Investigation of Mechanical Properties of Modern Dental Composites After Artificial Aging for One Year

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Clinical Relevance

Dental composite restorations may fail due to the deterioration of mechanical properties.

SUMMARY

This *in vitro* study investigated the aging behavior of dental composites with regard to surface roughness (SR), Vickers hardness (VH) and flexural strength (FS), and the study elucidated the impact of artificial aging parameters. One hun-

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dred and sixty-five rectangular specimens were prepared from five composites (Filtek Supreme XT, Filtek Silorane, CeramX, Quixfil, experimental ormocer) and subjected to various artificial aging protocols (storage in distilled water/ ethanol/artificial saliva for 7, 90 and 365 days; thermal cycling, 2 x 3000 cycles 5/55°C). SR, VH and FS were determined at baseline and after each aging treatment. Means and standard deviations were calculated; statistical analysis was performed using three-way ANOVA and the Tukey-Kramer multiple comparison test (α =.05). The results showed a significant influence in the composite and aging duration on mechanical parameters; the aging medium did not have a significant influence on VH and FS, but there was a significant influence on SR. The highest overall VH was found for the experimental ormocer; Filtek Silorane yielded the lowest values. For FS, the significantly highest values were found for Filtek Silorane, and the lowest

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values were found for the experimental ormocer. Prolonged aging periods (90 or 365 days) or thermal cycling led to significant decreases in both VH and FS and significant increases in SR. The findings of the current study indicate that composites differ significantly for SR and its mechanical properties with regard to FS and VH, as well as in aging behavior. Generally, artificial aging leads to a significant decrease in mechanical properties, which underlines the relevance of continuous improvement of dental composites.

INTRODUCTION

Dental composite materials are increasingly important in modern dentistry; these materials consist of a polymerizable resin matrix and filler particles that are chemically linked by silane coupling agents. A large number of different composite materials is available for direct dental restorations, comprising classical hybrid- and nanofilled-composite materials, siloranes, ormocers and compomers. However, differences among these materials with regard to the monomer system, filler composition and matrix-filler coupling chemistry may account for different mechanical performances and may further account for differences in the materials' resistance to chemical and mechanical degradation. It has been reported that damage to composite materials may result from deterioration of the matrix and fillers or is due to mechanical and environmental loads, interfacial debonding, microcracking or filler particle fracture, which may reduce the survival probability of composite restorations in vivo. When comparing the aging behavior of different dental materials, the clinical aging process is most commonly simulated in vitro, using defined artificial aging protocols. Numerous in vitro studies have focused on the mechanical performance of dental composite materials after artificial aging.3-7 Generally, the artificial aging of dental composites accelerates degradation of the material,89 which causes a significant decrease in mechanical properties. However, the comparability of studies dealing with artificial aging of dental composite materials is rather low, as very different aging conditions and durations have been applied and there is only limited evidence as to how far different aging protocols account for differences in the mechanical performance of a specific material. Moreover, only limited evidence is available concerning the aging behavior of modern dental composite materials, such as siloranes and ormocers. With regard to these aspects, this in vitro study: 1) compared the aging behavior of different composite materials with regard to surface roughness, hardness and flexural strength and 2) analyzed the impact of different artificial aging protocols on aging behavior. It was hypothesized that there are significant differences between various composite materials with regard to their mechanical properties and different artificial aging protocols provoke different results for the mechanical properties of various composite materials after artificial aging.

METHODS AND MATERIALS

Materials

Five modern composite materials, differing in resin and filler chemistry and composition, were used for investigation in the current study (Table 1). From each material, 165 rectangular specimens (2 x 2 x 25 mm) were prepared according to the manufacturers' instruc-

