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Investigations on a methacrylate-based flowable composite based on the SDR^{TM} technology

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ABSTRACT

Objective. Monomer development for a reduced shrinkage of composite materials still challenges the modern research. The purpose of this study was to analyse the shrinkage behavior of an innovative composite material for dental restorations based on a resin system that is claimed to control polymerization kinetics having incorporated a photoactive group within the resin.

Methods. Shrinkage stress development within the first 300 s after photoinitiation, gel point as well as micro-mechanical properties (Vickers hardness HV, modulus of elasticity E, creep Cr and elastic–plastic indentation work W_e/W_{tot}) were evaluated ($n = 10$). The experimental flowable resin-based composite (RBC) was measured in comparison to regular methacrylate-based micro- (Esthet X Flow) and nano-hybrid flowable RBCs (Filtek Supreme Plus Flow). Additionally, the high viscosity counterparts of the two regular flowable methacryate-based composites (Esthet X Plus and Filtek Supreme Plus) as well as a low shrinkage silorane-based micro-hybrid composite (Filtek Silorane) were considered. The curing time was 20 s (LED unit Freelight2, 3M-ESPE, 1226 mW/cm^2).

Results. The experimental material achieved the significantly lowest contraction stress (1.1 ± .01 MPa) followed by the silorane-based composite (3.6 ± .03 MPa), whereas the highest stress values were induced in the regular methacrylate-based flowable composites EsthetX Flow (5.3 ± .3 MPa) and Filtek Supreme Flow (6.5 ± .3 MPa). In view of gel point, the best values were obtained for the experimental flowable composite (3.1 ± .1s) and Filtek Silorane (3.2 ± .3 s), which did not differ significant from each others, whereas EsthetX Plus and Filtek Supreme Plus did also not differ significantly, inducing the shortest gel point. The experimental flowable material achieved also the lowest shrinkage-rate (maximum at 0.1 MPa/s). For all analysed materials, no significant difference in the micro-mechanical properties between top and bottom were found when measured on 2 mm thick increments 24 h after polymerization. The categories of flowable materials performed in the measured micro-mechanical properties significantly inferior when compared to the hybrid-composites, showing lower HV and E and predominantly higher creep and plastic deformation. Within the flowable RBCs, the experimental material achieved the lowest Vickers hardness, the highest modulus of elasticity, the highest creep and showed the significantly lowest elastic deformation.

Significance. The experimental flowable composite revealed the lowest shrinkage stress and shrinkage-rate values in comparison to regular methacrylate composites but intermediate

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micro-mechanical properties. Being at the same time more rigid (higher modulus of elasticity) and more plastic (low W_e/W_{tot} and high creep values) as the regular flowable materials, its effect on interfacial stress build-up cannot be easily predicted.

1. Introduction

Several negative effects in resin-based composite (RBCs) restorations, like marginal discrepancies [1,2], marginal staining, white lines around the restoration, cusps fractures [3], microleakage [4], debonding [3], recurrent caries, post-operative sensitivity or pain, are frequently connected to polymerization shrinkage stress. Even if these assertions are nowadays broadly accepted, only little clinical evidence exists to support a clear relationship between this negative outcomes and polymerization shrinkage stress [5]. Nevertheless, new materials having reduced internal stress as a result of low polymerization shrinkage are predicted to dominate the market in the future, even before a complete understanding of the clinical effect of shrinkage was achieved [6].

As a consequence, extensive efforts have been invested over years to develop low shrinkage dental restorative materials. Beside changes in filler amount, shape or surface treatment, changes in monomer structure or chemistry and modification of dynamics of the polymerization reaction are the most promising approaches.

The changes in monomer chemistry were at first directed to improve the already clinical successful methacrylate-based systems, by modifying the Bowen monomer (Bis-GMA: 2,2-bis[4-(2-hydroxy-3-methacrylyloxypropoxy)phenyl]propane) to create monomers with lower viscosity [7–9] like hydroxyl free Bis-GMA [10], aliphatic urethane dimethacrylates (UEDMA: 1,6-bis[methacrylyloxy-2-ethoxycarbonylamino]-2,4,4-trimethylhexane) [11], partially aromatic urethane dimethacrylate [12] or highly branched methacrylates [13]. But also ring-opening systems for non- or minimally shrinking dental composites like spiro-orthocarbonates as additives to dimethacrylates [14] and epoxy-base resins like the siloranes [15] as well as ormocers (organically modified ceramics) [16,17] were introduced on the market for the same purpose.

