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Influence of Thermal Stress on Simulated Localized and Generalized Wear of Nanofilled Resin Composites

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Clinical Relevance

When making treatment decisions, clinicians should consider the impact on nanofilled resin composite wear characteristics of degradation by occlusal and thermal stresses.

SUMMARY

Objective: This study investigated the influence of thermal stress on the simulated localized and generalized wear of nanofilled resin composites.

Methods: Six nanofilled resin composites were evaluated and then subjected to a wear challenge of 400,000 cycles in a Leinfelder-Suzuki (Alabama) wear simulation device after 24 hours of water storage (24-hour group) and 24 hours of water storage and 10,000 thermal cycles (TC group). Simulated localized wear was generated using a stainless-steel ball bearing, and simulated generalized wear was generated using a flat-ended stainless-steel cylinder. Wear testing was accomplished in a water slurry of polymethyl methacrylate beads. Simulated localized and generalized wear was determined using a noncontact profilometer (Proscan 2100) in conjunction with Proscan and AnSur 3D software.

Results: Wear was significantly different ($p<0.05$) among the resin composites for both simulated localized and generalized wear of either the 24-hour group or the TC group. The simulated localized wear of the TC group was significantly greater than that of the 24-hour group; however, the simulated generalized wear of most of the resin composites of the TC group was not significantly different from that of the 24-hour group.

Conclusion: The simulated localized and generalized wear of nanofilled resin composites is material dependent. The simulated localized wear of nanofilled resin composites appears to

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be influenced by thermal stress, whereas this effect is not as apparent in simulated generalized wear testing.

INTRODUCTION

Considerable improvements have been made over the past several years in resin composite materials with the modification of resin matrix formulations and filler characteristics.[1] More recently, improvements in resin composite technology have led to the introduction of nanofilled resin composites for clinical use.[2] Such resin composites have been reported to exhibit good mechanical properties,[3] improved surface characteristics,[4] better gloss retention,[5] and reduced polymerization shrinkage.[6]

However, controlled clinical studies on posterior restorations using nanofillled resin composites and microhybrid resin composites revealed no significant differences in clinical performance.[7,8] Wear properties of nanofilled resin composites remain a clinical concern, particularly in extensive posterior restorations and treatment of patients with abnormal occlusal behavior, such as clenching and/or bruxing.[9]

In clinical situations, occlusal stress is transmitted to resin composite restorations through rigid and brittle fillers into the more flexible resin matrix during both function and parafunction activities.[10] Stress concentrations at the filler–resin matrix interface can result in filler dislodgement and exposure of the resin matrix, leading to wear.[11] Such stress concentrations can also be generated by cyclic temperature changes.[12] During cyclic temperature changes, differences in thermal expansion coefficients between fillers and the resin matrix in resin composites may lead to interfacial stresses in restorations.[13]

A thermal cycling test is the process of subjecting a specimen to cyclic temperature changes through water immersion to mimic intraoral thermal changes.[14] Investigations in a previous study contended that 10,000 thermal cycles correspond to one year of clinical function of restorations. This estimate was based on the hypothesis that such cycles might occur 20 to 50 times per day.[15] A previous study reported that the flexural properties of nanohybrid resin composites significantly decreased after thermal cycling protocols (15,000, 30,000, and 45,000 cycles).[13] Another study reported that the effect of thermal cycling was material dependent in microhybird resin composites.[16] However, the influence of the combination of occlusal and thermal stress on nanofilled resin composites is still unclear. Evaluation of the interactive effects between occlusal and thermal stress on wear properties of resin composites is important for assessing their clinical performance[17] because these two types of stresses are different and may cause more wear in combination than they do individually. In addition, previous studies showed good agreement between simulated wear and clinical wear.[18,19]

Nanofilled resin composites incorporating a larger amount of nanosized filler particles in a more homogeneous distribution in the resin matrix have a larger interface area between fillers and a resin matrix than do conventional resin composites.[17] Thus, the total interfacial stress between filler and resin matrix generated by intraoral conditions in nanofilled resin composite has been estimated to be greater than that in conventional resin composites.[18] This may lead to unpredicted clinical performance of nanofilled resin composite restorations, especially in extensive posterior restorations and patients with abnormal occlusal habits, such as clenching or bruxing. Therefore, evaluation of the wear resistance of nanofilled resin composites after thermal cycling test may provide valuable information about the longevity of clinical restorations.

This study aimed to investigate the influence of thermal stress on simulated localized and generalized wear of nanofilled resin composites. The null hypotheses to be tested were 1) that no significant differences exist in simulated localized and generalized wear of different nanofilled resin composites and 2) that simulated localized and generalized wear of nanofilled resin composites are not influenced by thermal stress.

METHODS AND MATERIALS

Study Materials

Six nanofilled resin composites were evaluated in this study: 1) Filtek Supreme Ultra Universal Restorative (FS; 3M ESPE, St Paul, MN, USA), 2) MI Gracefil (MG; GC, Tokyo, Japan), 3) TPH Spectra high viscosity (TS; DENTSPLY Caulk, Milford, DE, USA), 4) Clearfil Majesty ES-II (CM; Kuraray Noritake Dental, Tokyo, Japan), 5) Estelite Σ Quick (EQ; Tokuyama Dental, Tokyo, Japan), and 6) Beautifil II (B2; Shofu, Kyoto, Japan). The nanofilled resin composites used in this study are listed in Table 1 with their associated lot numbers, components, and the percentage of filler loading provided by the manufacturers.