Name	Class	Manufacturer	Monomer	Filler	Filler Size	Filler Content (%wt)	
Filtek Supreme Composite, XT nanofilled		3M ESPE, Seefeld, Germany	Bis-GMA, TEGDMA, UDMA, Bis-EMA	Nanosilica fillers Zirconia/silica nanoclusters	Ø 20 nm 0.6-1.4 µm (5-20 nm) (nano particle size)	78.5	
Filtek Silorane	Composite, 3M ESPE, silorane-based Seefeld, Germany		Silorane Quartz fillers (3,4- epoxycyclohexylethylcyclo- Polymethylsiloxane, bis-3,4- epoxycyclohexyl- ethylphenylmethylsilane)		Ø 0.47 μm	76.0	
Exp Ormocer	Ormocer	Voco GmbH, Cuxhaven, Germany	not given	85% Ba-Al- borosilicate glass 15% SiO ₂	Ø2.5 μm 30-60 nm	84.5	
CeramX	Composite, nano-ceramic	Dentsply DeTrey, Konstanz, Germany	Methacrylate modified polysiloxane, dimethacrylate resin	Silanated Ba-Al- borosilicate glass silanated pyrogenic SiO ₂ (nano particle size)	Ø 1.1-1.5 μm Ø 10 nm (Ø 2.3 nm)	76.0	
Quixfil	Composite	Dentsply DeTrey, Konstanz, Germany	UDMA, TEGDMA, Di- and trimethacrylate resins, carboxylic acid modified dimethacrylate resin	Silanated strontium aluminum sodium fluoride phosphate silicate glass	1-10 μm	85.5	

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tions using custom-built silicone molds. The specimens were light-cured for 40 seconds from each side using a conventional polymerization device (800 mW/cm²; Elipar Trilight, 3M ESPE, Seefeld, Germany) and subsequently smoothed using grinding paper (grain 1000, Buehler, Lake Bluff, IL, USA) for removing material surplus and the oxygen-inhibited layer, and ensuring the standardized size of the beams.

Artificial Aging

Surface roughness, flexural strength and Vickers hardness were determined at baseline and after different artificial aging treatments. After preparation, the specimens were randomly allotted to one of the artificial aging protocols; 15 specimens were used for each material and protocol. Aging simulation was carried out either by storage in ethanol (Ethanol 96%, Carl Roth GmbH + Co KG, Karlsruhe, Germany), artificial saliva or distilled water for 7, 90 or 365 days (25°C, dark). Thermal cycling (alternate immersion of specimens in distilled water with a temperature of 5°C and 55°C; 2 x 3000 cycles, five minutes each) was carried out in a thermal cycler (Regensburger Kausimulator, EGO, Regensburg, Germany) and used as reference aging protocol. The artificial saliva consisted of 4.1 mM KH₂PO₄, 4.0 mM Na₂HPO₄, 24.8 mM KHCO₃, 16.5 mM NaCl, 0.25 mM MgCl₂, 4.1 mM citric acid and 2.5 mM CaCl210 and has been used in previous investigations for the artificial aging of resin specimens. 11 The pH of the artificial saliva solution was adjusted to 6.7 with 10 N HCl,10 and the solution was subsequently sterilized using single use filtration devices with a pore-size of 0.22 um (Vacuflo, Schleicher & Schüll Microscience GmbH, Dassel, Germany). 12 All aging media were exchanged every week during the artificial aging period.

Determination of Surface Roughness

Peak-to-valley surface roughness (SR; [µm]) was determined at three randomly selected spots of each specimen (two at the margins, one in the central position) using a profilometric contact surface measurement device (Perthen S6P, Feinprüf-Perthen, Göttingen, Germany). A distance of 1.75 mm was measured in one single line scan perpendicular to the expected grinding grooves using a standard diamond tip (tip radius 2 µm, tip angle 90°).

Determination of Flexural Strength

For determination of the flexural strength (FS; [MPa]), a three-point bending test was conducted according to ISO 4049 using a universal testing machine (Zwick 1446, Zwick, Ulm, Germany). After mounting the specimens in the testing device using rounded supports at a distance of 20 mm, the beams were loaded to failure using a crosshead speed of 1 mm/minute. FS was calculated according to the formula:

$$\sigma s = (F_{max} * 3 * l)/(2 * b * h^2)$$

where F_{max} is the fracture force (N) and l, b and h represent the length, width and height of the specimens.

Determination of Vickers Hardness

The Vickers hardness (VH; [HV]) was measured using a Zwick device (B3212001, Zwick) according to DIN 50133. A load of 0.5 kg was applied for 60 seconds using a pyramid-shaped die; the depth of the impression represented the hardness of the sample. VH is proportional to the quotient of the applied force and impression surface, which was part of a pyramid with a square base. The pyramid and impression were considered to have identical surface angles. VH was calculated using the formula:

$$VH = (0.102 * F * sin136^{\circ}/2)/d^{2}$$

where F represents the force $(9.81 \text{ m/s}^2 * \text{mass in kg})$ and d represents the diagonal of the pyramid basement.

