

Department of Clinical Dentistry Faculty of Health Sciences

Mechanical properties of bulk fill compared to universal composites

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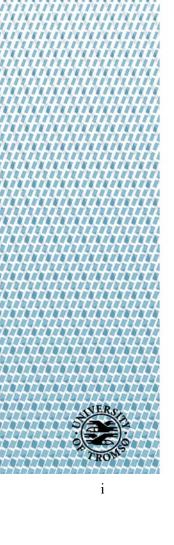


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Abstract

Objectives: Bulk fill composites are becoming increasingly popular due to claimed facilitated placement and curing. Varying results on the mechanical properties and depth of cure have been reported, however. The aim for the present study was to compare flexural strength, E-modulus, secant modulus, Knoop hardness and depth of cure of bulk fill and universal composite materials, subjected to different storage times and artificial aging.

Materials and methods: Two bulk fill (1 low and 1 high viscosity) vs. 2 universal composite resin based materials were subjected to mechanical and depth of cure testing. Evaluation was performed according to ISO4049:2009 with slight modifications regarding mould size and storage time/temperature. To simulate aging, increased storage temperature was used for additional analysis. Three groups were made of each material, 37°C (24 hours and 7 days) and 57°C (7 days). Mechanical testing was performed after assigned storage time. Micro CT was used for complimentary evaluation on 2 randomly selected samples. All data were tested for normality and analyses were performed with a 95% confidence interval.

Conclusion and significance: There were significant differences in mechanical properties among the tested materials. Storage and aging affected the materials in a dissimilar manner. The low viscosity bulk fill composite evaluated showed lower moduli and hardness due to filler loading in comparison to the other materials tested, but a significant higher flexural strength and depth of cure with increased storage temperature in comparison. The increase could be explained by the monomer composition in the material. The result indicates that the term "bulk fill" seems irrelevant since it is not a discriminating factor for assessment of different mechanical properties. More relevant information concerning filler loading and monomer content should be given and assessed. Negative effects on the mechanical properties can be obtained due to voids within the materials.

Introduction

In dentistry, conservative direct treatment with polymer resin based restorative materials used in dentistry (hereafter referred to as composites) was introduced at the late 50s, early 60s (1). After its entry, composite has undergone radical improvements from its early origin (1). Still, there are limitations concerning handling. Differences in filler type and content, photo initiators, inhibitors and monomer composition confines universal composites to 1,5-2mm oblique layers.

Reasons among others are that, best bond strength and lowest polymerization stress have been reported with the incremental layering technique, due to the decrease of the configuration factor (C-factor) (2-5). The C factor describes the relationship between the cavity configuration and development of stress. It is dependent on the bonded vs. unbonded areas as well as volume of the composite (6, 7). A high C-factor will inversely increase polymerization stress during cure of photo-polymerized composites, independent of composite used (7, 8). Examples of effects caused by high polymerization stress are; interfacial/cohesive failure, ingrowth of bacterial biofilm, cuspal flexure, secondary caries and failure of the restoration (9-11).

By introduction of more flexible and multi-functional monomers (e.g. high density UDMA, Bis-EMA etc.), new initiator systems (e.g. Ivocerin), shrinkage stress relievers and/or polymerization modulators (table 1), improvements of decreased polymerization stress and higher degree of conversion (DC) can be achieved (12, 13). Bulk fill composites is an example of a different class of materials developed with the attempt to decrease polymerization stress and having an adequate degree of conversion even when increments up to 5 mm are placed (14).

While universal composites are proven to attain the best properties when used in combination with the incremental layering technique, bulk-fill composites have several methods used for introducing material to the cavity (3, 15). Due to their different viscosities (i.e. high or low) some are manipulated with instruments like universal composites (e.g. Tetric EvoCeram bulk fill), while others are not (e.g. SDRTM and Sonic fill) (14, 16, 17). It can be speculated that the use of instruments when placing composites in the cavity will make the process more technique sensitive, due to the risk of introducing defects (e.g. voids etc.) compared to materials that can be more easily placed (e.g. low viscosity bulk fill composites). A high number of porosities/defects will affect the mechanical properties negatively. One reason

could be that no cross linkage of polymers is achieved in the area of a void. To the knowledge of the authors, however, there seems to be limited data on the subject at present. For evaluation of internal structures in relation to mechanical properties, Micro-CT offer a non-invasive suitable method for evaluation of the presence/distribution of voids in polymer-based materials (18-21).

The difference in viscosity is mainly due to filler content and/or resin composition (22). Low viscous bulk fill materials may be indicated as a base, before high viscous universal composites is deposited in direct occlusion. The main reason for placing a composite material of high viscosity in occlusion is because low viscosity bulk fill composites are less wear resistant due to their lower filler content and hardness (23, 24). They are therefore not recommended to be in occlusion, especially posterior due to inferior wear resistance (25). Still, composite of low viscosity may adapt more sufficiently to the surface of the cavity, leading to lower failure rate – as shown by Figueiredo et. al. with the use of universal low viscosity composites (2). These features are claimed to facilitate handling and reduce time spend reconstructing, especially for larger cavities. However, neither any improvements in survival rate of occlusal restorations, nor any decrease in polymerization stress has been reported using universal composites (15, 26). For low viscosity bulk fill restorations covered with universal high viscous composite occlusal has shown an annual failure rate comparable with cavities restored with universal composite alone (27).