Table 1: *Nanofilled Resin Composites*

Nanofilled Resin Composite (Code, Shade)	Resin Matrix Composition	Inorganic Filler (Content)	Manufacturer (Lot No.)
Filtek Supreme Ultra Universal Restorative (FS, A2)	Bis-GMA, UDMA, TEGDMA, Bis-EMA, PEGDMA	Nonagglomerated/nonaggregated silica filler, nonagglomerated/nonaggregated zirconia filler, aggregated zirconia/silica cluster filler (78.5wt%, 63.3vol%)	3M ESPE, St Paul, MN, USA (N697117)
MI Gracefil (MG, A2)	Bis-EMA, UDMA	Silica glass, strontium glass, fluoro-alumino-silicate glass (77.0 wt%, 65.0 vol%)	GC, Tokyo, Japan (1407281)
TPH Spectra High Viscosity (TS, A2)	Urethane modified Bis-GMA, TEGDMA	Silanated barium-alumino-borosilicate glass, silanated barium boron fluoro alumino silicate glass (77.2wt%, 57.0vol%)	DENTSPLY Caulk, Milford, DE, USA (150317)
Clearfil Majesty ES-II (CM, A2)	Bis-GMA	Silanated barium glass filler, (78.0wt%, 66.0vol%)	Kuraray Noritake Dental, Tokyo, Japan (A90026)
Estelite Σ Quick (EQ, A2)	Bis-GMA, TEGDMA	Silica zirconia filler (82.0wt%, 71vol%)	Tokuyama Dental, Tokyo, Japan (13394)
Beautifil II (B2, A2)	Bis-GMA, TEGDMA	Fluoro boro alumino silicate glass (83.3wt%, 68.6vol%)	SHOFU, Kyoto, Japan (011592)

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; UDMA, urethane dimethacrylate; Bis-EMA, ethoxylated bisphenol A dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; Bis-MEPP, bisphenol A ethoxylate dimethacrylate.

Specimen Preparation

Forty wear specimens of each of the six resin composites were prepared for simulated localized wear (occlusal contact area [OCA] wear), and 40 wear specimens of each material were prepared for generalized wear (contact-free area [CFA] wear). Custom stainless-steel fixtures were machined for localized wear testing with a cylindrical cavity 6.5 mm in diameter and 4.0 mm in depth. Stainless-steel fixtures for generalized wear testing were machined with a cylindrical cavity 4.5 mm in diameter and 4.0 mm in depth. Two increments of the resin composites (approximately 2.0 mm in thickness) were placed in the cylindrical-shape cavities of the localized and generalized fixtures, and each increment was cured for 40 seconds using a quartz-tungsten halogen unit (Spectrum 800, DENTSPLY Caulk) set at 600 mW/cm^2. The specimens for localized and generalized wear testing were randomly allocated into two groups (n=20 per group): 1) specimens stored in 37°C distilled water for 24 hours (24-hour group) and 2) specimens stored in 37°C distilled water for 24 hours and thermal cycled for 10,000 cycles (TC group). After 24 hours of water storage, all the composite surfaces were wet polished flat to a 4000-grit surface using a sequence of silicon carbide papers (Struers, Cleveland, OH, USA). For the TC group, thermal cycling was conducted using a custom-built apparatus, and each cycle consisted of water bath incubation for 30 seconds between water baths of 5°C and 55°C with a transfer time of five seconds for 10,000 cycles.

Wear Simulation

A Leinfelder-Suzuki (Alabama) device was used for wear simulation. The simulator has a plastic water bath, and the custom wear fixtures were mounted inside the four-station bath. A brass cylinder was then placed around each fixture in the bath to serve as a reservoir for the abrasive media (ie, a water slurry of unplasticized polymethyl methacrylate [PMMA] with an average particle size of 44 μm). The media was placed inside the brass cylinders to cover the surface of the resin composite in the custom fixtures. The water slurry of PMMA inside the brass cylinders was approximately 6.0 mm in thickness over the surface of the resin composite.

Two different wear antagonists were used in this study. For the localized (OCA) wear simulation, a stainless-steel ball bearing (2.387-mm radius) was mounted inside a collet assembly. The antagonist for the generalized (CFA) wear simulation was a stainless-steel cylinder (6.5 mm in diameter) with a flat-end stylus tip. The antagonist tips were mounted on spring-loaded pistons to deliver the wear challenges. During the application of the load, the antagonists rotated approximately 30 degrees as the maximum force was reached (maximum load of 78.5 N at a rate of 2 Hz) and then counterrotated back to the original starting position as the load was relaxed to complete the cycle. Each set of specimens was exposed to 400,000 cycles in the wear simulator.

Wear Measurements

Prior to wear simulation, each resin composite specimen was profiled using a Proscan 2100 noncontact optical profilometer (Scantron Industrial Products, Taunton, UK) with Proscan software. These profiles provided the pretest digitized contours (20 specimens for each of the six resin composite materials for both localized and generalized wear testing).

After the 400,000 wear cycles, the specimens were ultrasonically cleaned (L&R T-14B solid-state ultrasonic cleaner, L&R Manufacturing Company, South Orange, NJ, USA) in distilled water for three minutes and then profiled again using the Proscan 2100 unit. The X, Y, and Z coordinates of the before and after scans were exported from the Proscan software to another computer for analysis using AnSur 3D software (Minnesota Dental Research Center for Biomaterials and Biomechanics, University of Minnesota, Minneapolis, MN, USA).

Facet depth and volume loss measurements were determined from differences between the before and after data sets. A computerized fit was completed using the before and after data sets in AnSur 3D, and volume loss (VL; mm^3) was then determined for both localized and generalized wear simulation for each of the six resin composites. The maximum depth of the wear facet (MXD; μm) was also determined for the localized wear specimens, and the mean depth (MD; μm) of the wear facet was determined for the generalized wear specimens.

Scanning Electron Microscopic Observations of Resin Composite Surfaces

Ultrastructural observations were conducted on the polished surfaces of the nanofilled resin composites after argon-ion etching. Three specimens per group were observed using field-emission scanning electron microscopy (SEM; ERA 8800FE, Elionix, Tokyo, Japan).