Statistical Analysis

All the calculations and graphic display were carried out using SPSS 16.0 for Windows (SPSS Corporation, Chicago, IL, USA). Means and standard deviations were calculated. Normal distribution of data was verified using the Kolmogorov-Smirnov test. Three-way analysis of variance (ANOVA) was used to analyze the influence of aging duration (new, 7, 90, 365 days; thermal cycling), composite material and aging medium (ethanol, distilled water, artificial saliva) on SR, VH and FS. The Tukey-Kramer multiple comparison test was applied for post-hoc analysis. The level of significance was set to α =.05.

RESULTS

Surface Roughness (Table 2)

Three-way ANOVA indicated a significant influence of the composite material (p<.001), aging duration (p<.001) and aging medium (p<.001) on SR; interaction effects for composite material and aging duration, as well as aging duration and aging medium, were significant (p<.001). Post-hoc analysis showed that Quixfil yielded significantly higher SR than any other material (p<.001). The lowest values were observed for Filtek Silorane, CeramX and Filtek Supreme XT; the experimental ormocer yielded similar SR compared to *Filtek Supreme XT* (p=.849) but significantly higher values than Filtek Silorane (p=.009) or CeramX (p=.031). The lowest overall values for SR were detected for thermally-cycled specimens, which were significantly lower than at baseline (p<.001) or as all artificially aged specimens (p<.001, respectively). After artificial aging for 7, 90 and 365 days, post-hoc analysis showed a significant increase in SR compared to the baseline. Artificial aging in ethanol caused significantly higher values

Material	New	TC	Distilled Water			Ethanol		
			7	90	365	7	90	365
Filtek Supreme	0.06 (0.03)	0.07 (0.02)	0.06 (0.03)	0.09 (0.05)	0.06 (0.03)	0.07 (0.02)	0.07 (0.02)	0.07 (0.04)
Filtek Silorane	0.04 (0.0)	0.04 (0.01)	0.06 (0.03)	0.06 (0.02)	0.06 (0.03)	0.06 (0.02)	0.06 (0.03)	0.06 (0.04)
Exp Ormocer	0.06 (0.02)	0.07 (0.02)	0.07 (0.04)	0.08 (0.04)	0.07 (0.02)	0.07 (0.03)	0.09 (0.01)	0.09 (0.02)
CeramX	0.04 (0.01)	0.06 (0.04)	0.05 (0.02)	0.06 (0.02)	0.07 (0.02)	0.06 (0.04)	0.06 (0.02)	0.07 (0.04)
Quixfil	0.10 (0.03)	0.09 (0.02)	0.09 (0.02)	0.12 (0.04)	0.12 (0.05)	0.10 (0.02)	0.11 (0.01)	0.11 (0.02)
Material	Artificial Saliva							
	7	90	365					
Filtek Supreme	0.07 (0.04)	0.08 (0.02)	0.07 (0.05)					
Filtek Silorane	0.07 (0.03)	0.10 (0.02)	0.09 (0.03)					
Exp Ormocer	0.10 (0.02)	0.09 (0.02)	0.08 (0.02)					
CeramX	0.08 (0.04)	0.07 (0.03)	0.07 (0.03)					
Quixfil	0.11 (0.03)	0.13 (0.02)	0.13 (0.05)					