Recent trials to modify the dynamics of the polymerization reaction by incorporating a photoactive group in a urethane-based methacrylate resin showed a 60–70% reduction in shrinkage stress in the unfilled resin when compared to conventional methacrylate-based resins [18]. The activated resin demonstrated a relatively slow radical polymerization rate, suggesting that the photoinitiator incorporated into the resin is affecting the radical polymerization process. This lower curing stress was shown to be retained also in filled compositions, particularly in cases of low filler-loading [18]. These resins are patent-registered [19] as being based on the SDR^{TM} technology (=stress decreasing resin).

To additionally minimize shrinkage stress at the interface tooth–composite for a given composite material, several practical steps are proposed, most of them being related to the chosen cavity configuration (C-factor) [20], the method of cavity reconstruction [21], the selection of the curing method [22,23], or the introduction of stress absorbing intermediate layers [24]. Positive effects are reported by using flowable composites as stress absorbing intermediate layers. Used in Class II restorations together with packable (Filtek P60) or nanofilled composites (Filtek Supreme XT Universal Restorative), less microleakage was measured [25]. As additionally positive effects, flowable liners are considered to decrease sensitivity and to wet the cavity better than restorative composites due to their flowability, to have thus a better adaptation to the dentinal surface, with fewer voids at the interface of the restoration and tooth structure, when compared with bonding agent and resin composites alone [26]. Reducing shrinkage during polymerization in flowable composite liners will be an additional positive effect.

It was claimed that resin systems based on the SDR^{TM} technology with a polymerization modulator being chemically embedded in the polymerizable resin backbone controlling thus polymerization kinetics will induce lower polymerization shrinkage in the flowable composite based on it [18].

The aim of this study was therefore to analyse the shrinkage behavior and the micro-mechanical properties of an experimental flowable composite based on this technology, developed to serve as a flowable liner, in comparison to regular methacryate-based micro- and nano-hybrid flowable composites. Additionally, the high viscosity counterparts of the two regular flowable methacryate-based composites as well as a low shrinkage silorane-based micro-hybrid composite were considered.

The null hypotheses tested were:

1. The shrinkage behavior of the experimental flowable material during polymerization do not differ from the regular flowable materials.
2. A reduced final shrinkage stress will not lower the micro-mechanical properties.

2. Materials and methods

An experimental flowable composite material (Exp-Flow, Dentsply) was analysed in comparison to four commercially available methacrylate-based micro- and nano-hybrid composites, two of them being flowable composites, as well as with an silorane-based micro-hybrid composite (Table 1). The material's behavior was assessed by evaluating the shrinkage stress development during curing, shrinkage stress values yielded thereof and gel point as well as the micro-mechanical properties – Vickers hardness, modulus of elasticity, creep and elastic–plastic deformation – after polymerizing the materials for 20 s with a LED-curing unit (Freelight 2, 3M-ESPE, Germany). The spectral distributions and irradiance of the tested cur-

Table 1 – Materials, manufactures, batch numbers and composition.

Composite	Manufacturer	Type	Resin matrix	Filler
EsthetX plus LOT:0705114	Dentsply	Micro-hybrid	Bis-GMA adduct Bis-EMA adduct TEGDMA	Ba–F–Al–B–Si–glass nanofiller silica 77 wt%, 60 vol%
EsthetX Flow LOT:070215	Dentsply	Flowable Micro-hybrid	Bis-GMA adduct Bis-EMA adduct TEGDMA	Ba–F–Al–B–Si–glass nanofiller silica 61 wt%, 53 vol%
Exp-Flow LOT:QD2-161	Dentsply	Flowable	Modified UDMA EBPADMA TEGDMA	Ba–Al–F–B–Si–glass, St–Al–F–Si–glass 68 wt%, 44 vol%
Filtek Supreme Plus LOT:A2B 20070809	3M-ESPE	Nano	BIS-GMA BIS-EMA UDMA TEGDMA	Silica nanofiller, zirconia/silica nanocluster, 78.5 wt%, 59.5 vol%
Filtek Supreme Plus Flow LOT:A2B20070716	3M-ESPE	Flowable Nano	BIS-GMA BIS-EMA TEGDMA	Silica nanofiller, zirconia nanofiller zirconia/silica nanocluster, 65 wt%, 55 vol%
Filtek Silorane LOT:20070827	3M-ESPE	Micro-hybrid	3,4-Epoxycyclohexylethylcyclo-polymethylsiloxane Bis-3,4-epoxycyclohexylethyl-phenylmethylsilane	Silanized quartz yttrium fluoride 76 wt%, 55 vol%