Bulk-fill composites (both high- and low viscous) can according to their manufacturers, and depending on material, be deposited in layers up to 4/5mm before each curing cycle (14, 16, 17). Still, there are concerns about mechanical properties such as flexural strength, E-modulus and surface hardness (24). Therefore, uncertainty about the advantage of bulk fill materials in comparison to universal types of composites have been raised (24).

Composites are exposed to a high dynamic stress in the oral cavity – therefore the materials must endure high mechanical and physical stress. Several parameters are used to describe a materials ability, such as flexural strength, modulus of elasticity and micro hardness (28-30). Flexural strength describes the materials resistance to fracture and studies published indicated the correlation between high flexural strength and high fracture resistance (28, 31-34). ISO 4049:2009 state a minimum flexural strength requirement of 80 Mpa for light curing composites (35). Modulus of elasticity (Young's modulus, or E-modulus) is considered as one important feature for mechanical stability (29). Environmental conditions have been shown to

affect the E modulus, e.g. storage in water or alcohol as well as temperature and time have a diminishing effect on the modulus (36-40). Reports of correlation between low E-modulus and marginal fractures have been made, however, not conclusively substantiated (41). The hardness of a composite, or "micro hardness", is best determined by Knoop hardness (29). Micro hardness correlates with wear resistance, i.e. a higher hardness – yielding a higher wear resistance (42). Micro hardness also correlates with modulus of elasticity, viscosity and degree of polymerization (43).

Hypothesis:

The null hypothesis formulated for the present study were that no differences concerning flexural strength, E-modulus, secant modulus and Knoop hardness between universal composites and bulk fill composites could be recorded, and that storage time or artificial aging would in addition not affect the properties tested.

Aim:

To evaluate flexural strength, E-modulus, secant modulus, Knoop hardness and depth of cure of bulk fill composites compared with universal composites after water storage and simulated aging, using micro CT as a complimentary evaluation method.

Materials and methods:

Composites:

Two Bulk fill composites, SDRTM (low viscosity) (Dentsply DeTrey, Konstanz, Germany) and Tetric EvoCeram Bulk Fill® (high viscosity) (Ivoclar/Vivadent, Schaan, Lichtenstein) were compared with two universal composites, Ceram X Universal® (Dentsply DeTrey, Konstanz, Germany) and Tetric EvoCeram® (Ivoclar/vivadent, Schaan, Lichtenstein). The content of monomer and fillers of the materials investigated are presented in table 1. All samples were made in a shade comparing to A3, with exception of SDRTM that had one shade only.

Composites	LOT nr.	Monomer content	Filler content	Other
Ceram X Universal	1508000827	UDMA, Bis- EMA, TEGDMA	79 % (wt %)	Spere TEC TM : Combination of large and
(CXU)		and methacrylic	Pre-polymerized SphereTEC TM , non-	small fillers
(6125)		polysiloxane	agglomerated barium glass	
		nano-particles.	and ytterbium fluoride.	
Smart	1510000225	SDR™ patented	68% (wt %)	Claims low shrinkage
Dentin		high density	Barium and strontium	stress due to high density
Replacement		UDMA, di-	alumino-fluoro-silicate	UDMA and high glass
(SDR TM)		methacrylate resin	glasses.	filler loading. In addition
		and di- functional diluents.		to polymerization modulators embedded in
		diffuents.		monomers
Tetric	S01562	17% (wt %)	82-83% (wt %)	monomers
EvoCeram	501302	Bis-GMA,	Barium glass filler,	
(EC)		UDMA and	ytterbiumtrifluoride,	
		ethoxylated bis-	mixed oxide and	
		EMA.	prepolymer.	
Tetric	U17294	21% (wt %)	79% (wt. %)	Ivocerin initiator,
EvoCeram		Bis-GMA, bis-	Barium aluminium silicate	patented filler with low e-
Bulk Fill		EMA and	glass, ytterbium fluoride,	modulus.
(ECB)		UDMA.	spherical mixed oxide and prepolymer.	

Table 1: Content as described in "Scientific Compendium" from each manufacturer.

Sample preparation:

For each of the materials tested, 21 samples were made. Eighteen of the samples for each material were divided into 3 groups for the testing of mechanical properties after storage at different times and temperature (figure 1). Three samples of each material were subjected to tests for depth of cure.

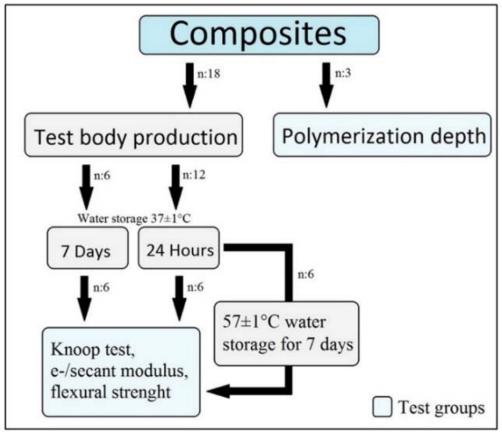


Figure 1: Flowchart of sample production and testing.

