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Investigations on mechanical behaviour of dental composites

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Abstract Since a direct comparison of composites efficacy in clinical studies is very difficult, our study aimed to analyse in laboratory tests under standardised and simulated clinical conditions a large variety of commercial composite materials belonging to eight different materials categories. Thus, 72 hybrid, nano-hybrid, micro-filled, packable, ormocer-based and flowable composites, compomers and flowable compomers were compared in terms of their mechanical behaviour. Flexural strength (FS), flexural modulus (FM), diametric tensile (DTS) and compressive strength (CS) were measured after the samples had been stored in water for 24 h at 37°C. Results were statistically analysed using one-way ANOVA with Tukey HSD post hoc test (α =0.05) as well as partial η^2 statistics. Large varieties between the tested materials within the same material category were found. The hybrid, nano-hybrid, packable and ormocer-based composites do not differ significantly among each other as a material type, reaching the highest FS values. Nano-hybrid composites are characterised by a good FS, the best DTS but a low FM. The lowest mechanical properties achieved the micro-filled hybrids. The flowable composites and compomers showed for all properties comparable result. Both flowable material categories do not differ significantly from the micro-filled composites for the most mechanical properties, showing only a higher DTS. The filler volume was shown to have the highest influence on the measured properties, inducing a maximum FS and FM at a level of 60%, whereas such dependence was not measured for DTS or CS. The

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Department of Restorative Dentistry, Dental School, Ludwig-Maximilians-University, Goethestr. 70, 80336 Munich, Germany e-mail: nicoleta.ilie@dent.med.uni-muenchen.de influence of the type of material on the mechanical properties was significant but very low, showing the strongest influence on the CS.

Keywords Composites · Mechanical properties · Strength · Modulus of elasticity

Introduction

During the last three decades, innovative improvements of direct restorative composite materials were made, leading nowadays to an excellent acceptance of methacrylate-based direct restorative materials. Versatile methods to modify the monomer matrix have been developed, starting with typical dimethacrylate monomers being replaced by methacrylates with reduced reactive groups (for example hydroxyl-free bis-GMA) or the development of the urethandimethacrylat. Other approaches proposed for reducing polymerisation shrinkage include the development of liquid crystal monomers or ring-opening systems so as to develop minimally or non-shrinking dental composites that contain spiroorthocarbonates as additives to dimethacrylate or epoxy-based resins [1-4]. However, only a few of the new monomers could be launched in commercial composite materials and the majority of conventional composite-type materials in use today continue to be based on the dimethacrylate resins introduced in the 1960s and 1970s. The most significant changes in commercial composites in the last decades have been made by far through improvements in the filler system [2]. Filler not only directly determine the mechanical properties of composite materials but also allow reducing the monomer content and consequently the polymerisation shrinkage, optimising wear, translucency, opalescence, radiopacity, intrinsic surface roughness and, thus, polishability as well as to enhance aesthetics and improve handling properties. The size of filler particles incorporated in the resin matrix of commercial composites has continuously decreased over the years from the traditional to the nano-hybrid materials. The modification of filler size and morphology resulted in improved mechanical properties and aesthetics compared with earlier composite materials [2].

These variations in filler size, morphology, amount, volume, distribution or chemical composition created a large variety of composite categories, confusing the practitioners nowadays in the choice of the proper restorative material. In an attempt to have a clear clinical indication for the existing commercial composites, classification criteria were developed, most of them influenced by the filler system [5–8]. These criteria are primarily based on the amount of inorganic filler fraction in volume percent or on the mean particle size. But also, the Young's modulus or the intrinsic surface roughness were taken into consideration as valuable classification parameters [8], in view of the crucial role of the modulus of elasticity in the deformability of a material under masticatory stresses, particularly in posterior regions, or considering the fact that for aesthetical demands a low intrinsic surface roughness is necessary. Despite all this efforts, due to the complexity of the materials, a clear and general valid classification of composites, especially in regard of their clinical success, could not be found.

The variety and amount of commercial composite materials for tooth defects restoration available today makes a direct comparison of their efficiency in clinical studies impossible. Thus, for comparative investigations of material behaviour, it is valuable to evaluate data measured under identical test conditions. Our study aims, therefore, to compare 72 frequently used composites belonging to several composite categories—hybrid, nano-hybrid, micro-filled, packable, ormocer-based and flowable composite as well as compomers and flowable composers—in terms of their mechanical behaviour. For this reason, flexural strength and modulus of elasticity, compressive and diametric tensile strength were evaluated.

The following null hypotheses were tested: there are no differences in the mechanical properties between the eight material categories, and the behaviour of the tested materials is similar in the three different loading conditions.

Materials and method

The flexural strength (FS) and flexural modulus (FM) were determined in a three-point bending test (n=8) in analogy to ISO/DIN 4049:1998. The samples were made by compressing the composite material between two glass plates with intermediate Polyacetate sheets, separated by a steel mould having an internal dimension of $2 \times 2 \times 16$ mm. The material was cured for 40 s from both sides in a lightcuring oven (Dentacolor XS, Kulzer, Wehrheim), which assures that the whole surface is cured at the same time. After curing, the specimens were removed from the mould and any flash material was trimmed away with sandpaper (grit size P4000 (FEPA)). All specimens were then stored in distilled water at 37°C prior to testing for 24 h. The samples were loaded until failure in the universal testing machine (MCE 2000ST, quick test Prüfpartner GmbH, Langenfeld, Germany) in a three-point bending test device, which is constructed according to the guidelines of NIST No. 4877 with 12-mm distance between the supports. During testing, the specimens were immersed in distilled water at room temperature. The crosshead speed was 0.5 mm/min. The universal testing machine measured the force during bending as function of deflection of the beam. The bending modulus was calculated from the slope of the linear part of the force-deflection diagram.

The compressive strength (CS) was determined on cylindrical specimens (8 mm height and 4 mm in diameter) made in a Teflon mould by curing and storing the samples similar above (n=8). The samples were placed with the flat end on the supporting plate of the universal testing machine and a compressive load was applied axially at a crosshead speed of 0.5 mm/min.

The diametric tensile strength (DTS) was determined on cylindrical specimen (3 mm height and 6 mm in diameter) prepared similar above. The samples (n=8) were placed with the outside surface of the cylinder contacting the supporting plates of the universal testing machine, and a compressive load was applied at a crosshead speed of 0.5 mm/min.