BIS-GMA: Bisphenol A dimethacrylate; BIS-EMA: Bisphenol A polyetheylene glycol diether dimethacrylate; UDMA: urethane dimethacrylate; TEGDMA: Triethyleneglycol dimethacrylate; EBPADMA: ethoxylated Bisphenol A dimethacrylate.

ing unit were determined by means of a calibrated fibre optic spectrally resolving radiometer equipped with an integrating sphere (S2000, Ocean Optics, USA). The total irradiance was obtained by integrating the irradiance as a function of the wavelength over the entire wavelength range, divided by the effective area of the curing unit tip. The diameter of the tip was measured with a digital micrometer, whereby as effective area was defined the area of the tip without cladding.

2.1. *Shrinkage stress measurement device*

Shrinkage stress and time until gelation (gel point) of the tested materials were investigated with a stress–strain analyzer (SSA T80, Engineering Consultancy Peter Dullin Jr, Munich) [27]. With due compensation for machine compliance, this device measured the contraction stress generated by polymerizing the restorative material with the light exposure mentioned above ($n = 10$).

2.1.1. *Experimental setup*

Composite specimens were placed between two aluminum attachments with a distance of 4 mm and a height of 2 mm. One attachment was connected to a load sensor, and the other to a piezo actuator. A PTFE coated plastic tray was mounted on the testing machine, fitting exactly between the two attachments. The two opposing attachments together with the plastic mould simulate a 4 mm × 4 mm × 2 mm cavity with a C-factor of 0.3 corresponding clinically to a premolar restoration with an increment of 2 mm. The functional surfaces of the two attachments were cleaned with Rocatec Pre (ESPE), coated with Rocate Plus (ESPE), followed by applying a silane coupling agent (ESPE-Sil, ESPE). Experimental composite was then applied in one 2 mm increment.

2.1.2. *Measurement procedure*

Contraction force generated due to composite polymerization was continuously measured and recorded for 300 s after photoinitiation. Each experiment was conducted at room temperature and repeated ten times for each material. Contraction stress was calculated as contraction force divided by the contact area (=8 mm^2). Maximum contraction stress during recording time and time needed to exceed a force threshold of 0.5 N (arbitrarily defined as the time until gelation or gel point) were compared using one-way ANOVA and Tukey's HSD post hoc test ($\alpha = 0.05$) (SPSS 17.0).

2.2. *Micro-mechanical properties*

To analyse the mechanical properties of the materials, measurements were made with a micro-hardness indenter (Fischerscope H100C, Fischer, Sindelfingen, Germany) according to DIN 50359-1:1997-10 [28]. Prior to testing, the samples were polished with a diamond suspension (mean grain size: 1 μm). Measurements were done at the top and bottom of the materials after storing the samples made for the shrinkage-stress measurements (4 mm × 4 mm × 2 mm) for 24 h in distilled water ($n = 10$, with 5 measurement per sample).

The test procedure was carried out force controlled; the test load increased and decreased with constant speed between 0.4 mN and 500 mN. The load and the penetration depth of the indenter were continuously measured during the load–unload-hysteresis. The Universal hardness is defined as the test force divided by the apparent area of the indentation under the applied test force. From a multiplicity of measurements, a conversion factor between Universal hardness and Vickers hardness was calculated and implemented in the software, so that the measurement results were indicated in the more familiar *Vickers hardness* units. The *indentation modulus* was calculated from the slope of the tangent of indentation depth-curve at maximum force and is comparable with the modulus of elasticity of the material. By measuring the change in indentation depth with constant test force, a relative change in the indentation depth can be calculated. This is a value for the *creep* of the materials. The mechanical work W_{tot} indicated

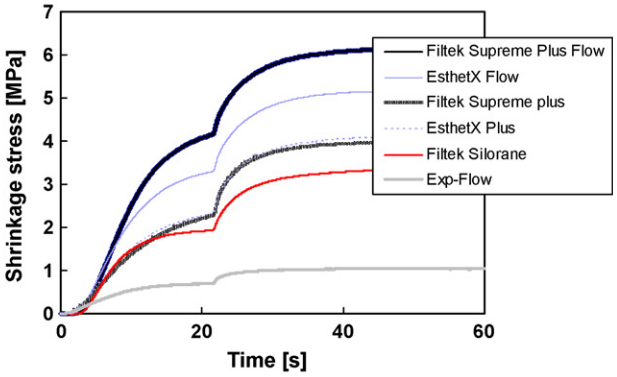

Fig. 1 – Comparison of the shrinkage stress development (averaged curves, $n = 10$) as a function of time for the experimental composite with a controlled polymerization kinetic, the silorane-based micro-hybrid composite and the 4 regular methacrylate-based composites.