Material	Baseline	TC	Distilled Water			Ethanol		
			7	90	365	7	90	365
Filtek Supreme	90.6 (6.8)	80.8 (14.4)	97.0 (2.1)	78.1 (10.1)	80.2 (5.5)	96.6 (8.3)	47.5 (8.3)	86.4 (7.3)
Filtek Silorane	67.6 (3.4)	63.9 (5.0)	70.1 (13.5)	60.0 (3.8)	59.9 (6.7)	81.5 (22.2)	61.6 (1.9)	58.6 (7.2)
Exp Ormocer	122.5 (32.2)	119.4 (19.4)	86.4 (13.9)	98.9 (23.8)	90.5 (4.7)	121.1 (10.8)	100.1 (7.5)	95.9 (4.9)
CeramX	87.2 (4.1)	77.3 (8.7)	70.5 (11.9)	77.9 (2.2)	75.9 (7.1)	81.0 (3.8)	71.2 (7.8)	74.1 (1.6)
Quixfil	87.6 (11.5)	74.4 (5.6)	57.6 (43.6)	92.3 (4.4)	89.6 (4.9)	97.1 (13.4)	68.8 (3.5)	58.1 (2.1)
Material	Artificial Saliva							
	7	90	365					
Filtek Supreme	89.8 (6.0)	88.7 (2.6)	78.9 (6.5)					
Filtek Silorane	68.2 (3.1)	62.6 (15.4)	80.6 (6.4)					
Exp Ormocer	94.9 (1.14)	94.7 (11.1)	95.8 (1.1)					
CeramX	79.3 (7.3)	75.7 (7.3)	77.6 (2.2)					
Quixfil	96.1 (9.9)	95.2 (22.1)	81.4 (4.7)					

for SR than artificial aging in distilled water (p=.001).

Vickers Hardness (Table 3)

Three-way ANOVA indicated significant influences of the composite material (p<.001) and the aging duration (p<.001) on VH; no significant influence was found for the aging medium (p=.380). Interaction effects for composite material and aging duration, as well as aging duration and aging liquid, were also significant (p<.001, respectively). Post-hoc analysis showed significantly lower VH for Filtek Silorane than for any other material (p<.001, respectively). Intermediate and not significantly statistically different values were found for CeramX, Quixfil and Filtek Supreme XT. A significantly higher VH was found for the experimental ormocer than for any other material (p<.001, respectively). The aging duration had a significant influence on VH: a significantly higher VH was found at the baseline than for specimens that had been stored for 90

(p<.001) or 365 days (p<.001) or those that had been thermally cycled (p=.025). Similar VH was found for specimens that had been aged for 7, 90 and 365 days, or those that were thermally cycled.

Flexural Strength (Table 4)

Three-way ANOVA indicated significant influences of the composite material (p<.001) and aging duration (p<.001) of FS, while no significant influence was found for the aging medium (p=.725). The interactions of the composite material and aging duration (p<.001), composite and aging medium (p=.025), as well as aging duration and aging medium (p<.001), were also significant. Post-hoc analysis showed significantly higher FS for *Filtek Silorane* and *Quixfil* than for the other materials (p<.001), respectively). Intermediate FS was found for *Filtek Supreme XT*, which was significantly higher than for the experimental ormocer (p<.001) and CeramX (p<.001), which showed the lowest FS. The aging duration had a significant influence on the FS of

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Material	Baseline	TC	Distilled Water			Ethanol		
			7	90	365	7	90	365
Filtek Supreme	144.8 (15.5)	63.2 (14.4)	125.6 (15.1)	113.8 (24.0)	78.8 (22.4)	146.8 (23.1)	112.7 (28.9)	47.8 (9.1)
Filtek Silorane	123.7 (18.3)	90.0 (12.3)	125.0 (17.7)	107.8 (13.8)	106.2 (23.8)	136.9 (26.3)	134.9 (19.3)	117.5 (12.6)
Exp Ormocer	118.8 (14.8)	84.9 (11.9)	116.5 (18.5)	83.1 (13.5)	73.9 (12.7)	117.3 (19.4)	73.8 (10.5)	44.6 (7.1)
CeramX	130.7 (16.6)	78.6 (14.0)	84.6 (37.0)	79.3 (17.2)	69.4 (17.2)	101.4 (30.2)	97.5 (9.4)	57.5 (5.9)
Quixfil	140.0 (29.8)	97.9 (20.9)	145.6 (16.3)	111.6 (24.3)	95.0 (10.9)	153.0 (18.7)	117.4 (16.3)	64.7 (4.4)
Material	Artificial Saliva							
	7	90	365					
Filtek Supreme	145.0 (15.1)	90.6 (13.1)	77.9 (16.7)					
Filtek Silorane	126.6 (15.2)	111.3 (26.9)	106.8 (13.2))					
Exp Ormocer	117.9 (13.9)	76.1 (13.1)	75.6 (13.8)					
CeramX	103.3 (21.2)	90.3 (19.9)	74.6 (13.7)					
Quixfil	148.2 (12.0)	105.7 (17.8)	97.4 (16.2)					

the specimens at baseline and for specimens that had been aged artificially for seven days; significantly higher FS was found than for specimens that had been thermally cycled or those that were aged artificially for 90 and 365 days (p<.001, respectively). For thermally-cycled specimens and those that had been aged for 365 days, significantly lower FS was found compared to those that had been aged for 90 days (p<.001, respectively).