For evaluation of the mechanical properties, samples were made according to ISO 4049:2009 for flexural strength. SDRTM was inserted with the supplied capsules without using any hand instruments. The other materials were inserted from their compules and placed by hand instruments. All materials were inserted in one layer and covered with a thin polyethylene sheet with a glass block placed on top. Great care was taken to avoid voids and ensure proper application. The composite was light cured in overlapping sections in accordance with ISO 4049:2009 after removal of the block.

The time used for each curing cycle was as recommended by the manufacturers. For SDRTM and Ceram X Universal (CXU), 20s as given at an irradiance >550mW/cm². For EvoCeram (EC) and EvoCeram Bulk Fill (ECB), 10s as given at an irradiance >1000mW/cm², or 20s (<1000mW/cm²). All curing was performed with a Bluephase G2 light curing unit (LCU) (Ivoclar/Vivadent, Schaan, Lichtenstein) set to "high mode".

This LCU has been shown to have a homogenous light beam profile (44-47). The irradiance was controlled regularly before sample preparation for each group, using a calibrated MARC resin calibrator (Bluelight Analytics, Halifax, Canada). Mean irradiance measured to 1330±42 mW/cm² (table 2).

Material	Output: (mW/cm ²⁾		Cure time:	RE:
	Before	After		
CXU	1391	1313	20	27 820
SDR	1366	1294	20	27 320
EC	1326	1285	10	13 260
EC 20S	1326	1285	20	26 520
ECB	1286	1312	10	12 860
ECB 20S	1286	1312	20	25 720

Table 2: LCU Output measurements before and after cure. RE: Radiant exposure (output x time).

After curing, the samples were removed from the mould and excess eliminated with Silicon Carbide paper (800 grit, Buehler, Lake Bluff, USA) and visually inspected. The samples were then individually placed directly in glass containers containing double distilled water; grade II (ISO 3696:1987) and stored in accordance to its respective test group (figure 1). For the purpose of storage, two identical heating cabinets (Memmert, VWR International, Radnor, Pennsylvania, US) were used for the two conditions determined (37±1°C and 57±1°C). The temperature of 37±1°C was used to simulate oral environment, and 57±1°C to give a 4 times aging effect – meaning that 7 days at 57°C was comparable to 28 days of aging in 37°C, based on the Arrhenius equation (48).

After storage, the samples in their containers were removed from their respective heating cabinet and given 30 minutes to recover to room temperature (\approx 23±1°C). The aim was that all samples should reach the same temperature before start of the test. The samples were then carefully dried using a thin clean paper towel, and submitted to testing.

To evaluate eventual bias effects caused by differences in recommended polymerization schemes, one additional group of EC (EC20s) and ECB (ECB20s) was made using 20s (in "high mode") instead of 10s, to assess the effect of the same cure time as SDRTM and CXU. These two groups were stored for 24 hours and then tested.

Evaluation of mechanical properties:

Three-point bending test was performed according to ISO 4049:2009 using a Zwick/Roell Z050 (Ulm, Germany, equipped with TestXpert II v.3.4 software). A Heidenhain ND 287 (software version V1.07) (Traunreut, Germany) digital measuring instrument connected to the Zwick/Roell was used for measurements of the specimen dimensions. The distance between the bars were 20 mm and the force was applied vertical with a cross-head speed of 0,75mm/min until fracture. Results were calculated relating to the mechanical factors described below:

Flexural strength

$$\sigma = \frac{3FL}{2bd^2}$$

(F = axial load, L = length of sample, b = sample width, d = sample depth)

E-Modulus

This factor, visualized as the angle of the elastic part of the stress/strain curve, describes the ability for a material to resist elastic deformation (22).

$$E = \frac{FL0}{A0\Delta L}$$

(F = force exerted, L_0 = sample length pre bending, A_0 = Cross section of sample, ΔL = change in length)

Secant modulus

To complement the description of the loadbearing properties of the test materials, secant modulus was additionally calculated. In contrast to E-modulus, the secant modulus describes the slope from 0 to a given point on the stress-bearing curve (22). It is calculated using the same formula as for E-modulus, only with an altered point of deflection, in this experiment the point of 60% flexure was used (49). The aim was to provide a more complex description of the total stress/strain of a material with viscoelastic properties (22).

Knoop Hardness

For evaluation of surface hardness, Knoop hardness was tested for on the same samples used for the 3-point bending test and in conjunction with the latter. A Zwick/Roell Indentec ZHVμ-A test machine (Ulm, Germany) equipped with Zwick/Roell ZHμ HD Micro Hardness Software (Ulm, Germany) was used. Test conditions were set to 50g load with a dwell time of 15s. This setting was determined after pilot tests that provided stable values. Four indentations per sample (2 on each fragment) were performed and the mean value was used to describe the Knoop hardness of the specimen.

Micro CT evaluation

Two random selected samples (containing 2 fragments each) from each material, regardless of group, was evaluated for defects and irregularities using a Micro-CT scanning (Skyskan 1272, Bruker, Kontich, Belgium). The samples were stored in a desiccator using dehydrated silica to enhance the contrast between pores and material prior to the Micro-CT scanning.