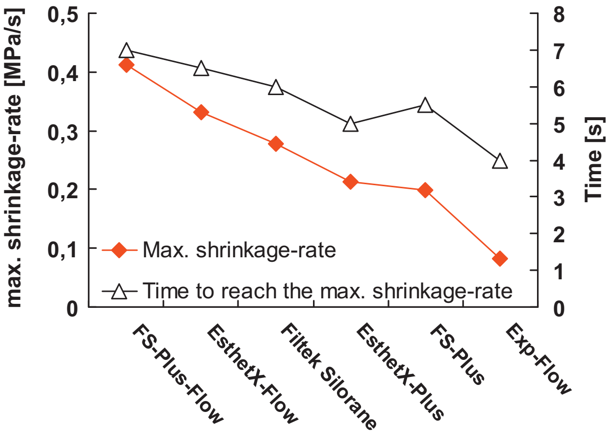

Fig. 3 – Maximal shrinkage stress-rate and time needed to achieve the maximal shrinkage stress-rate in the 6 measured composites ($n = 10$).

during the indentation procedure is only partly consumed as plastic deformation work W_{plast}. During the removal of the test force the remaining part is set free as work of the elastic reverse deformation W_{elast}. According to the definition of the mechanical work as $W = \int F\,dh$, $W_{tot} = W_{elast} + W_{plast}$. The parameter W_e/W_{tot} [%] representing the percentage of elastic work reported to the total mechanical is indicated in the results data.

2.3. Statistical analysis

Results were statistically compared using one-way ANOVA and Tukey HSD post hoc-test ($\alpha = 0.05$) as well as a Pearson correlation analysis. A multivariate analysis (general linear model with partial eta-squared statistics) tested the influence of the parameters *filler volume* and *weight* as well as *top-bottom* indicating the measurement location on the considered properties (SPSS 18.0).

3. Results

The power of the used curing unit was determined to be at 1226 mW/cm^2. Since the curing time was 20 s, a energy density of 14.5 J/cm^2 was applied on each sample. The shrinkage stress development as a function of time represented as averaged curves ($n = 10$) for the experimental composite, the silorane-based micro-hybrid composite and the four regular methacrylate-based materials, during the first 60 s after photo-initiation, are presented in Fig. 1. All curves were generally S-shaped, with initial near-linear contraction as soon as rigid contraction started. The sudden drop in temperature after light exposure produced thermal contraction, which added to the polymerization contraction, thereby causing a sharp upward bend of the shrinkage stress curves. Derived from these curves, the shrinkage stress rate development in time within the 20 s of composite irradiation (Fig. 2) presented a maximum, which appears as being material dependent. The two regular methacrylate-based flowable materials – Filtek Supreme Flow and Esthet X Flow – showed the highest shrinkage-rates (maximum at 0.4 and 0.3 MPa/s), whereas for the experimental material, the lowest shrinkage-rate was measured (maximum at 0.1 MPa/s), having the non-flowable composites in-between this range. Among the maximal shrinkage stress-rate and the time needed to achieve the maximal shrinkage stress-rate (Fig. 3) a very good correlation was found (Pearson: 0.97).

One-way ANOVA exhibited significant differences ($p < 0.05$) among the materials for shrinkage-stress and gel point (Fig. 4). The significantly lowest contraction stress was achieved by the experimental material (1.1 ± .01 MPa), followed by the silorane-based composite (3.6 ± .03 MPa). The highest stress values were induced in the regular methacrylate-based

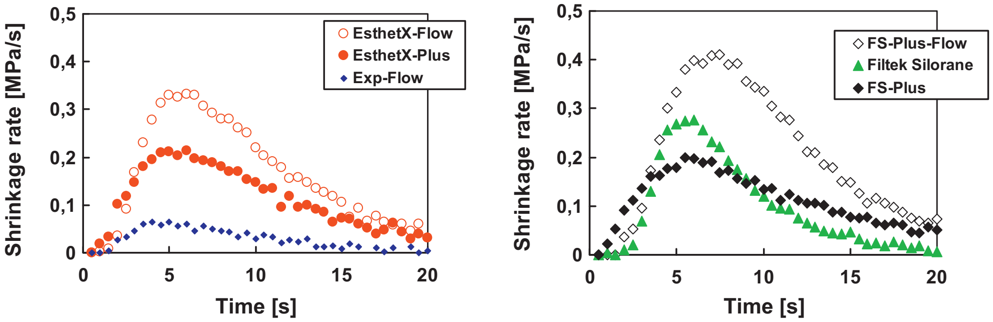

Fig. 2 – Shrinkage stress rate development in time within the 20 s of composite irradiation, mean curves ($n = 10$).