Results were statistically compared within each material category as well as among the material category using oneway ANOVA and Tukey HSD post hoc test (α =0.05). A multivariate analysis (general linear model) tested the influence of the parameters *filler volume* and *weight* as well as *material category* on the measured properties (SPSS 16.0).

Results

The results of the investigated properties for the examined materials are summarized in Tables 2, 3 and Fig. 1.

Large varieties between the tested materials within a material category were found. Post hoc multiple pairwise

Table 1 Materials, manufacture:	r, chemical composition of the m	atrix and filler and filler content by weight and volume	e %	
Composites	Manufacturer/batch	Resin matrix	Filler	Content (w/v)
Hybrid composites				
Adamant Cavifil	Vivadent/E 52179	BisGMA, UDMA, TEGMA	Ba glass, YbF ₃ , SiO ₂ , MO	81/62
Arabesk Top	VOCO/94816	BisGMA, UDMA, TEGDMA	Glass ceramic	77/56
Ariston pHc	Vivadent/B21705	BisGMA, UDMA, TEGDMA	Alkaline glass, Ba-Al-F-Si glass, YbF ₃ , SiO ₂	79/59
Beautifil	Shofu/050143	BisGMA, TEGDMA	F-B-Al-Si glass	83.3/68.6
Brillant	Coltene/IA350	BisGMA, BisEMA, TEGDMA	B glass, SiO ₂	78/59
CapoCom.4	GDF/P 9.42	BisGMA, UDMA, TEGDMA	Glass, pyrog. SiO ₂	IN/08
Charisma	Heraeus Kulzer/10023	BisGMA, TEGDMA	Ba–Al–F glass, SiO ₂	78/61
Clearfil ST	Kuraray/00002A	BisGMA, TEGDMA	Ba glass, SiO ₂	86/70
Ecusit [®] Composite	DMG/99260036	BisGMA; UDDMA, TEDMA	Ba glass, pyrogen, SiO ₂	77/57
EcuSphere Carat	DMG/539192	BisGMA, UDMA, TEGDMA	Glass, SiO ₂	77/57
ELS	Saremco/07	BisGMA, BisEMA, IBMA	Ba glass, Ba–Al–B–Si glass	74/>50
Enamel plus HFO	GDF/2000002388	BisGMA, UDMA, TEGMA	Glass filler, SiO ₂	75/53
Esthet-X	Dentsply/0112121	BisGMA, BisEMA, TEGDMA	Ba-F-Al-B-Si glass, SiO ₂	77/60
Herculite XRV	Kerr/909065	BisGMA, TEGDMA	Al-B-Si glass, SiO ₂	79/59
Hermes/Silorane	3M ESPE/A-094	 4-Epoxycyclohexylethylcyclo-polymethylsiloxane Bis-3.4-epoxycyclohexylethyl-phenylmethylsilane 	Silanized quartz, YF	76/55
InTenS	Vivadent/RZB 033	BisGMA, UDMA, BisEMA	Ba glass,YbF ₃ , copolymers	74/51
Miris	Coltene/KI272	Methacrylate	Ba glass, SiO ₂ glass	80/65
Pertac II	3M-ESPE/025	Bifunctional methacrylates	Quartz, SiO ₂ , YF ₃	80/61
Point 4	Kerr/102A48	BisGMA, TEGDMA	Ba-Al-B-Si glass, SiO ₂	76/57.2
Prodigy	Kerr/812898	BisGMA, TEGDMA, EBADM, UV-9	Ba-Al-Zn oxide, SiO ₂	79/59
Superlux Universalhybrid	DMG/991600018	BisGMA; UDMA, TEGDMA, HDDMA	Ba glass, pyrogen SiO ₂ , Al silicate, polymer powder	83/75
Synergy Duo Shade	Coltene/IG079	BisGMA, BisEMA, TEGDMA	Sr glass, Ba glass, SiO ₂	74/59
Tetric	Vivadent/C16884	BisGMA; UDMA, TEGDMA	Ba glass, YbF ₃ , SiO ₂ , MO	81/62
Tetric Ceram	Ivoclar-Vivadent D00037	BisGMA, UDMA, TEGDMA	Ba glass, Ba–Al–F–Si glass, YbF ₃ , SiO ₂ , MO	78.6/60
TPH Spectrum TM	Dentsply/9907000556	BisGMA, BisEMA, TEGDMA	Ba-Al-B-Si glass, SiO ₂	77/57
Venus	Heraeus Kulzer/010022	BisGMA, TEGDMA	SiO ₂ , Ba–Al–B–F–Si glass	79/61
Z100	3M ESPE/2EG	BisGMA, TEGMA	ZrO_2/SiO_2	84.5/64.2
Z250	3M ESPE/20011016	BisGMA; UDMA, BisEMA	ZrO_2/SiO_2	84.5/60
Nano-hybrid composites				
CeramX Duo	Dentsply/0502002164	MS, DM	Ba–Al-B–Si-glas, SiO ₂	76/57
CeramX Mono	Dentsply/0502002159	MS, DM	Ba–Al-B–Si-glass, SiO ₂	76/57
Filtek Supreme	3M ESPE RBX/2003	BisGMA, BisEMA, UDMA, TEGDMA	ZrO ₂ /SiO ₂ cluster SiO ₂ nano-filler	78.5/59.5
Filtek Supreme Enamel	3M ESPE/5EM	BisGMA, BisEMA, UDMA, TEGDMA	ZrO ₂ /SiO ₂ cluster SiO ₂ nano-filler	78.5/59.5
Filtek Supreme XT	3M ESPE 20060527	BisGMA, BisEMA, UDMA, TEGDMA	ZrO ₂ /SiO ₂ cluster SiO ₂ nano-filler	78.5/59.5