The surfaces of the nanofilled resin composites were wet polished with a sequence of SiC papers (#600, #1200, and #4000 grit) using a grinder-polisher (Ecomet 4, Buehler). The surfaces were then polished with abrasive discs (Fuji Star Type DDC, Sankyo-Rikagaku, Saitama, Japan) followed by a series of diamond pastes down to 0.25-μm particle size (DP-Paste, Struers, Ballerup, Denmark) to bring the surfaces to a high gloss. SEM specimens of the polished surfaces were dehydrated by immersion in aqueous *tert*-butanol solutions with ascending concentrations (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes, and 100% for two hours) and were then transferred to a critical-point dryer (Model ID-3, Elionix, Tokyo, Japan) for 30 minutes. These polished surfaces were etched for 30 seconds using an argon ion-beam (EIS-200ER, Elionix, Tokyo, Japan) directed perpendicular to the surface at an accelerating voltage of 1.0 kV and an ion current density of 0.4 mA/cm^2 to enhance the visibility of the filler particles. Surfaces were coated with a thin film of gold in a vacuum evaporator (Quick Coater SC-701, Sanyu Electron, Tokyo, Japan). SEM observations were carried out using an operating voltage of 10 kV.

SEM Observations of Wear Facets

Ultrastructural observations were carried out on representative wear facets of the nanofilled resin composites of the 24-hour group and the TC group after 400,000 cycles for both localized and generalized wear simulation using SEM (TM3000 tabletop microscope, Hitachi-High Technologies, Tokyo, Japan). After the wear analysis, representative specimens were coated with a thin film of gold-palladium in a vacuum evaporator (Emitech SC7620 Mini Sputter Coater, Quorum Technologies, Ashford, UK). The SEM observations were carried out using an operating voltage of 15 kV.

Statistical Analysis

The VL for localized and generalized wear, MXD for localized wear, and MD for generalized wear of the nanofilled resin composites were analyzed using a commercial statistical software package (SPSS Statistics Base, International Business Machines, Armonk, NY, USA). A two-way analysis of variance (ANOVA) and a Tukey *post hoc* test were used for data analysis of each data set, with a significance level of $\alpha = 0.05$.

RESULTS

Localized Wear Simulation

The two-way ANOVA of VL and MXD of wear facets showed a significant effect ($p<0.05$) for the factors of material type and thermal cycling. No significant interaction ($p>0.05$) was found between these factors for either VL or MXD.

The results for the simulated localized wear of nanofilled resin composites are shown in Tables 2 and 3. The VL and MXD of wear facets in both the 24-hour group and the TC group were significantly different ($p<0.05$) depending on the material, and

Table 2: *Results for Simulated Localized Wear of Resin Composites*[a]

Material	Volume Loss (VL, mm^3)		Maximum Depth of Wear Facets (MXD, μm)	
	24 h	10,000 TCs	24 h	10,000 TCs
FS	0.034 (0.012) aA	0.048 (0.009) aB	110.6 (13.5) aA	139.4 (14.5) aA
EQ	0.040 (0.015) aA	0.065 (0.017) abB	125.6 (12.2) abA	155.7 (11.1) bB
MG	0.045(0.012) abA	0.058 (0.010) abB	138.8 (18.0) bA	152.6 (10.7) bA
BII	0.057 (0.018) bA	0.084 (0.017) cB	144.9 (15.7) bcA	178.9 (12.6) cA
TS	0.058 (0.020) bA	0.076 (0.016) bcB	148.4 (15.5) bcA	165.8 (10.5) cA
CM	0.059 (0.021) bA	0.089 (0.025) cB	151.5 (14.7) cA	179.5 (12.8) cA

Abbreviations: BII, Beautifil II; CM, Clearfil Majesty ES-II; EQ, Estelite Σ Quick; FS, Filtek Supreme Ultra Universal Restorative; MG, MI Gracefil; TS, TPH Spectra High Viscosity.
[a] *Values in parentheses are standard deviations (n=20). Same small letter in same vertical column indicates no significant difference (p>0.05). Same capital letter within individual rows indicates no significant difference (p>0.05).*

the VL and MXD of wear facets of the TC group were significantly greater ($p<0.05$) than those of the 24-hour group.

Generalized Wear Simulation

The two-way ANOVA of VL and MD of wear facets showed a significant effect ($p<0.05$) for the factor of material type. No significant effect ($p>0.05$) was found for the factor of thermal cycling or for the interaction of material type and thermal cycling for either VL or MD.

The results for the simulated generalized wear of nanofilled resin composites are presented in Tables 4 and 5. The VL and MD of wear facets for both the 24-hour group and the TC group showed significant differences ($p<0.05$) among materials when comparing the wear within each group. However, the VL and MD of wear facets of the TC group were, in general, not significantly different ($p>0.05$) from those of the 24-hour group. The exception was EQ, for which both VL and MD were significantly greater ($p<0.05$) for the TC group than for the 24-hour group.

SEM Observations of Resin Composite Surfaces

Representative SEM images of polished nanofilled resin composite surfaces with argon-ion etching are shown in Figure 1. The argon-ion etching revealed clear differences in filler particle size, shape, and distribution. Resin composites exhibited a wide variety of filler particle sizes and shapes.

SEM images of polished FS surfaces with argon-ion etching showed a wide size range (1 to 8 μm) of irregular particles and small irregular particles. SEM images of polished MG surfaces with argon-ion etching showed a wide size range (1 to 2 μm) of irregular particles and small irregular particles. SEM images of polished EQ surfaces with argon-ion etching showed uniform small spherical particles. SEM images of polished CM surfaces with argon-ion etching showed a wide size range (1 to 15 μm) of irregular particles and small irregular particles. SEM images of polished TS surfaces with argon-ion etching showed a wide size range (1 to 4 μm) of irregular particles and small irregular particles, and filler particle plucking was clearly observed. SEM images of polished B2 surfaces with argon-ion etching

Table 3: *Results for Simulated Generalized Wear of Resin Composites*[a]

Material	Volume Loss (VL, mm^3)		Mean Facet Depth (MD, μm)	
	24 h	10,000 TCs	24 h	10,000 TCs
FS	0.284 (0.082) aA	0.296 (0.095) aA	21.6 (5.9) aA	24.5 (8.5) aA
EQ	0.269 (0.075) aA	0.432 (0.115) bB	24.6 (5.7) aA	38.6 (10.4) bB
MG	0.346 (0.066) abA	0.391 (0.082) abA	31.7 (6.1) bA	34.6 (7.7) bA
BII	0.402 (0.117) bcA	0.412 (0.107) abA	31.5 (8.9) bA	33.9 (9.6) bA
TS	0.520 (0.094) cA	0.501 (0.080) bcA	38.5 (6.5) bcA	37.4 (6.5) bA
CM	0.553 (0.153) cA	0.589 (0.141) cA	40.2 (7.3) cA	44.5 (9.8) bcA