Depth of cure (DOC)

ISO 4049:2009 was used as a reference for the depth of cure, with exception of the diameter of the mould due to availability. The diameter used in this experiment was 6 mm in contrast to 4mm stated by the standard. The measurements were performed straight after cure. "High mode" (1330±42mW/cm²) was used with the same setting as in the mechanical test and

curing time as described by the manufacturer. However, when high mode was used, no discrimination in cure depth between the materials tested could be observed – due to complete cure of the entire sample. Therefore, a second test in "Low mode" (783±31mW/cm²) where performed. All composites in this group were cured for 20s in accordance with the manufacturers recommendation respectively (16, 17, 50, 51). Sample dimension were measured using a Heidenhain ND287 height measurement device (Heidenhain, Traunreut, Germany), to a precision of 0,001mm. Depth of cure was defined as the total polymerized sample depth divided by 2 (35). A total of 3 tests per material were performed, as required by ISO 4049:2009.

Statistical analysis

All data were tested for normality (Shapiro-Wilk). The data for the Knoop hardness and depth of cure test fulfilled the assumptions for One Way ANOVA parametric test. The E – modulus, secant modulus and flexural strength data did nor fill the assumptions for a parametric test; thus, Kruskal-Wallis non-parametric One Way ANOVA were used. Analyses were conducted using Sigmaplot 13 (Systat Software Inc., San Jose, CA, USA), at a level of significance set to 5%.

Results

The results achieved are presented in table 3.

		(mPa)	(mPa)	(mPa)		(mm)
Composite:	Group:	Flex.strgt.	E-mod	Secant-mod	KH	Poly depth
CXU	24hours:	97±16	7780±128	7470±145	50±2	5,2±0,02
	57degree:	85±6	7340±149	7080±118	46±2	
	7days:	102±13	7990±225	7690±229	51±2	
EC	24hours:	93±7	6650±128	6400±187	38±3	4,6±0,3
	57degree:	72±4	5520±144	5330±126	39±2	
	7days:	80±11	6320±100	6060±115	38±2	
	20s	83±7	6510±255	6280±251	40±2	
ECB	24hours:	98±6	8110±146	7790±121	45±1	6,6±0,4
	57degree:	76±4	6840±154	6570±160	45±2	
	7days:	82±11	7380±115	7090±74	46±2	
	20s	95±8	7860±90	7548±109	48±1	
SDR	24hours:	103±4	4510±426	4390±395	24±1	7,8±0,06
	57degree:	114±4	4970±194	4830±198	29±1	
	7days:	101±11	4610±296	4480±278	26±2	

Table 3: Mean values and standard deviation (respectively) for all performed tests.

Flexural strength

At 24 hours of storage (37°C) all materials displayed flexural strength values well above the limit set in the ISO standard (i.e. 80 MPa). With increased storage time (7 days) at 37°C, the materials EC and ECB showed a decrease in flexural strength to a lager degree compared to CXU and SDRTM respectively (table 3). The difference recorded between the materials was, however, not significant (p>0.05). In contrast, with increased storage temperature (57°C), a change was recorded with a pronounced increase in flexural strength for SDRTM and a decrease for the other materials tested (table 3). The difference was significant (p<0.05) and the increase was also significant when SDRTM was compared at 24 hours (37°C) and 7 days (57°C). For EC and ECB, the flexural strength decreased below the limit of the ISO standard after 7 days' storage (57°C).

E- and secant moduli

When the moduli were tested and calculated for, no significant difference between the materials CXU, EC and ECB was recorded. Concerning changes with time and temperature, ECB showed the largest decrease in moduli after 7 days both in 37°C and 57 °C environment, despite having the highest value after 24 hours (37°C) of storage. SDRTM showed a comparable lower E- and secant moduli (p<0.05) at all test occasions, but not significant to the material EC (table 3). As expected, the secant modulus as a test parameter showed slightly lower values than that of the E-modulus for all materials tested.

Knoop hardness

CXU showed highest Knoop hardness at all test occasions while SDRTM displayed the lowest (table 3). The differences between the materials were significant (p<0.05) at all conditions tested for with exception for CXU and ECB at 7 days (57°C). When changes depending on time and storage temperature within each group of material were tested for, no statistical changes in Knoop hardness could be recorded, with one exception, however. CXU stored in 57°C (7 days) had significantly lower value (46±2 KH) than when stored in 37°C (7 days) (table 3).

Micro CT evaluation

A qualitative assessment of size, number of voids, and their distribution are presented in table 4. A representative illustration from each material is given in figure 2.

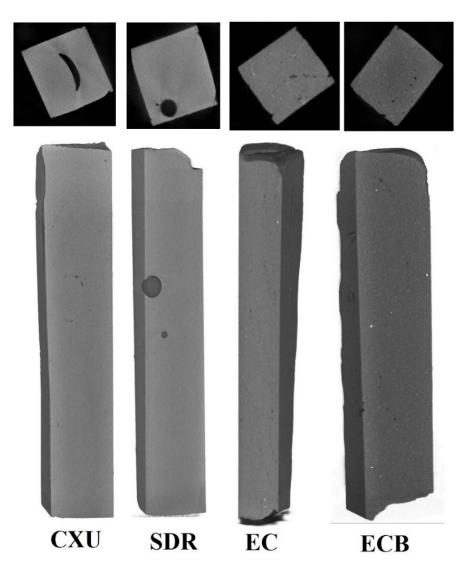


Figure 2: Illustrations of 3D modeled Micro CT scanning.