Table 1 (continued)				
Composites	Manufacturer/batch	Resin matrix	Filler	Content (w/v)
Premise Enamel	Kerr/014533	BisGMA, UDMA, TEGDMA	PPF, Ba glass, SiO ₂	84/69
Tetric Evo Ceram	Ivoclar-Vivadent JO9286	BisGMA, UDMA, DMDMA	Ba glass, YbF ₃ , MO, PPF	76/55
Micro-filled composite				
Durafill	Heraeus Kulzer/121	BisGMA, TEGDMA, UDMA	SiO ₂ , Splitter polymerisate	60/40
EcuSphere Shine	DMG/544101	BisGMA-based resins	Pyrogene silica	60/46
Heliomolar radiopaque	Ivoclar-Vivadent D03569	BisGMA, UDMA, DDMA	YbF ₃ , SiO ₂ 77,8 wt-gesamt%	66.7/46
Silux Plus TM	3M 9EY/19990630	BisGMA, TEGDMA	SiO_2 , Prepolym.	56/40
Packable composites				
Alert	Jeneric/Pentron 840971	PCDMA, UDMA	Ba-B-Al-Si-glas, SiO ₂ , Glasfiber	81/67
Filtek P60	3M ESPE	BisGMA, BisEMA, UDMA	ZrO_2/SiO_2	83/61
Prodigy Condensable	Kerr/812898	BisGMA, TEGDMA	SiO ₂ , Ba-Al-B-Si-glass	80/61.7
Quix Fil	Dentsply 0509000740	UDMA, TEGDMA, Di- and	Sr-Al-Na-F-P-Si-Glass	86/ 66
		trimethacrylate resins, Carboxylic acid modified dimethacrylate resin		
Solitaire 1	Heraeus Kulzer/42	Multi-acrylate monomers	Porous SiO ₂ glass Ba-Al-F-Si glass	65/NI
Solitaire 2	Heraeus Kulzer 020224	UDMA, BisGMA, TEGDMA, TFM	Porous SiO ₂ , Ba-Al-B-F-Si glass	75/58
Surefil	Dentsply/020307	UDMA	Ba-F-Al-B-Si-glas, SiO ₂ -glass	82/66
Synergy Compact	Coltene/IH405	BisGMA, BisEMA, UDMA	Sr glass, Ba glass, SiO ₂	74/59
Tetric Ceram Condensable	Ivoclar-Vivadent RZ-A025	BisGMA, UDMA, DDM	Ba glass, Ba-Al-F-Si-glass, YbF ₃ , SiO ₂ , MO	81/63
Ormocer-based composites				
Definite	Degussa/1985201	Ormocere [®] , Polysiloxan, BisEMA, DDM	Ba glass, Aerosile, modified Apatit	77/61
Admira	V0C0/94512	Ormocere [®] , BisGMA, UDMA, TEGDMA, BHT	SiO ₂ , Ba–Al-B–Si-glass	77.0/60.2
Compomeres				
Compoglass F	Ivoclar-Vivadent 900507	UDMA, TEGDMA Cycloaliphatic dicarboxylic acid dimethacrylate	YbF3, Ba–Al-F–Si-glass, MO	77/55
Dyract	Dentsply 9909000736	UDMA, TCB	Sr-Al-Na-F-P-Si-glass; SrF ₂	72/49
Dyract AP	Dentsply 9909000819	UDMA, TCB, APM	Sr-Al-Na-F-P-Si-glass; SrF ₂	75/53
Elan	Kerr/901201	polycarboxylic acid, UDMA, TEGDMA	Ba-Al-B-silicate, Na-F-silicate, SiO ₂	Ι
F2000 Rasant	3m 9BR / 19990817	polycarboxylic acid, GDMA, polyvinylpyrrolidone;	F-Al-Silicate glass	84/67
Glasiosite	VOCO 92538	BisGMA, diUDMA, TEGDMA, BHT	glass ceramics, silicates	77.5/NI
Hytac	ESPE FW0050451	Co-monomers	YF ₃ , SiO ₂ , (Ca, Zn)FAISi -Glass	-/99
Luxat	DMG 99270049	Monomers	Anorganic filler	I
Flowable composites				
Admira Flow	VOCO/99791	Ormocere [®] , Methacrylate, BHT	Inorganic filler	64/50.5
Arabesk Flow	Voco/91658	BisGMA, UDMA, TEGDMA	SiO ₂ ,Ba-Sr-B-silicate	64.0/50.3
Definite Flow	Degussa 3004400	Ormocere [®] , Polysiloxan, BisEMA , DDM	Ba glass, Aerosile, modified Apatit	63/-
FLOWline	Heraeus Kulzer 80021	Methacrylates, TEGDMA	Iorganic filler	62/41

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Grandio Flow	VOCO/661487	BISGMA, TEGDMA, HEDMA	F–Si-glass, SiO ₂ ,	80.2/65.7
Palfique Estelite High Flow	Tokuyama/106	BisGMA, TEGDMA	SiO ₂ -ZrO ₂ , SiO ₂ -TiO ₂	68/50
Palfique Estelite Low Flow	Tokuyama/506	BisGMA, TEGDMA	SiO ₂ -ZrO ₂ , SiO ₂ -TiO ₂	65/50
Reference Flow	ESPE 643902001	BisGMA	Inorganic filler	Ι
Revolution	Kerr 9-1294	BisGMA	Inorganic filler	Ι
Tetric Flow	J12549	BisGMA, UDMA, TEGDMA	Ba glass, YbF ₃ , Ba–Al-F–Si-glass, SiO ₂ , MO	68.1/43.8
Flowable compomers				
Compoglass Flow	Ivoclar-Vivadent A03341	UDMA, TEGDMA Cycloaliphatic dicarboxylic acid dimethacrylate	YbF ₃ , Ba–Al-F–Si glass, MO	66.8/41.8
Dyract Flow	Dentsply 10800023	Ammonium salt of PENTA and N,N-dimethyl aminoethyl methacrylate, Carboxylic acid modified methacrylates. DGDMA	Sr-Al-Na-F-P-Si-glass, SiO ₂	59/43
Estelite Flow Quick	Tokuyama/EE60476	BisGMA, TEGDMA	SiO ₂ -ZrO ₂ , SiO ₂ -TiO ₂	71/53
PrimaFlow	DMG/99260209	UDMA, acidic methacrylates	IonomerGlas, SiO ₂	64/40
Data are provided by manufactur BieGMA hisnband A dictoridy	ers; ''–'' no information availabl Lether dimetheorylate BisFM	e A histhend A nolvethevlene olvool diether dimethao	ervlate TEGDM4 triethvlene alvord dimethaervlate 11	IDM4 urethane