Abbreviations: BII, Beautifil II; CM, Clearfil Majesty ES-II; EQ, Estelite Σ Quick; FS, Filtek Supreme Ultra Universal Restorative; MG, MI Gracefil; TS, TPH Spectra High Viscosity.
[a] *Values in parentheses are standard deviations (n=20). Same small letter in same vertical column indicates no significant difference (p>0.05). Same capital letter within individual rows indicates no significant difference (p>0.05).*

Table 4: Simulated Localized Wear of Resin Composites -Volume Loss (VL, mm^3)

Material	24h group	TC group
FS	0.034 (0.012)^{a,A}	0.048 (0.009)^{a,B}
EQ	0.040 (0.015)^{a,A}	0.065 (0.017)^{a,b,B}
MG	0.045 (0.012)^{a,b,A}	0.058 (0.010)^{a,b,B}
B2	0.057 (0.018)^{b,A}	0.084 (0.017)^{c,B}
TS	0.058 (0.020)^{b,A}	0.076 (0.016)^{b,c,B}
CM	0.059 (0.021)^{b,A}	0.089 (0.025)^{c,B}

Values in parenthesis are standard deviations (n = 20). Same small letter in same vertical column indicates no significant difference ($p>0.05$). Same capital letter within individual rows indicates no significant difference ($p>0.05$).

Table 5: Simulated Localized Wear of Resin Composites Maximum depth of wear facets (MXD, μm)

Material	24h group	TC group
FS	110.6 (13.5)^{a,A}	139.4 (14.5)^{a,A}
EQ	125.6 (12.2)^{a,b,A}	155.7 (11.1)^{b,B}
MG	138.8 (18.0)^{b,A}	152.6 (10.7)^{b,A}
B2	144.9 (15.7)^{b,c,A}	178.9 (12.6)^{c,A}
TS	148.4 (15.5)^{b,c,A}	165.8 (10.5)^{c,A}
CM	151.5 (14.7)^{c,A}	179.5 (12.8)^{c,A}

Values in parenthesis are standard deviations (n = 20). Same small letter in same vertical column indicates no significant difference ($p>0.05$). Same capital letter within individual rows indicates no significant difference ($p>0.05$).

showed a wide size range (1 to 10 μm) of irregular particles and small irregular particles.

SEM Observations of Wear Facets

Representative SEM images of wear facets after simulated localized wear of the 24-hour group and the TC group are shown in Figure 2. Low-magnification SEM images show that the localized wear facets of the nanofilled resin composites of the TC group were larger than those of the 24-hour group, and high-magnification SEM images showed that wear facets of the TC group exhibit more filler particle plucking or cracking than do those of the 24-hour group.

Representative SEM images of wear facets of the 24-hour group and the TC group after simulated generalized wear are shown in Figure 3. SEM images of the generalized wear facets of the nanofilled resin composites, except those of EQ, did not show any clear difference between the wear of the 24-hour group and the TC group. The generalized wear facets of EQ revealed severe cracking and damage for the TC group compared to the 24-hour group.

DISCUSSION

Typically, the wear resistance of restorative materials is evaluated by laboratory methods that involve an electric device that repeatedly makes contact with the material being tested using an antagonist object, such as a stylus.[17] In 2001, the International Organization for Standardization (ISO) published a technical specification, "Wear by Two- and/or Three-Body Contact," describing eight laboratory methods.[20] One of the methods identified by the ISO publication was the Alabama method, which was developed at the University of Alabama.[21] Barkmeier and others[22] have developed two distinct types of wear simulation using the Alabama wear simulation device. One of these is simulated localized wear (the type of wear represented by direct tooth to material contact), and the other is simulated generalized wear (the type of wear generated by a food bolus during mastication). Barkmeier and others[22] also reported test methods for determining simulated localized and generalized wear with the Alabama machine using a Proscan 2100 noncontact profilometer in conjunction with AnSur 3D software for wear analysis. In the present study, measurements of simulated wear of resin composites were conducted according to these methods.

Nanofilled resin composites incorporating a larger amount of nanosized filler particles in a more homogeneous distribution in the resin matrix have a larger interface area between fillers and a resin matrix than do microhybrid resin composites.[23] Thus, the total interfacial stress between filler and resin matrix generated by intraoral conditions in nanofilled resin composite has been estimated to be greater than that in microhybrid resin composites.[24] As some but not all microhybrid resin composites showed reduced wear resistance after thermal cycling,[16] it is important to confirm the effect of such treatment on nanofilled resin composites.

The simulated localized and generalized wear of resin composites of the 24-hour group was found to be significantly different depending on the material.

Bayne and others[25] have reported that the mean filler particle size affects the simulated wear of resin composites and that resin composites that include smaller filler particles exhibit less simulated wear. The results of the present study are consistent with this observation to a certain extent. EQ, which contains smaller spherical filler particles (less than 1.0 μm), exhibited less simulated wear in the 24-hour group, whereas the wear of CM (1 to 15 μm), which includes a wider size range of larger filler particles,