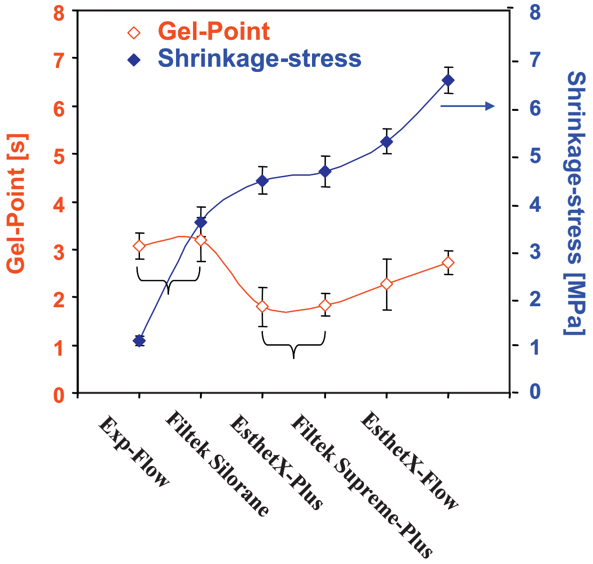

Fig. 4 – Gel point and shrinkage-stress 300 s after starting the photo-initiation ($n = 10$). Data are presented in ascending order of shrinkage-stress. One-way ANOVA analysis revealed a statistic significant increase in shrinkage-stress proceeding from the Exp-Flow to Filtek Supreme-Plus Flow. The materials grouped in bracket do not differ significantly from each other.

flowable composites EsthetX Flow (5.3 ± .3 MPa) and Filtek Supreme Flow (6.5 ± .3 MPa). In view of gel point, the highest values were obtained for the experimental flowable composite (3.1 ± .1s) and Filtek Silorane (3.2 ± .3 s), which do not differ significant from each others, whereas EsthetX Plus and Filtek Supreme Plus do also not differ significantly, inducing the shortest gel point. The correlation between gel point and shrinkage stress was weak (Pearson: −.4).

The micro-mechanical properties – Vickers hardness HV, modulus of elasticity E, creep Cr and elastic–plastic deformation W_e/W_{tot} – measured on the top and bottom of the samples stored for 24 h in dest. water are presented in Fig. 5 and Table 2. For all analysed materials, no significant difference in micro-mechanical properties between top and bottom of the 2 mm increments were found. The categories of flowable materials performed in all measured micro-mechanical properties significantly inferior when compared to the hybrid-composites. Within the flowables, the experimental material achieved the lowest Vickers hardness, the highest modulus of elasticity, the highest creep and showed the significant lowest elastic deformation.

The correlation between shrinkage stress and Vickers Hardness and, respectively, modulus of elasticity was low (Pearson: 0.2 and 0.1), whereas the parameter creep and elastic–plastic deformation correlated stronger with the shrinkage stress (0.6 and 0.8).

The influence of the parameters "filler volume", "filler weight" and "top-bottom measuring position" were analysed in an ANOVA multivariate test. The micro-mechanical properties – modulus of elasticity E, Vickers hardness HV, creep Cr and elastic–plastic deformation – as well as shrinkage stress and gel point were selected as depended variables. The significance values of the first two main effects were less than 0.05, indicating that they contribute all to the model. The parameter "top-bottom" was not a significant factor of influence. The results of the ANOVA multivariate test are summarized in Table 3, showing a strong influence of all parameters on the measured properties. The filler weight influenced stronger the shrinkage-stress and gel point then the filler volume, whereas the influence on the micro-mechanical properties of this two parameters was in the same range. From the measured micro-mechanical properties, the Vickers hardness and the elastic–plastic deformation were stronger influenced by the filler then the modulus of elasticity and creep.