DISCUSSION

The findings of this *in vitro* study support the research hypothesis, indicating that the composite material itself, the aging duration and, in part, the aging medium, all have a decisive influence on the mechanical properties of dental composites.

The mechanical properties of a light-cured dental composite material are particularly dependent on its filler content, the type of incorporated fillers and the efficiency of the filler-resin coupling.¹³ For this reason, composite materials differing in matrix, filler chemistry and composition have been examined in the current study, including a composite with a bis-GMAbased matrix and nano-scaled SiO2 and zirconia fillers (Filtek Supreme XT), materials with nano-scaled SiO₂ or Ba-Al-borosilicate glass particles and an ormocer-(Exp Ormocer) or methacrylate-based matrix (CeramX), a novel microfilled composite with SiO₂ and yttrium fluoride fillers and a silorane-based matrix (Filtek Silorane) and macrofilled composite materials with filler sizes between 1 and 10 µm and a conventional methacrylate-based resin matrix (Quixfil). SR, FS and VH have been chosen as representative mechanical parameters of dental composite materials and have been analyzed prior to and after different artificial aging treatments. FS has been defined as the maximum stress that a material can resist before failure when it is subjected to bending load6 and it is

regarded as the most significant measure of strength of dental materials, as considerable flexural stresses may occur during the complex mastication process.⁶ The hardness of composite materials is particularly dependent on the filler type and content and it correlates with mechanical properties, such as abrasion resistance or polishability.¹⁴⁻¹⁵ Additionally, the increased roughness of dental composites may foster the accumulation of plaque on the surface of the restoration, which results from surface deterioration due to degradation of the resin matrix. Thus, VH, SR and FS may be regarded as material properties contributing to the survival of direct composite restorations *in vivo*, which justifies their selection as test parameters in the current study.

For simulating different aspects of aging in vitro, four different aging protocols have been applied in the current study. Immersion in ethanol,6-7 artificial saliva7 and distilled water 6-7,16 have been widely used in previous investigations on similar topics. However, particularly for artificial saliva, it has been reported that most formulations are haphazard,17 because, for numerous artificial salivas that have been used in the past, no attempts have been made to justify the inclusion of individual components or analyze their impact on the tested materials.17 These considerations clearly underline the need for sufficient standardization of artificial saliva for the *in vitro* analysis of dental materials, as different artificial saliva formulations may provoke different test results. Nevertheless, it has been reported that the filler leachability of composite specimens was higher after storage in artificial saliva than in distilled water, 18 which justifies the use of artificial saliva as an artificial aging protocol in the current study. Thermal cycling has been applied as another protocol for simulating a clinical aging process; the thermal cycling protocol used in the current study has been adapted from a thermal cycling and mechanical loading protocol simulating a five-year clinical service. ¹⁹ Repeated temperature changes, which appear in the oral cavity, may induce degradation of matrix-filler bonds due to the different thermal expansion coefficients of fillers and the resin matrix.

Generally, numerous studies agree that artificial aging leads to a significant decrease in the mechanical properties of dental composite materials. 4-5,8,16 With regard to this aspect, the findings of this in vitro study indicate that the aging medium has a negligible influence on mechanical performance, such as FS and VH. Generally, aging in aqueous solutions or the oral cavity may contribute to the leaching of composite components, degeneration of the cross-linked resin matrix and fostering hydrolysis of the filler-matrix interfaces. 2-3,20-22 Some previous studies found that the release of particular filler particles, such as strontium or barium, were different after aging in ethanol, artificial saliva and distilled water. 4,23-24 However, as only little correlation has been observed between filler leaching and flexural strength, Drummond and others assumed that the deterioration of dental composites is more closely related to degradation of the resin matrix and the matrix-filler bonds than degradation of the glass fillers,25 which is in accordance with other reports.²⁰ Zhang reported that storage of specimens in a mixture of ethanol and water led to a significantly higher decrease in FS than did immersion in distilled water or artificial saliva.7 The differences between these reports and the findings of the current study might, however, be partially attributed to the use of model composite materials with identical filler content and shape. Drummond observed a significant influence of the composite material and aging medium, but not aging time and temperature,8 which partially contradicts the findings of the current study. In this investigation, a significant decrease in both VH and FS after artificial aging for 90 and 365 days, as well as thermal cycling, was observed in comparison to baseline and artificial aging for seven days. However, there are only a few studies that document simulated aging processes over more than several weeks, which might serve as an explanation for the different outcomes. Though no statistically significant differences could be observed in the current study, storage in ethanol for 365 days provoked the lowest values for VH and FS for any material, which underlines the suitability of this aging protocol.