Material	Micro CT assessment
CXU	Fairly homogenous. Voids show some tendency to follow the interface between the different composite layers. Small voids, with exception of a large operator induced void between two composite layers.
SDR	Homogenous. Voids seem to be located in connection to the surface of the mould. Has the fewest voids in the samples seen, however, a few large voids ("air bubbles") are present.
EC	Slightly heterogeneous. Random distribution, but clear tendency for accumulation between composite layers. Small voids, radiopaque spots also visible.
ECB	Slightly heterogeneous. Random distribution, but tendency for accumulation between composite layers. Small voids, radiopaque spots also visible.

Table 4: Evaluation of the test materials based on micro CT scanning of 2 random samples from each material.

Depth of cure:

The results are presented in figure 3 and the differences were significant for all materials (p<0.05). All composites respectively fulfilled the requirements claimed by their manufacturer. Both bulk fill materials showed higher depth of cure than the universal composites.

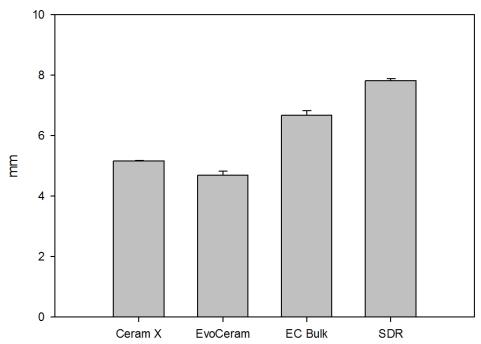


Figure 3: Polymerization depth of all composites. X axis depicting polymerization depth in millimeters. The error-bar on top of each bar indicates standard deviation. Results presented were attained using "Low mode" setting on the LCU.

Discussion

Aging due to temperature increase affected the materials in a dissimilar manner. No differences regarding flexural strength between the materials were shown at the lower storage temperature. At 57 °C however, the differences were more pronounced. SDRTM showed a significant increased strength in comparison and also the moduli for SDRTM increased. Concerning Knoop hardness, time and temperature seemed to have less effect, the differences between the materials was however, more pronounced.

Based on the results obtained, the null-hypothesis stated was considered as rejected for the low viscosity bulk fill material tested.

In the present study, standardized evaluated methods (i.e. ISO 4049:2009) were used with slight modifications concerning the storage conditions (i.e. 7 days instead of 24 hours for two of the test groups). The modification was done to increase the possibilities of discrimination between the materials tested. In that respect, also the temperature was set to 57°C to increase the aging effect in accordance with the Arrhenius equation suitable for polymers (48). That will give a possibility to study changes in the materials over time. Increasing the temperature to simulate aging is a well-established method for studying aging effects in polymer resin based materials. Using the Arrhenius equation, a 20°C increase will correlate to 4 times increased aging, meaning that 7 days of storage in 57°C will correspond to 28 days in 37°C. Twenty – eight days, is still a short time and if longer times could have been used, a more pronounced aging effect could have been evaluated.

As the same sample was used for the 3-point bending and Knoop hardness test – the mechanical properties can also be more accurately validated as they originated from the same sample lowering the risk of bias due to differences in handling. In addition, evaluation of the internal structure of the sample using micro-CT examination gave the opportunity for increased insight into the variability seen within – and between groups.

For measurements of depth of cure, a mould diameter of 6mm, instead of the 4mm recommended by the ISO 4049:2009, was used. A larger diameter has been shown to yield higher and more consistent values than the 4 mm mould diameter stated by the standard for evaluation of depth of cure (52). The reason is due to the diameter of the mould. If that is close to the one of the LCU tip diameter, it will capture most of the irradiance emitted from the LCU, and therefore result in a more effective cure of the material (52).

The number of samples tested in in vitro studies are often restricted for several reasons, but should always be larger than 5 (35). As for the present study, six samples from each group were tested. Still, that amount per test group may restrain the level of significance. It should be noted that low power (small number of samples in each group) increases the risk for type II errors – meaning that a higher power might have induced more statistical significant results. This may affect the results achieved in the present study, even though tendencies were apparent.

Radiant exposure (irradiance x time) for EC and ECB was half of that compared to CXU and SDRTM, in accordance with the instructions for use from the manufacturers. Comparison of the 20s group of EC and ECB vs. their equivalent 10s group (24 hours) showed no statistical differences for mechanical parameters tested for, with exception from a slight increase in Knoop hardness for ECB (not shown in table). These findings support the recommended curing scheme for EC and ECB by the manufacturer (16, 50).

SDRTM was chosen for this experiment along with 3 other high viscosity composites due to its frequent use as a bulk fill composite in Troms county, Norway (53). SDRTM was also one of the first bulk fill materials launched more than 7 years ago. Evaluation of differences in mechanical and physical properties, between a low viscosity based bulk fill system versus a high viscosity bulk fill system seems important since differences between universal low and high viscosity composites are evident mainly due to differences in filler loading (54-57).

The stability of, or slight increase of the mechanical properties after 7 days (37°C) for SDRTM could be explained by post irradiant processes (58). Interestingly, for SDRTM the increased temperature, simulating aging, also increased the flexural strength (i.e. highest flexural strength results achieved). That was in contrast compared to any of the other materials tested.