IBMA isobornylmethacrylate, PENTA TCB resin tetracarboxylic acid-hydroxyethylmethacrylate-ester, HPMA 3-hydroxpropyl methacrylate, ETMAL ethyltriglycol methacrylate, APM dimethacrylate resin, MS methacrylate-modified polysiloxane, DDMA decandioldimethacrylate, TFM tetrafunctionalmonomers, DDM decandiol dimethacrylate, glycol dimethacrylate, diethylene dimethacrylate, DMDMA decamethylendimethacrylate, DGDMA diethylene glycol dimethacrylate, DGDMA ς Ω penta acrylate monophosphate, filler prepolymerised alkanoyl-poly-methacrylate, DM oxide, PPF dipentaerythritol MO mixed

comparisons with Tukey's HSD test (p < 0.05; Table 2) showed that within the tested material categories, the hybrid, nano-hybrid, packable and ormocer-based composite do not differ significantly among each other as a material group, reaching the highest flexural strength values. The lowest mechanical properties—including FS, FM and DTS—achieved the micro-filled hybrids. Considering the flowables, the tested flowables composites and compomers showed for all properties comparable results. Both material categories do not differ significantly from the micro-filled composites in the most mechanical properties, showing only a higher DTS.

Nano-hybrid composites are characterised by a good FS, the best DTS but a lower modulus of elasticity.

The influence of the parameters "Filler volume", "Filler weight" and "Material category" as well as their interaction products were analysed in an ANOVA multivariate test. The mechanical properties-flexural strength, modulus of elasticity, diametric tensile strength and compressive strength-were selected as depended variables. The significance values of these three main effects, as well as of the interaction product "Filler volume x Filler weight" were less than 0.05, indicating that they contribute all to the model. The results of the ANOVA multivariate test are summarised in Table 3, showing that the strongest influence on the mechanical properties (higher η^2 values) was performed by the Filler volume, followed by Filler weight, whereas the influence of the material category was low but still significant. Furthermore, the parameter which was more sensitive influenced by the filler was the Flexural modulus, followed by the FS, CS and DTS. The influence of the material category was very low, showing the strongest influence on the CS.

An excellent linear correlation was found between filler volume and filler weight (0.93), a moderate one between FS and FM (0.5), whereas the correlation between FS–DTS (.24) and FS–CS (.15) was very low.

Figure 1 presents the variation of the FS and FM as function of filler volume and weight for all materials, without taking in consideration their belonging to a material group. An Epanechnikov kernel function [9] was used for contouring the areal density of the measured data, showing a maximum FS and FM for a filler volume of ca. 60%.

Discussion

In a review published 2005, Sarrett asserted that clinical data indicate secondary caries and restoration fracture as the most common clinical problems in posterior composite restorations [10]. Brunthaler et al. concluded in a survey of prospective studies on the clinical performance of posterior resin composites published between 1996 and 2002 that the

Table 2 Flexural strength (FS) and modulus (FM), diametral tensile strength (DTS) and compressive strength (CS)