Prolonged aging had a dramatic influence on the FS of the specimens; as early as after an artificial aging period of 90 days, a significant decrease in FS could be observed. For dental composite materials that need to yield sufficient durability against mastication forces, a minimum flexural strength of 80 MPa is demanded according to ISO 4049.²⁶ The findings of the current study indicate that all tested composites yield deci-

sively higher FS at baseline, yet after prolonged artificial aging, several materials (experimental ormocer, Filtek Supreme XT, CeramX, Quixfil) showed values lower than this threshold value, which might induce failure of the restoration in vivo due to fracture. The FS of a composite material is not only determined by its chemical composition but also by its surface texture; thus, the significant increase in SR after prolonged immersion of the specimens in ethanol, distilled water or artificial saliva might serve as an explanation for the significant decrease in FS. Suprisingly, the higher surface roughness that had been measured for Quixfil, due to its high content of large filler particles, did not correlate with the low FS in general. Moreover, after thermal cycling, no significant difference in SR was observed compared to the baseline; thus, it is clear that the influence of thermal cycling on the mechanical properties of composite materials needs to be further investigated. Most likely, repeated temperature changes lead to continuous weakening of the interface between the resin matrix and the filler particles due to their different thermal expansion coefficients, which has an impact on mechanical properties, such as FS. However, not all surface irregularities can be detected by means of profilometry; thus, further studies might employ additional methods, such as atomic force microscopy to determine surface defects.

Though the aging protocols that have been applied in the current study may be more rigid and not fully representative of a clinical aging process, it is clear that the mechanical aging behavior of dental composite materials needs to be further improved. The generally rather low FS that has been detected for the experimental ormocer and CeramX may be due to weaker bonds between fillers and the resin matrix compared to the other materials. It has been reported that ethanol has a similar solubility coefficient as Bis-GMA,27 which fosters the elution of unpolymerized monomers and, thus, degradation of the resin matrix due to plastization. With regard to this aspect, it might be possible that the high values for FS that have been found for Filtek Silorane are due to its particular monomer chemistry, which does not contain Bis-GMA-based resins. In addition, it is likely that the water absorption of Filtek Silorane is lower than that of conventional composite materials due to its hydrophobic siloxane backbone, which would result in the reduced elution of unpolymerized monomers.28 However, in contrast to FS, the experimental ormocer yielded the highest values for VH, which indicates that this composite has a high amount of filler particles; the low VH found for Filtek Silorane indicates that this material is rather low-filled (filler content 76% as purported by the manufacturer). The conversion rate of a composite material might also have an impact on VH, which indicates that further studies are necessary to investigate

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this aspect. However, it has been reported that the conversion rate of *Filtek Silorane* is similar to conventional composite materials.²⁹

CONCLUSIONS

The findings of the current study highlight that artificial aging has a decisive influence on the mechanical properties of dental composite materials and, for Vickers hardness and flexural strength, the aging duration and the material had a significantly more pronounced effect than the aging medium. Additionally, the findings show that different dental composites are not equally affected by artificial aging, though none of the tested materials showed satisfying behavior for all three test parameters. Reduced Vickers hardness, flexural strength and increased surface roughness of a composite restoration may cause increased wear, abrasion and plaque formation, as well as higher probabilities of restoration fractures in vivo; thus, within the limitations of an in vitro study, the findings indicate that the survival probability of composite restorations may still decrease with time due to significant deterioration of their mechanical properties.

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