The difference can be explained by differences in monomer content with effect on cross-linkage between the materials tested. Difference in monomer composition have shown to affect the degree of conversion of composite materials (36, 59). SDRTM contain UDMA or modified UDMA. UDMA is known as a monomer with high mobility and thus, a high/increased degree of conversion can be achieved (60). It is therefore plausible to assume that the new developed isomer of UDMA (i.e. modified UDMA) will have the same properties. The mobility of traditional UDMA (MW=470g/mol) or its isomer (high density UDMA – MW=859g/mol) during the light curing and the post curing process will therefore lead to higher degree of cross-linkage and enhanced flexural strength and moduli (13, 37) also

shown for SDRTM at 57°C in the present study. As reported by Gajewski et. al, UDMA presented the highest flexural strength of a selection of monomer commonly used in resin based composites (TEGDMA, Bis-EMA, Bis-GMA) (37). This was explained by stronger hydrogen bonding potential caused by hydrogen interactions with the hydroxyl and urethane groups in the structure of UDMA (37). Since these type of bonds also is dependent on the molecular weight (i.e. the higher weight the stronger ability to bond) it is plausible that higher density UDMA in SDRTM (table 5) will enhance these properties (36, 48, 61). In addition, a high depth of cure for SDRTM was recorded in the present study (Fig 3). The mobility, in addition to high monomer vs. lower filler ratio may further facilitate the reaction process in SDRTM, enhancing the cross-linking over time. Especially when an increase in temperature simplify monomer movement in the post curing phase (58, 60).

Monomer	Full name	Weight
TEGDMA	Triethylene glycol dimethacrylate	286 g/mol
Bis-EMA	Ethoxylated bisphenol A glycol dimethacrylate	540 g/mol
Bis-GMA	Bisphenol A glycol dimethacrylate	513 g/mol
UDMA	Urethane dimethacrylate	470 g/mol
UDMA (SDR TM)*	Urethane dimethacrylate*	859 g/mol

Table 5: Monomers and their molecular weight. *SDRTM patented altered UDMA molecule.

The results of the present study showed that depth of cure varied between the materials. This can probably also partly be explained by the differences in monomer composition. The differences recorded in moduli and Knoop Hardness between the materials would on the contrary, be more influenced by differences in filler loading and therefore not affected by the storage time and temperature to the same extent.

The decline in moduli and flexural strength for CXU, and particularly for EC and ECB at 57°C might be due to increased hydrolytic processes due to penetration of water – acting as a softener, affecting the mechanical properties by decreasing the cohesion (61). Temperature can increase the distances between polymer chains, if not very densely cross-linked, by decreasing the secondary forces interacting between the chains. The result will be an increase in chain movements depending on degree of cross linkage as well as increased diffusion of water acting as a softening agent, affecting the mechanical properties (e.g. increase of the viscoelastic properties) (61).

Of the composite samples investigated by micro-CT, SDRTM displayed a more homogenous structure and the lowest degree of voids compared to the other materials evaluated. That factor can be a result of the monomer composition as well as lower filler content. However, if the structure and low amount of voids will affect SDR's flexural strength as well as the

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materials ability to withstand early aging effects can only be hypothesized on, and further research on the subject seems needed. Still, a few large voids could be seen inside the SDRTM (possibly due to air in the compule) same that might cause early failure if situated in the area tested using 3-point bending test. Several of the other samples also displayed uneven distribution of voids. This might imply that the 3-point bending test normally used for evaluation of flexural strength might have a lower opportunity to detect potentials for early failure due to voids, than e.g. the 4-point bending test. The reason is that the latter would have the opportunity to subject a larger test area to stress in comparison to the one subjected in the 3-point bending test. Qualitative assessment of samples studied showed a tendency for accumulation of voids in the interface between composite layers, the fact that this visible interface exists may in itself also be a defect. Samples in this experiment were made under optimal circumstances. If this interface will be enhanced in a clinical situation can only be speculated on at present. Regardless, it may lead to an argument for placing the whole filling in one layer, avoiding the creation of several interfaces in the restoration – minimizing possible sources for early failure.

Conclusions and significance

Within the limitations of the present study the following conclusions were drawn.

The null hypothesis was rejected concerning the low viscosity bulk fill material tested. The material SDRTM, showed lower moduli and hardness in comparison to high viscosity bulk fill and universal composites tested. On the contrary, SDRTM showed a higher flexural strength and depth of cure. The flexural strength significantly increased with increased storage temperature simulating aging compared to the other materials tested. The differences in moduli and hardness could be explained by differences in filler loading while the increase strength and high depth of cure could be explained by the monomer composition in SDRTM. The result indicates that the term bulk fill seems not relevant since that it is not a discriminating factor for assessment of different properties. More relevant information concerning filler loading and monomer content (i.e. type of monomers used) should be given and assessed.

Depending on monomer content, aging seemed to affect the mechanical properties of the tested materials – differently. Therefore, values given by manufacturers may not be as descriptive as desired when it comes to comparing materials in a longer time perspective, as materials may react differently to aging.

The tendency for voids to accumulate between composite layers might be an indication for placing the whole filling in one layer (e.g. by using a bulk fill system), as this might affect mechanical strength negatively. however, further studies of voids in composites might be an interesting topic in relation to improving the mechanical properties of these materials.