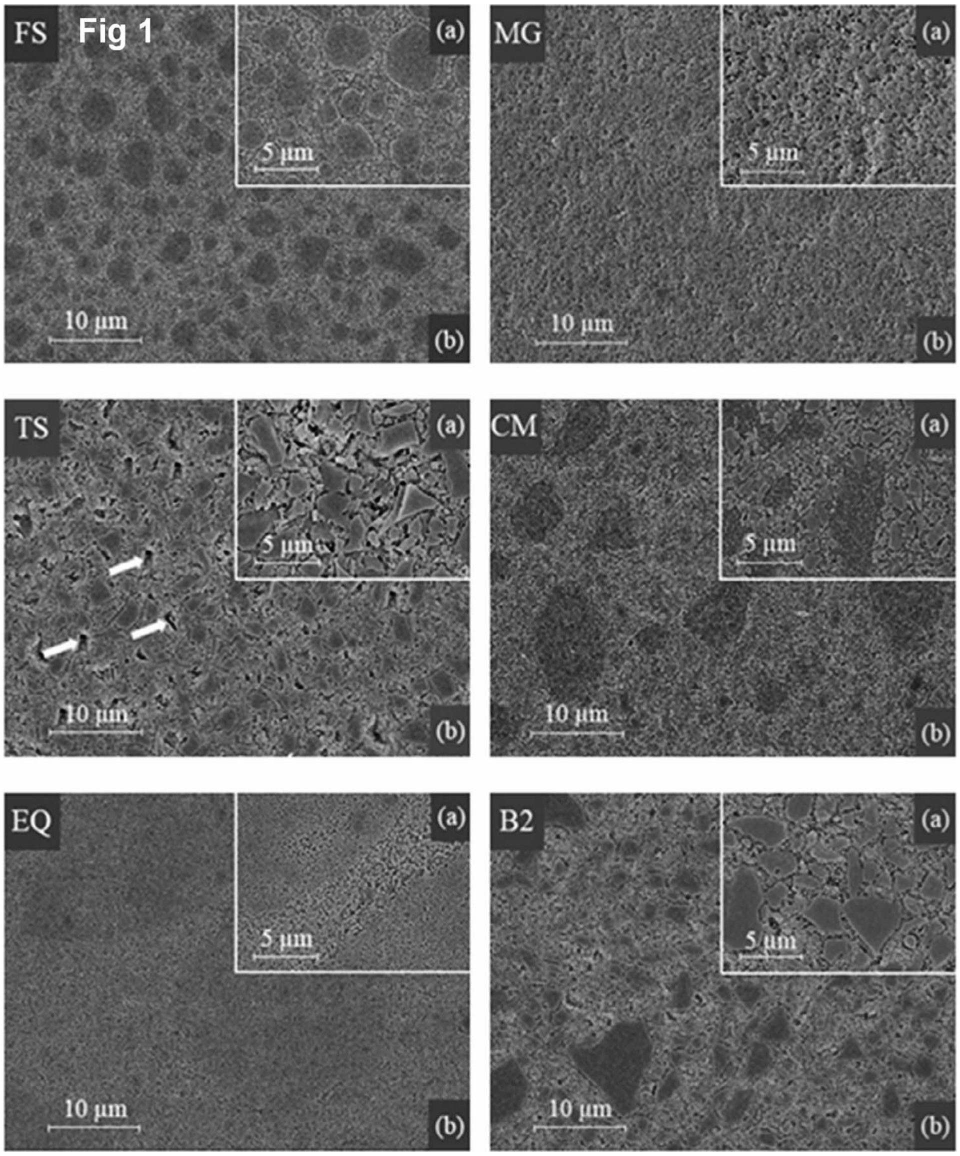

Figure 1. *Representative scanning electron microscopic (SEM) images of the polished resin composite surfaces at 10,000× magnification (a) and 2500× magnification (b). FS: wide size range (1 to 8 μm) of irregular-shaped particles and small irregular-shaped particles; MG: wide size range (1 to 2 μm) of irregular-shaped particles (black arrow) and small irregular-shaped particles (white arrow); TS: wide size range (1 to 4 μm) of irregular-shaped particles and small irregular-shaped particles; CM: wide size range (1 to 15 μm) of irregular-shaped particles and small irregular-shaped particles; EQ: uniform small spherical particles; B2: wide size range (1 to 10 μm) of irregular-shaped particles and small irregular-shaped particles. Size and distribution of fillers of nanofilled resin composites differed depending on the material. Filler particle plucking was notably observed in TS (white arrows).*

was greater regardless of the testing method. The difference in mean filler particle size between EQ and CM was clearly visible in SEM observations of their polished and argon-ion–etched surfaces. The difference in wear between these materials has been hypothesized to result from the smaller interparticle spacing between the small filler particles. The smaller filler particles are more closely packed; thus, the resin matrix between them is protected from further wear.[26] By contrast, when larger filler particles are dislodged from the resin composite surfaces, a void is produced, and the underlying resin matrix is exposed to the wear challenges.[27] In addition, the dislodged particles may act as a further abrasive to the resin composite surface, resulting in increased wear.[28]

On the other hand, TS, which includes relatively small filler particles (1 to 4 μm), exhibited significantly greater simulated localized and generalized wear than MG (1 to 2 μm) and SUFS (1 to 8 μm) and was similar to CM (1 to 15 μm) in both the localized and the generalized wear simulation and to B2 (1 to 10 μm) in localized wear simulation. Therefore, in this study, mean filler size did not directly influence the simulated localized and generalized wear of resin composites. As simulated wear is affected by many factors, it is not surprising that the association with a single factor is relatively weak. For instance, in SEM observations, TS clearly exhibited filler particle plucking not only in the worn surfaces of wear simulation specimens but also on polished surfaces after argon-ion etching. Although wear simulation