Composites	FS [MPa]	FM [GPa]	DTS [MPa]	CS [MPa]	
Hybrid composites					
Z250	160.8^{i} (22.2)	$10.3^{\rm lm}$ (0.6)	33.1 ^{abcde} (3.6)	282.9 ^k (25.6)	
Synergy Duo Shade	143.5 ^{hi} (16.3)	8.7 ^{hijk1} (0.6)	28.2^{abc} (3.7)	272.5 ^{jk} (27.8)	
Ecusit Composite	140.7 ^{ghi} (15.5)	9.6^{ijkl} (0.5)	30.3^{abc} (5.1)	263.5 ^{hijk} (19.5)	
Tetric	139.9 ^{ghi} (15.0)	9.9 ^{jklm} (0.7)	30.6^{abcd} (3.9)	261.2 ^{hijk} (27.8)	
TPH Spectrum	138.6 ^{ghi} (15.6)	8.8^{hijkl} (0.3)	42.1^{def} (6.9)	261.2 ^{hijk} (28.0)	
Enamel plus HFO	138.4 ^{ghi} (10.2)	8.5^{ghijk} (1.7)	33.2^{abcde} (4.0)	199.1 ^{efghi} (44.9)	
Adamant Cavifil	135.9 ^{fghi} (18.0)	7.7^{efgh} (1.2)	47.7 ^f (15.9)	206.1 ^{efghij} (58.7)	
Z100	134.5 ^{fghi} (18.0)	$11.3^{\rm m}$ (0.5)	33.5^{abcde} (4.8)	256.6 ^{hijk} (36.2)	
Prodigy	126.6^{efgh} (20.9)	7.8^{fgh} (0.3)	38.5 ^{bcdef} (5.5)	253.6 ^{hijk} (26.9)	
Arabesk Top	123.1^{defgh} (23.3)	$8.1^{\text{fghi}}(0.6)$	26.9^{ab} (4.7)	248.8^{ghijk} (48.4)	
Herculite XRV	121.8^{cdefgh} (12.4)	$8.5^{\text{ghij}}(0.6)$	31.8^{abcd} (2.3)	$251 4^{\text{hijk}} (33.0)$	
Ariston pHc	$119 3^{\text{bcdefgh}} (11 1)$	$7 3^{\text{efgh}} (0.9)$	$44 2^{\text{ef}} (5.6)$	$179 3^{cdefg} (62.0)$	
Brillant	119.5 (11.1) $110.2^{bcdefgh}$ (13.0)	$7.5^{\text{efgh}}(0.6)$	$31 0^{abcd} (3.7)$	2180^{fghijk} (33.0)	
Difficient Pertac II	119.2 (13.9) 117.5^{bcdefgh} (12.6)	7.7 (0.0) 8 7 ^{hijkl} (0.3)	30.4^{abcd} (3.3)	$242 2^{\text{ghijk}} (38.0)$	
Tetria Caram	117.5 (12.0)	8.7 (0.3) $8.1^{\text{fghi}} (0.4)$	30.4^{cdef} (3.3)	242.2 (38.0) 248.0^{ghijk} (24.8)	
Superlux Universally	$113 0^{\text{bcdefg}} (0.2)$	$6.0^{efg} (0.7)$	37.2 (3.7) 32.0^{abcd} (7.8)	270.0 (24.0) 267 7^{ijk} (53 Λ)	
Superiux Universalityofid	113.9 = (9.2)	0.9 = (0.7)	32.0 (7.8)	207.7° (33.4)	
Ecusphere Carat	$112.8 \stackrel{(17.0)}{=} (17.0)$	0.0 (0.8)	34.7 (4.4)	$232.5 \circ (27.0)$	
venus	108.6 (14.7)	$2.9^{\circ}(0.6)$	31.0^{abcd} (5.0)	150.8 (35.0)	
Hermes	108.5 (24.0)	7.5 ^{rg} (1.4)	$31.0^{-10}(7.2)$	129.1^{ik} (43.3)	
Esthet-X	106.8^{bode} (13.6)	7.8 ^{-get} (0.6)	29.3^{ab} (3.7)	$2/2.4^{12}$ (13.2)	
Point 4	104.2^{bcde} (14.0)	5.7^{eu} (1.5)	27.0^{ab} (11.5)	150.2 ^{codd} (35.2)	
Reference	102./abcde(8.5)	10.1^{mm} (1.8)	37.5 ^{coder} (6.7)	284.4 ^x (35.0)	
Charisma	102.4 ^{abcde} (11.1)	7.1 ^{erg} (0.5)	27.3^{ab} (3.0)	263.3 ^{mg} (15.3)	
Beautifil	98.4 ^{abcde} (16.6)	5.2 ^{cd} (1.0)	29.0 ^{abc} (7.9)	121.6 ^{auc} (27.1)	
Clearfil ST	97.5 ^{abcde} (16.8)	6.1^{cde} (1.0)	27.6^{abc} (5.0)	150.8^{bcder} (45.1)	
Miris	94.9 ^{abcd} (16.6)	$4.7^{\rm bc}$ (1.2)	22.4 ^a (7.9)	70.8^{a} (23.5)	
InTenS	93.1^{abc} (9.8)	4.8^{bc} (0.5)	35.9^{bcder} (6.3)	195.3 ^{dergn} (37.6)	
CapoCom.4	91.8 ^{ab} (19.0)	$3.4^{ab}(0.8)$	30.3^{abc} (4.9)	100.4 ^{ab} (17.5)	
ELS Extra low shrinkage	73.6 ^a (19.0)	$2.4^{a}(0.8)$	26.9^{ab} (6.5)	138.3^{abcde} (56.6)	
Nano-hybrid composites					
Filtek Supreme Enamel	125.5 ^d (13.1)	5.4 ^{bc} (0.6)	39.2 ^{abc} (8.3)	267.4 ^b (38.3)	
Filtek Supreme Dentin	122.1 ^{cd} (14.4)	5.2 ^{ab} (0.5)	41.8 ^{abc} (7.8)	103.7 ^a (20.9)	
Filtek Supreme XT	108.6 ^{bcd} (19.0)	6.1 ^c (1.1)	35.8 ^{ab} (7.6)	134.3 ^a (46.7)	
CeramX Duo	100.9 ^{abc} (17.9)	4.5^{ab} (0.5)	32.0^{a} (4.9)	214.9 ^b (25.6)	
Tetric Evo Ceram	96.0 ^{ab} (14.3)	5.3^{bc} (0.5)	38.5^{abc} (5.2)	219.7 ^b (46.9)	
CeramX Mono	95.0 ^{ab} (13.3)	$4.3^{\rm a}$ (0.4)	39.6^{abc} (7.9)	240.7 ^b (25.8)	
Premise Enamel	82.2 ^a (8.1)	5.0 ^{ab} (0.5)	45.1 ^{bc} (4.5)	242.8 ^b (36.1)	
Micro-filled composites					
Heliomolar radiopaque	87.6° (3.0)	4.1 ^b (0.2)	25.1 ^{ab} (2.4)	231.3 ^a (22.9)	
Durafill	76.4 ^b (9.2)	3.0^{a} (0.2)	21.5^{a} (2.4)	289.8 ^b (33.1)	
EcuSphere Shine	68.4^{a} (6.1)	$3.3^{a}(0.3)$	27.2^{b} (4.5)	219.2 ^a (22.2)	
Silux Plus	$62 8^{a} (3.4)$	$5.0^{\circ}(0.3)$	$27.2^{\circ}(1.5)$ $22.4^{\circ}(2.1)$	$2.54 3^{ab} (30.4)$	
Shint 1 100	02.0 (0.1)	5.5 (0.5)	22.1 (2.1)	201.0 (00.1)	
Packable composites	k				
Filtek P60	136.2 ^a (9.9)	$10.2^{\circ} (0.5)$	37.3 ^{ca} (6.4)	273.7° (43.8)	
Alert	124.7 ^{ca} (22.1)	12.5^{a} (2.1)	46.8 ^e (3.1)	275.6° (22.9)	
Synergy Compact	124.1 ^{cd} (11.8)	8.2^{bc} (0.6)	22.9 ^a (4.5)	195.4 ^{ab} (34.8)	

Table 2 (continued)