4. Discussion

The study analysed the shrinkage behavior and the micro-mechanical properties of an experimental flowable RBC, intended to be used as a liner in Class I and II restorations. The material was recently lounged on the American market under the name SureFil® SDR^{TM} flow, respectively, as SDR^{TM} Posterior Bulk Fill Flowable Base on the European market. Two regular micro- and nano-hybrid flowable methacryate-based composites, their high viscosity counterparts as well as a low shrinkage silorane-based micro-hybrid composite were considered as references.

The information provided by the manufacturer regarding the chemical composition of the experimental flowable indicates that the organic matrix comprises a patent-registered urethane dimethacrylate with incorporated photoactive groups able to control polymerization kinetics. The other contained polymerizable resins are similar to those also used in EsthetX.

One mechanism to reduce shrinkage stress is to delay the gel point. From the chemically point of view, the gel point represents the increase of viscosity by network formation. In the pre-gel phase, the formed polymer chains are very flexible so that material can flow from the free surface of the cavity. The viscosity of the developing polymer is still low; consequently shrinkage stress can be compensated by plastic flow occurring during the pre-gelation phase such that internal stresses within the material undergo stress relaxation [29]. The time, at which the material is not any more able to compensate the polymerization contraction (time until gelation), is therefore determining for the final tensions in the material. About the reaction mechanism in the experimental material no information are available so far. Nevertheless, when compared to the measured methacrylate-based materials, the experimental composite behaves differently, showing a delayed point of gelation which is statistically comparable only with the silorane-based composite. A further mechanism to compensate stress in composites is a slow polymerization rate of the resin, thereby increasing its flow capacity and being associated with lower stress build-up and better interfacial integrity scores [30,31]. This seems to be the case of the experimental flowable composite which achieved the lowest shrinkage-rate with a maximum being 3–4-fold lower as measured in the other two flowable materials. Another particularity of the experimental flowable material is that this

Table 2 – Micro-mechanical properties – Vickers hardness HV [N/mm^2], modulus of elasticity E [GPa], creep Cr [%] and elastic–plastic deformation W_e/W_{tot} [%] – measured on top and bottom of the samples stored for 24 h in dest. water are detailed in mean values and standard deviations (in parentheses). Superscript letters indicate statistically homogeneous subgroups (Tukey's HSD test, $\alpha = 0.05$).

Material	HV	E	Cr	W_e/W_{tot}
Exp-Flow				
Top	36.3^{a} (3.6)	9.2^{c} (1.0)	5.4^{e} (0.3)	33.6^{a} (1.8)
Bottom	40.1^{ab} (3.7)	9.3^{c} (1.0)	4.9^{de} (0.3)	36.1^{ab} (2.1)
EsthetX-Flow				
Top	43.8^{bc} (3.8)	7.7^{ab} (0.7)	4.0^{c} (0.2)	45.5^{c} (1.6)
Bottom	43.9^{c} (6.7)	7.6^{ab} (1.2)	4.0^{c} (0.3)	45.6^{c} (2.1)
Filtek Supreme Flow				
Top	46.7^{c} (8.3)	7.3^{ab} (1.0)	3.7^{bc} (0.3)	49.4^{de} (2.4)
Bottom	49.3^{cd} (4.8)	8.3^{b} (1.0)	3.9^{c} (0.2)	47.3^{de} (2.5)
EsthetX-Plus				
Top	67.1^{ef} (7.5)	10.7^{de} (1.5)	3.9^{c} (0.3)	47.7^{de} (2.5)
Bottom	62.5^{e} (6.1)	10.5^{d} (1.2)	4.0^{c} (0.2)	46.3^{cd} (2.1)
Filtek Silorane				
Top	71.5^{g} (8.1)	12.0^{fg} (1.3)	3.5^{ab} (0.7)	47.1^{de} (1.8)
Bottom	67.9^{fg} (6.1)	11.4^{ef} (1.0)	3.3^{a} (0.1)	46.4^{cd} (2.2)
Filtek Supreme plus				
Top	83.9^{h} (5.3)	12.5^{g} (1.3)	3.6^{b} (0.1)	48.3^{f} (2.0)
Bottom	85.4^{h} (4.3)	13.2^{gh} (1.2)	3.5^{ab} (0.1)	47.7^{ef} (2.4)