References:

- 1. Stephen CB. Beginnings of the dental composite revolution. JADA. 2013;144:880-4.
- 2. Figueiredo Reis A, Giannini M, Ambrosano GM, et al. The effects of filling techniques and a low-viscosity composite liner on bond strength to class II cavities. Journal of dentistry. 2003;31:59-66.
- 3. Felix SA, Gonzalez-Lopez S, Mauricio PD, Et al. Effects of filling techniques on the regional bond strength to lateral walls in Class I cavities. Oper Dent. 2007;32:602-9.
- 4. Park J, Chang J., Ferracane J, Et al. How should composite be layered to reduce shrinkage stress: incremental or bulk filling? Dental materials: official publication of the Academy of Dental Materials. 2008;24:1501-5.
- 5. Sunbula HA, Silikasa N, Watts DC. Polymerization shrinkage kinetics and shrinkage-stress in dental resin-composites. Dental materials : official publication of the Academy of Dental Materials. 2016;32:998-1006.
- 6. Feilzer AJ, De Gee AJ, Davidson CL. Setting stress in composite resin in relation to configuration of the restoration. J Dent Res. 1987;66:1636-9.
- 7. Zhengzhi Wang, Martin Y.M. Chiang. Correlation between polymerization shrinkage stress and C-factor depends upon cavity compliance. Dental materials: official publication of the Academy of Dental Materials. 2016;32:343-52.
- 8. Seung-Hoon Han, Alireza Sadr, Junji Tagami, Et al. Internal adaptation of resin composites at two configurations: Influence of polymerization shrinkage and stress. Journal of Dental Materials. 2016;32:1085-94.
- 9. Braga RR, Ballester RY, Ferracane JL. Factors involved in the development of polymerization shrinkage stress in resin-composites: a systematic review. J Dent Mater. 2005;21:962-70.
- 10. Davidson CL, Feilzer AJ DGA, Davidson CL. Polymerization shrinkage and polymerization shrinkage stress in polymer-based restoratives. Journal of dentistry. 1997;25:435-40.
- 11. Do T, Church B, Veríssimo C, Et al. Cuspal Flexure, Depth-of-cure, and Bond Integrity of Bulk-fill Composites. American Academy of Pediatric Dentistry. 2014;36:468-73.
- 12. Ivoclar Vivadent. Tetric EvoCeram Bulk fill Scientific Compendium. 2012.
- 13. Scientific compendium SDR.
- 14. Kerr Corporation. Sonic Fill 2 IFU. 2016.
- 15. Kwon Y, Ferracane J, Lee IB. Effect of layering methods, composite type, and flowable liner on the polymerization shrinkage stress of light cured composites. Dental materials : official publication of the Academy of Dental Materials. 2012;28:801-9.
- 16. Tetric EvoCeram Bulk Fill, Intructions for Use.
- 17. SDR, Intructions for Use.
- 18. Gotfredsen P, Hörsted P, Kragstrup J. Porosity of restorative resins. European journal of Oral Sciences. 1983;91:312-5.
- 19. Dijken JWV van, Ruyter IE, Holland RI. Porosity in posterior composite resins. European Journal of Oral Science. 1986;94:471-8.
- 20. Nazari A, Sadr A, Saghiri MA, Et al. Non-destructive characterization of voids in six flowable composites using swept-source optical choherence tomography. Journal of Dental Materials. 2013;29:278-86.
- 21. Balthazard R, Jager S, Dahoun A, Et al. High-resolution tomography study of the porosity of three restorative resin composites. Clinical oral investigations. 2014;18:1613-8.
- 22. Darwell B. Materials Science for Dentistry. 9 ed: Woodhead Publishing; 2009.
- 23. Kim RJ, Kim YJ, Choi NS, Et al. Polymerization shrinkage, modulus, and shrinkage stress related to tooth-restoration interfacial debonding in bulk-fill composites. Journal of dentistry. 2015;43:430-9.
- 24. Leprince JG, Palin WM, Vanacker J, Et al. Physico-mechanical characteristics of commercially available bulk-fill composites. Journal of dentistry. 2014;42:993-1000.
- 25. Waltimo A, Könönen M. A novel bite force recorder and maximal isometric bite force values for healthy young adults. Scand J Dent Res. 1993;101:171-5.