Composites	FS [MPa]	FM [GPa]	DTS [MPa]	CS [MPa]		
Surefil	110.5 ^{bcd} (24.3)	7.4 ^b (2.0)	31.9 ^{bc} (7.8)	180.9 ^a (82.2)		
Prodigy Condensable	102.4 ^{bc} (32.3)	9.1 ^{bc} (1.4)	32.2^{bc} (5.7)	141.5 ^a (25.7)		
Solitaire 2	100.2^{bc} (9.4)	7.7 ^b (0.5)	32.0 ^{bc} (2.9)	285.0 ^c (26.7)		
Quix Fil	99.5 ^{bc} (29.4)	7.6 ^b (1.2)	42.7 ^{de} (7.8)	212.7 ^{abc} (30.3)		
Solitaire 1	82.3 ^{ab} (9.9)	$4.3^{a}(0.3)$	26.4 ^{ab} (2.3)	257.2 ^{bc} (25.6)		
Tetric Condensable	65.4 ^a (17.3)	8.7 ^{bc} (2.0)	38.9 ^{cde} (4.7)	171.0 ^a (39.7)		
Ormocer-based composites						
Definite	120.5 (3.2)	7.1 (0.6)	26.9 (3.5)	223.7 (32.2)		
Admira	88.2 (19.7)	7.9 (3.5)	43.4 (5.2)	208.4 (31.0)		
Compomeres						
Luxat	131.6 ^d (24.2)	12.4 ^d (0.6)	48.9 ^d (5.4)	256.6 ^b (19.4)		
Hytac	123.5 ^{cd} (16.7)	11.7 ^d (0.7)	45.2 ^d (5.4)	227.7 ^b (14.5)		
Compoglass F	104.0 ^{bcd} (21.7)	8.8^{bc} (0.6)	29.7 ^{abc} (3.3)	201.4 ^a (48.3)		
Dyract	101.0 ^{bc} (22.9)	$7.3^{a}(0.8)$	33.1 ^{bc} (4.1)	247.8 ^{ab} (22.7)		
Glasiosite	100.1 ^{bc} (15.5)	$9.0^{\rm c}$ (0.5)	36.4 ^c (5.7)	237.6 ^{ab} (32.6)		
Dyract AP	84.9 ^b (16.0)	7.7^{ab} (0.6)	25.4 ^a (2.2)	201.5 ^a (19.4)		
Elan	81.5 ^b (18.2)	7.9^{abc} (1.0)	25.5 ^a (4.1)	256.4 ^{ab} (45.2)		
F2000 Rasant	31.0 ^a (9.5)	8.4 ^{bc} (0.8)	26.8 ^{ab} (2.8)	218.5 ^{ab} (15.4)		
Flowable composites						
FLOWline	133.0 ^g (13.9)	5.1^{bcde} (0.4)	53.6 ^d (6.5)	286.3 ^{bc} (29.4)		
Arabesk Flow	131.7 ^g (22.0)	$6.4^{e}(0.7)$	38.4 ^{abc} (7.0)	266.8 ^{bc} (36.0)		
Tetric Flow	114.7 ^{efg} (13.1)	4.8^{bcd} (1.4)	38.6 ^{abc} (4.9)	257.3 ^{abc} (48.6)		
Palfique Estelite Low Flow	105.1 ^{def} (15.3)	$2.3^{a}(0.3)$	35.8 ^{abc} (12.0)	239.2 ^{abc} (60.7)		
Reference Flow	103.8 ^{cdef} (10.1)	5.8 ^{de} (0.3)	40.1 ^{bc} (9.1)	299.0 ^c (23.0)		
Revolution	92.0 ^{bcde} (5.0)	4.0^{b} (0.4)	28.4 ^{ab} (6.0)	253.9 ^{abc} (14.3		
Grandio Flow	85.2 ^{abcd} (23.1)	4.1 ^b (0.6)	44.6 ^{cd} (4.4)	233.1 ^{ab} (16.3)		
Definite Flow	81.7 ^{abcd} (31.4)	4.3^{bc} (0.7)	33.4 ^{abc} (9.1)	250.3 ^{abc} (49.3)		
Palfique Estelite High Flow	69.3 ^{ab} (10.3)	$1.6^{a} (0.4)$	38.7 ^{abc} (12.1)	285.5 ^{bc} (42.9)		
Admira Flow	62.8 ^a (11.0)	5.4 ^{cde} (1.4)	31.2 ^{ab} (7.2)	277.3 ^{bc} (19.2)		
Flowable compomeres						
Compoglass Flow	120.4 ^{fg} (10.0)	$5.5^{\text{cde}}(0.3)$	39.6 ^{bc} (7.2)	262.2 ^{bc} (38.4)		
PrimaFlow	95.7 ^{bcdef} (11.8)	4.5^{bc} (0.2)	38.9^{abc} (7.2)	195.1 ^a (10.5)		
Estelite Flow Quick	77.5^{abc} (16.0)	$2.5^{a}(0.3)$	$26.8^{a}(5.0)$	261.7 ^{bc} (45.2)		
Dyract Flow	62.9 ^a (14.7)	$4.3^{\rm bc}$ (0.3)	28.7^{ab} (4.7)	232.2 ^{ab} (29.9)		
Material type					Vol. %	Wt. %
Hybrid Composites	116.6 ^d (23.9)	7.3 ^b (2.3)	32.5 ^b (8.0)	79.0 ^{ef} (3.2)	211.5 ^a (71.1)	60.5 ^{de} (5.7)
Packable Composites	105.9 ^{cd} (28.1)	8.4 ^{bc} (2.5)	34.3 ^{bc} (8.8)	80.3 ^f (3.5)	217.4 ^a (66.5)	63.0 ^e (3.2)
Ormocer-based Composites	104.3 ^{bcd} (21.6)	7.5 ^b (2.4)	35.2 ^{bc} (9.5)	77.0 ^d (0)	216.0 ^a (31.5)	60.6^{de} (0.4)
Nano-hybrid Composites	103.1 ^{bcd} (19)	$5.0^{\rm a}$ (0.8)	40.5 ^d (8.3)	78.4 ^{de} (2.5)	210.8 ^a (63.3)	59.7 ^d (3.9)
Compomers	94.7 ^{bc} (34)	9.2 ^c (1.9)	33.9 ^{bc} (9.4)	74.9 ^c (5.6)	230.9 ^{ab} (35.3)	56.0° (6.8)
Flowable Compomers	89.1 ^{ab} (25.2)	$4.2^{a}(1.1)$	33.5 ^{bc} (8.3)	64.4 ^b (4.1)	237.8 ^{ab} (42.4)	43.3 ^a (4.3)
Flowable Composites	99.8 ^{bc} (27.4)	4.4 ^a (1.6)	38.3 ^{cd} (10)	66.1 ^b (4.3)	264.2 ^b (41.5)	48.4 ^b (6.5)
Micro-filled Composites	73.5 ^a (10.9)	$3.8^{a}(0.8)$	24.2 ^a (3.8)	60.7^{a} (4.0)	246.9 ^{ab} (37.8)	42.7 ^a (3.0)

Data are arranged in descending order of the flexural strength value

Superscript letters indicate statistically homogeneous subgroups within a material category (Tukey's HSD test, α =0.05)

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 Table 3 Influence of fillers—volume and weight—and material categories on the mechanical properties

Variables	Vol.%	Wt.%	Material type
Flexural Modulus of Elasticity (FM)	.439	.253	.037
Flexural Strength (FS)	.345	.172	.016
Compressive Strength (CS)	.229	.074	.065
Diametral Tensile Strength (DTS)	.129	.067	.023

The higher the partial η^2 values, the higher is the influence of the selected variables on the mechanical properties

most common reason for failure for observation periods lower than 5 years was restoration fracture, followed by secondary caries, whereas for longer observation periods— 6–17 years—secondary caries was the main reason for replacement of fillings [11]. The predominant reason for failure of composite restoration placed in larger cavities was found to be fracture also for periods longer than 11 years [12, 13]. This trend in increased fractures in fillings placed in load-bearing area, compared to the decay before, was certainly caused by the extension of the indication for composites to larger multi-surface class II cavities. But also, the compromise necessarily to create a universal composite by decreasing the filler size in order to improve aesthetics could have leaded to decreased mechanical properties.