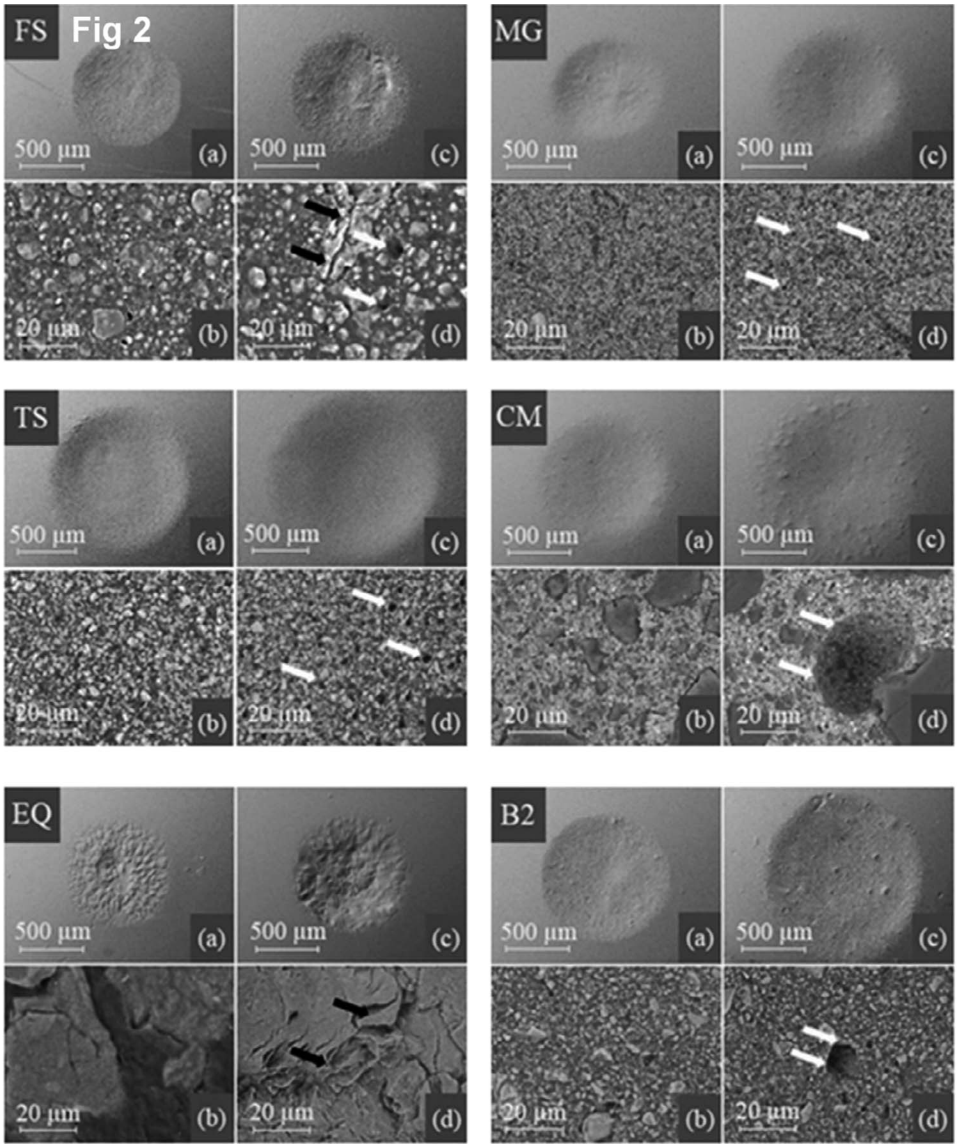

Figure 2. *Representative scanning electron microscopic (SEM) images of the localized wear facets of nanofilled resin composites after 24 hours of water storage, as viewed at 100× magnification (a) and 2500× magnification (b) and after 24 hours of water storage and thermal cycling at 100× magnification (c) and 2500× magnification (d). These low-magnification SEM images show that the localized wear facets of nanofilled resin composites after thermal cycling were larger than those after 24 hours of water storage. These high-magnification images show that wear facets after thermal cycling exhibit more filler particle plucking or cracking than those of 24 hours of water storage*.

specimens of other resin composites also showed filler particle plucking, filler particle plucking was not clearly observed after argon-ion etching of the polished surfaces for any tested resin composites apart from TS. One possibility is that weaker interfacial bonding between the filler and the resin matrix, which is associated with the surface treatment of the fillers, may be responsible for TS showing greater simulated wear. In addition, filler load has been reported to play a particularly important role in the wear resistance of resin composites, and a higher filler load has been reported to reduce the level of wear.[29] The values for filler load provided by the manufacturers vary greatly among the tested materials (Table 1). This may be another reason why the mean filler size did not directly determine the simulated wear of resin composites.

Although the simulated localized wear of tested resin composites in the TC group was significantly greater than that of the resin composites in the 24-hour group, the simulated generalized wear of resin composites of the 24-hour group and the TC group surprisingly did not show a significant difference, except in the case of ES. SEM observations of the worn surfaces of the localized wear simulation specimens in the TC group tended to indicate more cracking and filler particle plucking than was observed for corresponding specimens in the 24-hour group, whereas the generalized wear simulation specimens did not show any clear difference between wear in the 24-hour group and the TC group. In

