonded to dentin, and the resultant dentin bond strengths between the two, exhibited wide variability. In particular, the tensile strengths of the restorative resins gave higher Weibull moduli. This great disparity in tensile strength and bond strength results, as revealed by Weibull probability of failure method, was attributed to the multiple variables at play during tensile and bond strength tests — such as specimen preparation and storage, test rig design, experimental technique, and even the brittle nature of the restorative materials.

In a study which used a shear punch test[10], the Weibull modulus range obtained was 4–10 — which was also similar to the range of 5–10 obtained for shear bond strength in this study. Nonetheless, on the overall, most of the values were lower than 6 — a result range attributed to the application of bonding agent on the tooth surface. It could thus be said that the shear bond strength results in this study were quite reliable, when compared with adhesive bonding data that typically produce Weibull modulus values in the range of 1–4 or 1–5[7,20]. As for flexural strength, most composite resins showed Weibull modulus values which were 1.5–2.4-folds that of shear bond strength. However, two materials showed similar Weibull moduli for flexural strength and shear bond strength: Point 4 Flowable with $m=7$ and Point 4 with $m=9$. It was thought that the EBPADM/TEGDMA content in Point 4 pair, which renders greater toughness and degree of conversion to the composites[21], has played an important role. In the same vein, therefore, factors that may influence the Weibull modulus values of shear bond strength — such as chemical bonding efficacy to tooth tissues and influence of bonding agents and composite resins — should be further investigated and evaluated in future experiments.

McCabe *et al.* stated that a Weibull modulus value of at least 10 is necessary for a test to be acceptable for multicenter testing[22]. In view of the Weibull modulus values presented here, it seemed that the shear bond strength test performed in this study did not meet this criterion. Therefore, to be valid for multicenter testing, it is of utmost importance that the test method is selected after careful consideration of conditions that may affect test results.

For the purpose of clinical use, the most straightforward indicator would be a Weibull modulus with the highest value[7,23]. In this study, however, we counter-proposed stable values — as those shown by Point 4 pair — to be used instead as a valid indicator for excellent composite restoratives. Furthermore, their favorable flexural strength and shear bond strength values augured well for clinical application. With reference to the data interpretation approach adopted in this study, we recommended that experimental data should be interpreted carefully and discerningly, paying particular attention to the trends and tendencies of Weibull distributions. This is because Weibull analysis not only can identify the characteristic strength for a spread of data, but also the stress level that a dental material should meet — if not exceed — in order to be accepted as an excellent material.

Many cases of clinical failure occur stemming from the 10%– 20% chance of failure[9,20]. Failures in such circumstances are unlikely to be predicted by mere consideration of the mean value of any mechanical property. However, with Weibull analysis, the strength of 10% probability of fracture ($\sigma_{0.10}$) was also determined without considering the mean value. In other words, Weibull statistics is a technique that can also be used to predict the probability of failure of dental restorative materials at any level of flexural stress application[9]. In addition, the values of $\sigma_{0.90}$ — the stress level at which one would expect a 90% chance of fracture — could also be analyzed to predict the upper limit of strength[12].

The Weibull cumulative plots (Figs. 1 and 2) show the failure prediction for all FCRs and CCRs. Compared to their CCR counterparts, almost all FCRs showed higher 24-hour flexural strength values. The weakest link in composites appeared to be the interface between the filler particles and resin matrix. As the filler contents of FCRs were lower than those of CCRs, it thus seemed reasonable that cracks occurred more readily in CCRs than in FCRs. Moreover, besides monomer type, filler content, filler particle size, interparticle spacing, and coupling agent, water influences the strength of restorative materials by affecting the filler-matrix interface[19,23,24]. Nonetheless, with FCRs, water sorption — which is accompanied by hygroscopic expansion — will help to compensate the effects of polymerization shrinkage.

CONCLUSIONS

Flowable composites were found to have higher 24-hour flexural strengths than the conventional ones. However, the shear bond strength to tooth enamel of flowable and conventional composites were similar, due to the application of a bonding agent. In the selection of an excellent resin composite material, results of this study showed that a high, stable Weibull modulus value could be a sound indicator.

ACKNOWLEDGEMENTS

The authors wish to thank the manufacturers for their generous donation of materials for this research project.

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