ISO 4049 classifies two types of light-curing direct filling resins according to the flexural strength to: Type 1:

fillings for the occlusal areas, flexural strength \geq 80MPa and Type 2: fillings for other indications, flexural strength \geq 50 MPa. Accordingly, our result showed that the only material categories which cannot be used in occlusal area are the micro-filled composites, since a high amount of the flowables also exceed the limit value of flexural strength. But a material with a low modulus of elasticity, particularly placed in load-bearing areas, will result in a higher deformability under masticatory stresses, having as final consequence catastrophic failures. It is, thus, very important to introduce also a modulus of elasticity limit for composites to be paced in load-bearing areas.

Micro-filled composites were developed to satisfy the need for an aesthetic, more polishable composite. These materials have a very fine particle size, mostly of colloidal or fumed silica (0.04 µm), dispersed in a resin matrix (Table 1). The very large surface area of the filler significantly limits the volume of filler that can be incorporated. Consequently, the filler volume does not differ statistically compared to the flowables compomers and is even lower compared to the flowable composites (Table 2). The micro-filled composites presented the lowest mechanical properties as material category, being quite homogeneous within the group. It was shown that the use of micro-filled composites results clinically in chipping occurring three to four times more often than by using conventional composites resins [14], which probably explains why micro-filled composites fail catastrophically

Fig. 1 Flexural strength and modulus as function of filler weight and volume. An Epanechnikov kernel function [9] was used for contouring the areal density of the measured data



when used as posterior restorative materials on stress-bearing surfaces [15]. On the other side, it was shown that some micro-filler composites (Durafill VS, Metafil CX, Heliomolar RO) revealed a lower sliding wear in vitro compared to compomers (Dyract AP, Compoglass F, Compoglass and Hytac) or hybrid composites (Estilux Hybrid, Arabesk, Artglass, Charisma F, Pertac II, Charisma, Degufill Ultra, TPH Spectrum, Z100, Tetric classic, Pertac Hybrid) [16].

Using resin composites successfully in load-bearing surfaces of posterior teeth implies high mechanical properties, good wear resistance, high fracture toughness, small inherent flaw size and high fatigue resistance of the restorative material. Evidence shows that wear may be of minimal importance for restorations of small to moderate size. However, the literature does suggest that failure rates are higher for larger restorations and that wear may still be a significant mode of failure for patients with bruxing and clenching habits [17].

The size of filler particles incorporated in the resin matrix of commercial composites has continuously decreased over the years from the traditional to the nano-hybrid materials in order to obtain materials with improved aesthetics. But the larger surface area to volume ratio of the fillers present in the nanofilled materials also tend to increase the water uptake and resultant degradation of the filler/matrix interface, affecting thus the mechanical properties when compared to a microhybrid composite (Filtek Z250) [18]. Compared to hybrid composites, the filler weight and volume of the nano-hybrids as well as FS and CS were similar. The material category is characterising by a lower modulus of elasticity but a better DTS. Nevertheless, the modulus of elasticity of dental composites is regarded as a fundamental property, since a material with a low modulus will more readily elastically deform under functional stresses, questioning the clinical success of using nano-composites in stress-bearing area.

The high-viscosity packable composites were developed in an attempt to limit wear and fracture of the restoration within the body and at the margins, to reduce polymerisation shrinkage and the technique sensitivity, including the ability to obtain a better proximal contact in the final restoration. The materials purportedly offer the advantages of having handling characteristics similar to those of amalgam, having physical properties better than those of traditional hybrids and micro-fills. Comparing the packables-Solitaire, Surefil and ALERT-with a packable ormocer-definite-and a hybrid composite-Tetric Ceram-it was shown that ALERT exhibited the highest flexural strength, modulus and fracture toughness, but the lowest wear resistance, whereas Solitaire-filled with porous glasses-behave completely opposite, showing low mechanical properties and the best wear resistance. Surefil exhibited better mechanical properties and wear than Tetric Ceram and Definite, leading the authors to conclude that fracture and wear behaviour of composite resins are highly influenced by the filler system and not by the material category [19]. Other in vitro studies have found that, in general, the mechanical properties of packable resin composites (ALERT, Pyramid-Dentin, Pyramid-Enamel, Solitaire, SureFil) are similar to typical hybrid resin composites (Z100) except Solitaire which performed worse [20, 21]. These results are also confirmed by our tests showing large differences within the material category, but none of the measured physical properties of the packables as material group differed significantly from the group of the hybrid composites. Thus, from laboratory tests, no advantages of the packables in stress-bearing areas are expected when compared to hybrid composites.

Another purported advantage of high-viscosity packables resin composites was that they have better handling characteristics making it easier to establish an adequate proximal contact, since they more effectively distend the matrix band during placement than do less viscous composites. Studies showed however no difference in the tightness or contours of proximal contacts created with either a packable or hybrid composite [22, 23]. Also for different application techniques—bulk and incremental—the proximal contact strengths produced by composites of different viscosities was shown to be comparable for the packable (Solitaire) and the traditional (medium-viscosity) hybrid (Tetric Ceram) and better than by using flowable materials (Tetric Flow) [22].