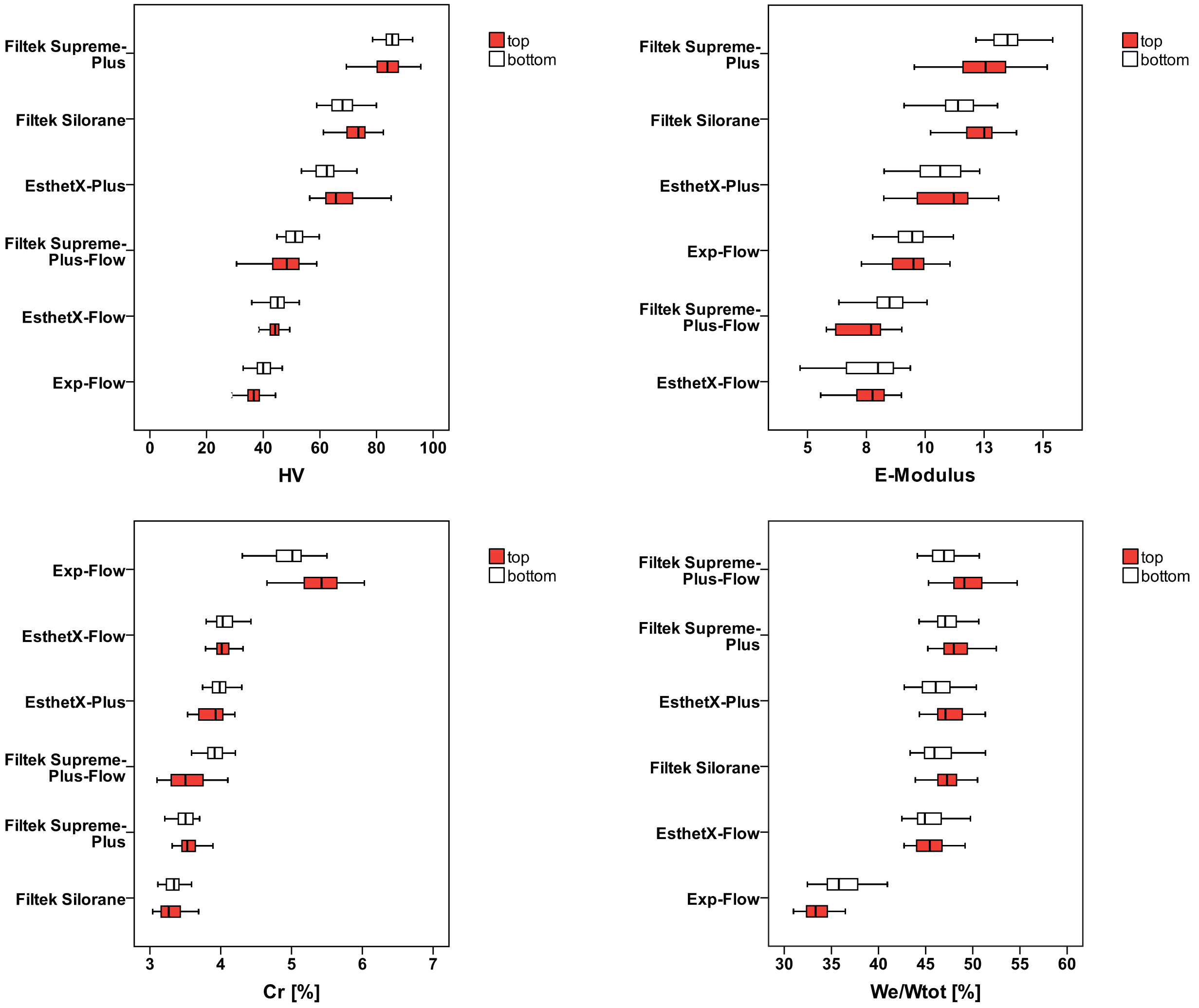

Fig. 5 – Micro-mechanical properties – Vickers hardness HV, modulus of elasticity E, creep Cr and elastic–plastic deformation W_e/W_{tot} – measured on top and bottom of the samples stored for 24 h in dest. water.

Table 3 – Influence of fillers – volume and weight – on the micro-mechanical properties – modulus of elasticity *E*, Vickers hardness HV, creep Cr and elastic–plastic deformation W_e/W_{tot} – as well as shrinkage stress and gel point. The higher the partial eta-squared values, the higher is the influence of the selected variables on the measured properties.

	E	HV	Cr	W_e/W_{tot}	Shrinkage Stress	Gel point
Filler volume	0.58	0.81	0.59	0.83	0.72	0.67
Filler weight	0.68	0.86	0.60	0.84	0.98	0.71

maximum shrinkage-rate, although the lowest measured, was achieved in the shortest time. Thus, the first study hypothesis that the shrinkage behavior of the experimental composite does not differ from that of the measured commercial flowable composite was rejected.