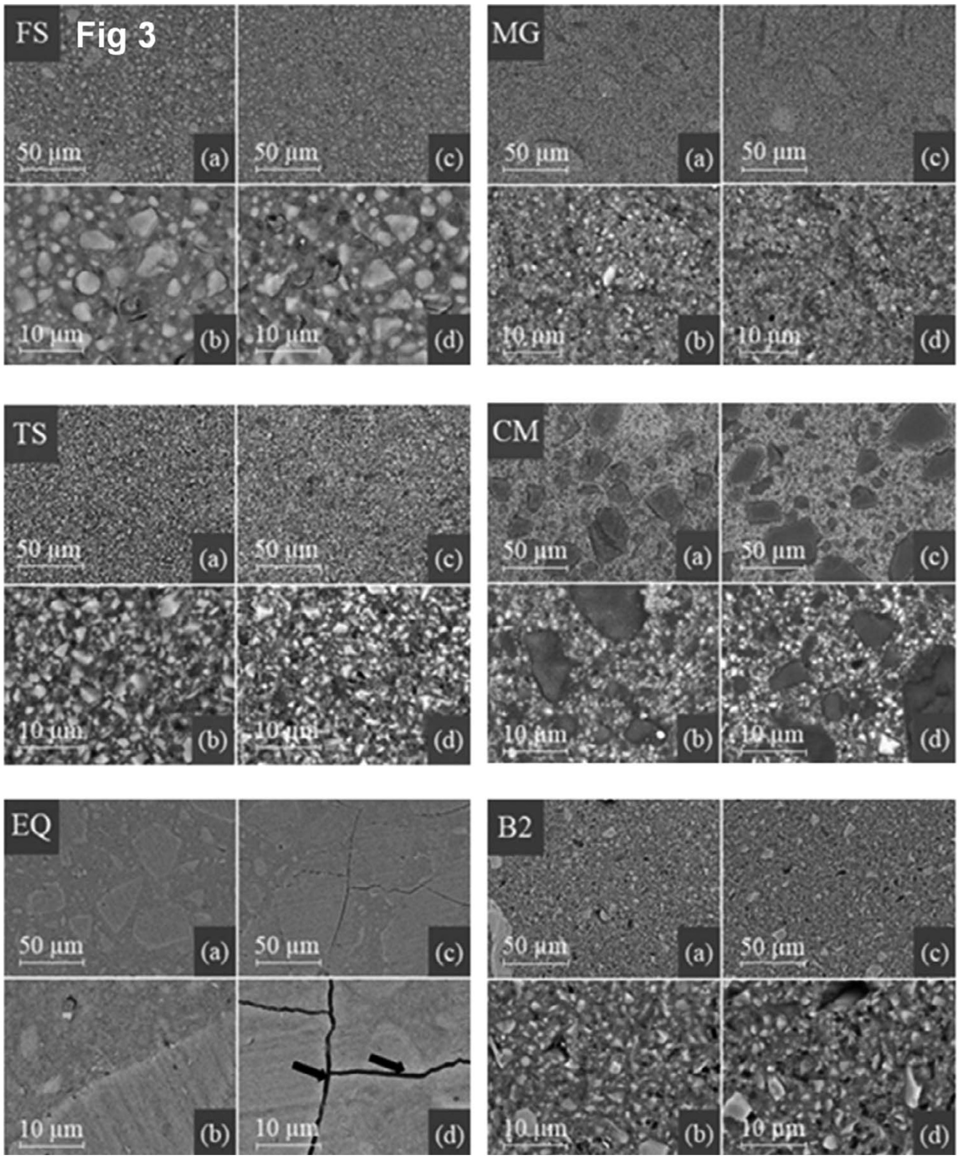

Figure 3. *Representative scanning electron microscopic (SEM) images of the generalized wear facets of nanofilled resin composites after 24 hours of water storage, as viewed at 1000× magnification (a) and 5000× magnification (b) and after 24 hours of water storage and thermal cycling at 1000× magnification (c) and 5000× magnification (d). SEM images of the generalized wear facets of nanofilled resin composites, except EQ, did not show any clear difference between wear after 24 hours of water storage and after thermal cycling. SEM images of the generalized wear facets of EQ after thermal cycling revealed severe cracking and damage compared to worn surfaces after 24 hours of water storage.*

addition to the thermal stresses introduced by differential thermal expansion between the filler and the matrix, immersion in water causes water penetration into the resin matrix of the resin composite, resulting in softening of the polymer matrix.[30] However, long-term thermal cycling may positively affect the resin composites and improve their mechanical properties with the resultant postcuring effects of time and heat.[31] The material degradation associated with the cyclic temperature changes and immersion in water of resin composites may be mitigated by the postcuring process. However, localized wear simulation of restorative materials appears to be much more aggressive than generalized wear simulation. Simulated localized wear facets may be more than twice as deep as the generalized facet depth because of accelerating filler plucking and the loss of matrix resin.[32] Therefore, material degradation of both the filler-resin matrix interface and the resin matrix itself caused by localized stress appeared to more directly influence the results of simulated localized wear of the resin composites in the TC group when compared with the results of simulated generalized wear.

The one exception in the aforementioned experiments was EQ, in which wear in the TC group was significantly greater than that of the 24-hour group regardless of the testing method. Similarly, SEM observations of the worn surface of wear simulation specimens of EQ in the TC group showed severe cracking and damage when compared with the 24-hour group. One possible explanation is that the increase in surface area of particles that accompanies the reduction in filler size in EQ results in a material more prone to water uptake, which might negatively affect mechanical properties as previously reported.[3] In addition, in connection with the larger surface area of filler particles, silanated fillers are susceptible to hydrolytic degradation,[6] which might adversely influence the filler-resin matrix interfaces of EQ over time.

On the basis of the results of this study, the first null hypothesis that no significant differences exist between simulated localized and generalized wear of nanofilled resin composites is rejected. The second null hypothesis was rejected for the simulated localized wear of nanofilled resin composites but is not rejected for the generalized wear of most nanofilled resin composites.

Overall, the simulated localized and generalized wear of the resin composites tested in this study appeared to be material dependent. Furthermore, the simulated localized wear of the resin composites was influenced by the thermal stress associated with thermal cycling, although such an influence was not as apparent in generalized wear simulation. However, several testing methodologies for laboratory simulation of wear are currently available, and these other methods may lead to substantially different results.[33] The results from other wear testing methods should therefore be carefully considered when attempting to discern the wear susceptibility of resin composites. Further *in vitro* wear tests combined with thermal cycling are important as initial evaluation methods of resin composites, especially for new products, to get an estimate of their clinical performance.