- 26. Ernst CP, Canbek K, Aksogan K, Et al. Two-year clinical performance of a packable posterior composite with and without a flowable composite liner. Clinical oral investigations. 2003;7:129-34.
- 27. Dijken JWV van, Pallesen U. Posterior bulk-filled resin composite restorations: A 5-year randomized controlled clinical study. Journal of dentistry. 2016;51:29-35.
- 28. Heitze SD, Zimmerli B. Relevance of In-vitro Tests of Adhesive and Composite Dental Materials pt1. Schweiz Monatsschr Zahnmed. 2011;121:810-6.
- 29. Heitze SD, Zimmerli B. Relevance of In-vitro Tests of Adhesive and Composite Dental Materials pt2. Schweiz Monatsschr Zahnmed 2011;121:916-23.
- 30. Heitze SD, Zimmerli B. Relevance of In-vitro Tests of Adhesive and Composite Dental Materials pt3. schweiz Monatsschr Zahnmed. 2011;121:1024-32.
- 31. Ernst CP, Martin M, Stuff S, Et al. Clinical performance of pacable resin composite for posterior teeth after 3 years. Clinical oral investigations. 2001;5:148-55.
- 32. Krämer N, Garcia-Godoy F, Latta M, al. E. Evaluation of resin composite materials. Part II: in vivo investigations. Am J Dent. 2005;18:75-81.
- 33. Gallo JR, Burgess JO, Ripps AH, Et al. Two-year clinical evaluation of a posterior resin composite using a fourth- and fifth-generation bonding agent. Operative Dentistry. 2005;30:244-50.
- 34. Burke F J, Crisp R J, Balkenhol M, et al. Two-year evaluation of restorations of a packable composite placed in UK general dental practices. Br Dent J. 2005;199:293-6.
- 35. ISO 4049:2009. 2009.
- 36. Cornelio RB, Wikant A, Mjøsund H, Et al. The influence of bis-EMA vs bis GMA on the degree of conversion and water susceptibility of experimental composite materials. Acta Odontologica Scandinavica. 2013;72:440-7.
- 37. Gajewski VES, Pfeifer CS, Fróes-Salgado NRG, Et al. Monomers Used in Resin Composites: Degree of Conversion, Mechanical Properties and Water Sorption/Solubility. Braz Dent J. 2012;23:508-14.
- 38. Ferracane. Hygroscopic and hydrolytic effects in dental polymer networks. Dental materials : official publication of the Academy of Dental Materials. 2006;22(3):211-22.
- 39. Söderholm Z, Ragan, Fischlschweiger, Bergman Hydrolytic degradation of dental composites. Journal of Dental Research. 1984;63:1248.
- 40. Ilie N, Hickel R. Mechanical investigations on silorane and methacrylate-based composites. Dental materials: official publication of the Academy of Dental Materials. 2009;25:810-9.
- 41. Rassmusson CG, Lundin SA. Class II restorations in six different posterior composite resins: five-year results. Swe Dent J. 1995;19:173-82.
- 42. Mandikos MN, McGivney GP, Davis E, Et al. A comparison of the wear resistance and hardness of indirect compoiste resins
 - J Prosthet Dent. 2001;85:386-95.
- 43. Li J, Li H, Fok AS, Et al. Multiple correlations of material parametres of light-cured dental composites. Dental materials : official publication of the Academy of Dental Materials. 2009;25:829-36.
- 44. Price RB, Labrie D, Rueggeberg FA, Et al. Correlation between the beam profile from a curing light and the microhardness of four resins. Dental materials: official publication of the Academy of Dental Materials. 2014;30:1345-57.
- 45. AlShaafi MM, Harlow JE, Price HL, et al. Emission Characteristics and Effect of Battery Drain in "Budget" Curing Lights. Operative Dentistry. 2016;41:397-408.
- 46. Harlow JE, Sullivan B, Shortall C, Et al. Characterizing the output settings of dental curing lights. Journal of dentistry. 2016;44:20-6.
- 47. Price RB, Ferracane JL, Shortall AC. Light-Curing Units: A Review of What We Need to Know. Journal of Dental Research. 2015;94:1179-86.
- 48. Terselius B. Polymerers kemiska och fysikaliska egenskaper. Stockholm (in swedish): KTH; 1995
- 49. Örtengren U., Elgh U., Spasenoska V., Et al. Water Sorption and Flexural Properties of a Composite Resin Cement. The International journal of Prosthodontics. 2000;13:141-7.

- 50. Tetric EvoCeram, Instructions for Use.
- 51. Ceram X Universal, Instructions for Use.
- 52. Price RB, FA R, Harlow J, Et al. Effect of mold type, diameter, and uncured composite removal method on depth of cure. Clinical oral investigations. 2016;20:1699-707.
- 53. Troms County Public Dental services. Dental composite purchase statistics. 2015.
- 54. Chung KH, Greener EH. Correlation between degree of conversion, filler concentration and mechanical properties of posterior composite resins. Journal for Oral Rehabilitation. 1990;17:487-95.
- 55. Masouras K, Silikas N, Watts DC. Correlation of filler content and elastic properties of resincomposites. Dental materials: official publication of the Academy of Dental Materials. 2008:24:932-9.
- 56. Kim KH, Ong JL, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. The Journal of Prostetic Dentistry. 2002;87:642-9.
- 57. Sung-Ae Son, Jeong-Kil Park, Deog-Gyu Seo, et al. How light attenuation and filler content affect the microhardness and polymerization shrinkage and translucency of bulk-fill composites. Clinical oral investigations. 2016;21:559-65.
- 58. Peutzfeldt A, Asmussen E. The effect of postcuring on quantity of remaining double bonds, mechanical properties, and in vitro wear of two resin composites. Journal of dentistry. 2000;28:447-52.
- 59. Sideridou I, Tserki V, Papanastasiou G. Effect of chemical structure on degree of conversion in light-cured dimethacrylate-based dental resins. Biomaterials 2002;23:1819-29.
- 60. Peutzfeldt A. Resin composites in dentistry: the monomer systems. European Journal of Oral Science. 1997;105:97-116.
- 61. Gedde U. Polymer Physics. 1 ed: Springer Netherlands; 1999.