In contrast to the methacrylate-based composites, in the silorane-based system, the mechanism of compensating stress is reached by opening a ring oxirane element during polymerization [15]. The distance molecules have to compensate in order to react is thus decreased and the time when stress begins to be accumulated in the composite delayed. Other than the predominant radical polymerization initiation in the measured methacrylate-based composite, the silorane composite is based on a cationic initiation of polymerization, which is insensitive to oxygen. This should overcome inter alia the disadvantage of the oxygen inhibition layer occurred in methacrylate based composites due to the neutralization of polymerization initialising radicals [15]. Compared to the commercial methacrylate-based composite, the shrinkage-stress induced in the silorane composite was significantly lower and the time of gelation significantly longer.

The two tested flowable and non-flowable composite systems – Filtek Supreme Plus vs. Filtek Supreme Plus Flow as nano-composites and EsthetX plus vs. EsthetX Flow as micro-hybrid composites – offer the possibility to directly compare the effect of the filler amount on the shrinkage stress and gel point, since the chemical composition of the matrix in each system is similar. In both material categories the flowables showed a higher shrinkage stress and a longer gel point, meaning a delayed starting of gelation when compared with their higher filled counterparts. Generally, increasing the volume of the inert material present in a composite, be it inorganic or organic filler, may reduce the overall shrinkage of composites, due to less monomer available for the polymerization reaction [32]. But high filler loading also results in a high degree of stiffness, which can lead to high shrinkage stress; hence, increases in volume fraction of filler do not invariably produce a substantial reduction in shrinkage [33]. The last assertion applies when the two regular flowable materials are directly compared, showing a significantly increase in shrinkage stress in the higher filer loaded Filtek Supreme Plus Flow (55 vol%) when compared to EsthetX Flow (53 vol%).

Another important detail to be considered in the experimental material is the substantial lower filler volume amount (44 vol%) when compared to the other two flowable materials (55 and 53 vol%), whereas the filler weight amount was the highest in the range of the measured flowable RBCs. The explanation thereof must be the chemical composition of the filler, consisting besides Ba–Al–F–B–Si–glass like in Esthet X Flow also in Sr–Al–F–Si–glass, arousing with the Strontium element a higher radiopacity but also a higher filler weight. The resultant effect on the micro-mechanical properties is a higher modulus of elasticity, when compared to the other flowables. The elastic modulus of the shrinking material has also been found to be a factor on which shrinkage stresses depend. In vitro studies have shown that the interfacial stress during the setting shrinkage of a resin composite is positively correlated with the rigidity of the setting material [34].

An inherent characteristic of biomaterials and polymers is their visco-elastic behavior, which implies a time-dependent recovery. When subjected to a constant force (in our study 500 mN for 5 s), the RBCs experience a time-dependent plastic deformation. Lower polymerization time but also plasticizer like water, saliva, alcohol or a temperature above the glass transition point reduces their capacity to resist deformational change under load [35,36]. A reduced amount of the resin matrix due to a high filler content and homogeneous filler distribution has been found to lead to higher creep resistance [37]. As for the measured experimental flowable RBCs, the higher creep values could be an effect of a lower cross-linking of the organic matrix, fact that must be further analysed in future studies.

A dynamic measuring principle like applied in our study recorded simultaneous the load and the corresponding penetration depth of the indenter [28,38] thus the plastic as well as elastic part of the indentation can be separated from the analysis of the load–displacement data. As for W_e/W_{tot} being the elastic part of the indentation work, the lower elastic deformation of the experimental flowable material, respectively, the higher plastic deformation also allude for a more deformable polymer network when compared to the other measured flowable materials. Thus the second research hypothesis asserting that a reduced final shrinkage stress will not lower the micro-mechanical properties is only partial rejected.

5. Conclusions

The experimental flowable material showed a total different behavior when compared to the reference flowable composites. The shrinkage stress after polymerization was lower not only when compared to the flowable materials but also when compared to nano- and micro-hybrid composites or even with the silorane-based material. The experimental flowable material showed the lowest shrinkage-stress-rate but was also more rigid (higher modulus of elasticity) and more plastic (low W_e/W_{tot} and high creep values) as the regular flowable materials, making thus its effect on interfacial stress build-up difficult to predict.

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