CONCLUSION

The results of this study indicate that the simulated localized and generalized wear of nanofilled resin composites is different, depending on the material. In addition, the simulated localized wear of nanofilled resin composites was influenced by thermal cycling, but the simulated generalized wear of most nanofilled resin composites was not. It appears that thermal cycling has potentially detrimental consequences for nanofilled resin composite restorations, particularly in occlusal contact areas. While this does not mean that such composites cannot be used in high-stress–bearing areas, it is important for clinicians to bear in the mind the risk of wear and to routinely check the restorations to determine whether repair is necessary.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Nihon University School of Dentistry.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Ferracane JL (2011) Resin composite—State of the art *Dental Materials* **27(1)** 29-38.
2. Mitra SB, Wu D, & Holmes BN (2003) An application of nanotechnology in advanced dental materials *Journal of the American Dental Association* **134(10)** 1382-1390.
3. Ilie N, & Hickel R (2009) Investigations on mechanical behaviour of dental composites *Clinical Oral Investigations* **13(4)** 427-438.
4. Ergücü Z, Türkün LS, & Aladag A (2008) Color stability of nanocomposites polished with one-step systems *Operative Dentistry* **33(4)** 413-420.
5. Egilmez F, Ergun G, Cekic-Nagas I, Vallittu PK, & Lassila LV (2012) Estimation of the surface gloss of dental nano composites as a function of color measuring geometry *American Journal of Dentistry* **25(4)** 220-226.
6. Sideridou ID, Karabela MM, & Vouvoudi ECh (2011) Physical properties of current dental nanohybrid and nanofill light-cured resin composites *Dental Materials* **27(6)** 598-607.
7. Palaniappan S, Bharadwaj D, Mattar DL, Peumans M, Van Meerbeek B, & Lambrechts P (2011) Nanofilled and microhybrid composite restorations: Five-year clinical wear performances *Dental Materials* **27(7)** 692-700.
8. Frankenberger R, Reinelt C, & Krämer N (2014) Nanohybrid vs. fine hybrid composite in extended class II cavities: 8-year results *Clinical Oral Investigations* **18(1)** 125-137.
9. Ferracane JL (2013) Resin-based composite performance: Are there some things we can't predict? *Dental Materials* **29(1)** 51-58.
10. Hahnel S, Schultz S, Trempler C, Ach B, Handel G, & Rosentritt M (2011) Two-body wear of dental restorative materials *Journal of the Mechanical Behavior of Biomedical Materials* **4(3)** 237-244.
11. Oliveira GU, Mondelli RF, Charantola Rodrigues M, Franco EB, Ishikiriama SK, & Wang L (2012) Impact of filler size and distribution on roughness and wear of composite resin after simulated toothbrushing *Journal of Applied Oral Science* **20(5)** 510-516.

12. Blumer L, Schmidli F, Weiger R, & Fischer J (2015) A systematic approach to standardize artificial aging of resin composite cements *Dental Materials* **31(7)** 855-863.

13. Morresi AL, D'Amario M, Monaco A, Rengo C, Grassi FR, & Capogreco M (2015) Effects of critical thermal cycling on the flexural strength of resin composites *Journal of Oral Science* **57(2)** 137-143.

14. Chadwick RG (1994) Thermocycling—The effects upon the compressive strength and abrasion resistance of three composite resins *Journal of Oral Rehabilitation* **21(5)** 533-543.

15. Gale MS, & Darvell BW (1999) Thermal cycling procedures for laboratory testing of dental restorations *Journal of Dentistry* **27(2)** 89-99.

16. Yap AU, Wee KE, Teoh SH, & Chew CL (2001) Influence of thermal cycling on OCA wear of composite restoratives *Operative Dentistry* **26(4)** 349-356.

17. Heintze SD (2006) How to qualify and validate wear simulation devices and methods *Dental Materials* **22(8)** 712-734.

18. Barkmeier WW, Latta MA, Erickson RL, & Lambrechts P (2004) Comparison of laboratory and clinical wear rates of resin composites *Quintessence International* **35** 269-274.

19. Barkmeier WW, Latta MA, Erickson RL, & Wilwerding TM (2008) Wear simulation of resin composites and the relationship to clinical wear *Operative Dentistry* **33(2)** 77-182.

20. ISO Standards (2001) ISO14569 Dental materials—Guidance on testing of wear—Part 2: Wear by two- and/or three-body contact *International Organization for Standardization* **1st edition** 1-31.

21. Leinfelder KF, & Suzuki S (1999) In vitro wear device for determining posterior composite wear *Journal of the American Dental Association* **130(9)** 1347-1353.

22. Barkmeier WW, Takamizawa T, Erickson RL, Tsujimoto A, Latta M, & Miyazaki M (2015) Localized and generalized simulated wear of resin composites *Operative Dentistry* **40(3)** 322-335.

23. Ilie N, Rencz A, & Hickel R (2013) Investigations towards nano-hybrid resin-based composites *Clinical Oral Investigations* **17(1)** 185-193.

24. Hosseinalipour M, Javadpour J, Rezaie H, Dadras T, & Hayati AN (2010) Investigation of mechanical properties of experimental Bis-GMA/TEGDMA dental composite resins containing various mass fractions of silica nanoparticles *Journal of Prosthodontics* **19(2)** 112-117.

25. Bayne SC, Taylor DF, & Heymann HO (1994) Protection hypothesis for composite wear *Dental Materials* **8(5)** 305-309.

26. Tsujimoto A, Barkmeier WW, Takamizawa T, Latta MA, & Miyazaki M (2017) Influence of thermal stress on flexural properties and simulated wear of computer-aided design/computer-aided manufacturing resin composite *Operative Dentistry* **42(1)** 101-110.

27. Hu X, Marquis PM, & Shortall AC (1999) Two-body in vitro wear study of some current dental composites and amalgams *Journal of Prosthetic Dentistry* **82(2)** 214-220.

28. Finlay N, Hahnel S, Dowling AH, & Fleming GJ (2013) The in vitro wear behavior of experimental resin-based composites derived from a commercial formulation *Dental Materials* **29(4)** 365-374.

29. Condon JR, & Ferracane JL (1997) In vitro wear of composite with varied cure, filler level, and filler treatment *Journal of Dental Research* **76(7)** 1405-1411.

30. Ferracane JL, Berge HX, & Condon JR (1998) In vitro aging of dental composites in water-Effect of degree of conversion, filler volume, and filler/matrix coupling *Journal of Biomedical Materials Research* **42(3)** 465-472.

31. Weir MD, Moreau JL, Levine ED, Strassler HE, Chow LC, & Xu HH (2012) Nanocomposite containing CaF(2) nanoparticles: Thermal cycling, wear and long-term water-aging *Dental Materials* **28(6)** 642-652.

32. Latta MA, Barkmeier WW, Wilwerding TM, & Blake SM (2001) Localized wear of compomer restorative materials *American Journal of Dentistry* **14(4)** 238-240.

33. Heintze SD, Faouzi M, Rousson V, & Ozcan M (2012) Correlation of wear in vivo and six laboratory wear methods *Dental Materials* **28(9)** 961-973.