The polymerisation contraction of the packable composites was shown to be similar to or higher than that of the non-packable composites [20]. In terms of clinical performance, packable resin-based composites placed using different adhesive systems generally were considered satisfactory for the restoration of posterior teeth after 2 years [24–26]. A comparison after 3.5 years showed that the clinical performances of packable (SureFil) and conventional hybrid composite resins (Spectrum-TPH) were similar and satisfactory for the restoration of Class 1 and moderate-sized Class 2; nevertheless, in large intracoronal Class 2 molar restorations, an increased risk of bulk fracture was attributed for both materials types [27].

In an attempt to overcome the problems created by the polymerisation shrinkage of conventional composites, the organically modified ceramics (Ormocers) as new material class were developed [28]. Having a very similar coefficient of thermal expansion to natural tooth structure, the materials were formulated as a novel three-dimensionally cross-linked inorganic–organic polymer, synthesised from multi-functional urethane- and thioether(meth)acrylate alkoxysilanes as sol–gel precursors. Alkoxysilyl groups of the silane permit the formation of an inorganic Si–O–Si network by hydrolysis and poly-condensation reactions. The methacrylate groups are available for photochemical polymerisation. These materials proved to generate lower wear rate compared with composites [29, 30] and an

shrinkage equal to that of hybrid composites, despite having less filler content [31]. But due to problems with upscaling of prototypes and handling properties, conventional methacrylate had to be added to the ormocer matrix of the first commercial products, diminishing the initial promising advantages. As a material group, with the limitation of the low amount of tested ormocer composites, their mechanical properties do not differ compared to the hybrids. However, new developed, still experimental ormocers, synthesised from amine or amide dimethacrylate trialkoxysilanes and being dimethacrylate-diluent free, showed a clearly improved flexural modulus of elasticity compared to the ormocer-based composite Definite [32], promising a revival of this material category.

Compomers (polyacid-modified composite resins) were introduced in an attempt to combine the benefits of both glass ionomers (e.g. fluoride release, chemical bonding) and composite resins (e.g. ease of use, better polishing, aesthetics) and have been marketed for use in all classes of restorations. Laboratory studies have shown however lower mechanical properties (e.g. compressive and flexural strength) for compomer materials compared with hybrid composites [33]. Compomers (Compoglass F, F2000, Dyract) were found to be also less resistant to crack propagation showing lower fracture toughness than hybrid composite resins (Tetric Ceram, Z250, Esthet-X) and, thus, less adequate to be used in load-bearing areas of posterior teeth [34]. In general, fracture toughness was found to be highest in the more heavily filled resins, independent of degree of conversion in the matrix [35], and compomers are less filled than hybrid composites (Table 1). A large variation in mechanical properties within the compomers was found in our study. Whereas, the FS of some materials was below 80 MPa (F2000 Rasant) performed some of the tested compomers as good as hybrid or packable composites.

In a 3-year study by Wucher et al. compomer restorations (Dyract) exhibited significantly greater occlusal wear and degradation of marginal integrity compared with the composite (Spectrum-TPH) restorations [36]. An accelerated wear in restorations receiving direct contact was measured for the same compomer also in a 3-year study in primary molars. Nevertheless, due to the excellent handling characteristics and the low failure rate, the authors suggested that this compomer (Dyract) is a reliable restorative material [37]. Several compomers—Luxat, Hytac—presented in our laboratory study even consistently higher mechanical properties than Dyract, especially in regard of FM, but excellent clinical data are, with some exception, only available for the Dyract material group (Dyract [37], Dyract AP [38], Dyract eXtra).

The loss of marginal integrity was shown to be the main cause of restoration failure in Class I and II cavities of permanent posterior teeth in a 4-year clinical study by using Hytac, with an average annual failure rate of 3.85% and a probability of a clinically acceptable after 4 years calculated to be 89% (Kaplan–Meier) [39]. Since the polymerisation shrinkage of Hytac was comparable to the hybrid composite Tetric Ceram [40], the mechanical properties even slightly higher (Table 2), and the above study unfortunately does not include a reference material, the reasons for loss in marginal integrity must be searched elsewhere.

Compared to the hybrid composites, compomers showed as material category a significant lower FS but a higher FM, whereas the other mechanical properties were comparable.

Considering the variation of the FS and FM as function of the filler volume without taking in account the material category, a trend to enhance the mechanical properties until a filler volume of ca. 60% was observed. It seems that introducing a higher volume of filler than 60% will probably also introduce a higher amount of defects. Since the modulus of elasticity decrease exponential with the volume of voids, the reinforcement effect expected through adding a higher amount of filler is thus diminished.

The modulus of elasticity increased continuously with the filler weight; on the other hand, the flexural strength increased just until a filler weight of ca. 80%. It was also shown that composites with considerably low or high filler content (<60% or >80% by weight) have significantly lowered tensile fatigue resistance when evaluated by the staircase method [41]. In consequence, an increased filler level does thus not necessarily improve the fatigue resistance or the flexural strength of a resin composite, even if the modulus of elasticity continues to increase.

Nevertheless, due to the very complex composition of the tested materials and considering that the evaluation of the variation of mechanical properties with the filler ignore the chemical diversity of the matrix, the filler, as well as the filler size, shape and distribution, the results should be taken only as a trend. Consolidated findings are possible only by systematically variation of all parameters in experimental materials, tests which are missing so far, due to the complexity of such attempts.

Further, the method to measure the displacement, done like in the ISO standard by read-out of the crosshead displacement, could be a point of criticism, due to eventually induced errors as a result of the machine compliance. A measurement of the displacement directly on the sample is however difficult to realize, due to the miniaturised sample geometry.

Conclusion

1. Direct comparison of composite efficacy in clinical studies is very difficult, mandating the kind of scientific study that this paper represents.

The variability of the mechanical properties measured within a material category was found to be very large, making it inequitable to predict the performance of a single material from its belonging to a material group. Less difference were found between hybrid, packable, ormocerbased, nano-composites and compomers, attesting higher mechanical properties than the flowables composites, flowable compomers and micro-filled composites, rejecting thus our initial null hypothesis. The filler volume was shown to have the highest influence on the measured properties, attending a maximum FS and FM at a level of ca. 60%, whereas such dependence was not measured for DTS or CS. A good correlation was found only between FS and FM which also correlates well with the filler volume, whereas DTS and CS were less sensitive.

 ISO standards should incorporate a revised limit for FM of load-bearing composites.

Conflict of interest The authors declare that they have no conflict of interest.

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