

# Abrasion resistance of different resin composites

## *Resistência à abrasão de diferentes resinas compostas*

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### Abstract

**Objective** – This study evaluated the influence of toothbrushing and thermal cycling on surface roughness and weight alterations of four different composite resins. **Methods** – Five specimens of two nanofilled composites (Filtek Supreme and Esthet X), one submicron composite (spherical filler) (Palfique Estelite) and one microhybrid composite (irregular filler) (Filtek Z250/3M ESPE), were prepared according to the manufacturer's directions. Each specimen was thermal cycled 1,200 times and subjected to 40,000 toothbrushing cycles. At baseline and after thermal cycling and toothbrushing cycles, surface roughness and weight were determined. **Results** – After thermal cycling and toothbrush abrasion no statistically significant difference in weight was found, however, the Esthet X and Palfique Estelite showed decrease in weight while Filtek Supreme and Z250 showed increase in weight. The surface roughness increased after thermal cycling and toothbrushing ( $p < 0.01$ ). The spherical submicron filler (Palfique Estelite) showed the smoothest surface. **Conclusion** – According to the methodology applied in this study, all resins studied showed resistance to abrasion, however, Palfique Estelite after the tests showed the smoothest surface.

**Descriptors:** Composite resins; Nanotechnology; Dental restoration wear

### Resumo

**Objetivo** – Este estudo avaliou a influência da escovação e alterações térmicas quanto às alterações superficiais de rugosidade e peso de quatro diferentes resinas compostas. **Métodos** – Foram empregadas duas resinas de nanopartículas (Filtek Supreme e Esthet X), uma microparticulada de partículas esféricas (Palfique Estelite) e uma microhíbrida de partículas irregulares (Filtek Z250). Foram preparados cinco corpos de prova para cada material de acordo com as instruções do fabricante. Cada corpo de prova foi ciclado 1.200 vezes e submetido a 40.000 ciclos de escovação. O peso e rugosidade da superfície foram determinados antes e após os ciclos térmicos e de escovação. **Resultados** – Esthet X e Palfique Estelite mostraram diminuição do peso enquanto que Filtek Supreme e Z250 apresentaram aumento, no entanto, não houve diferença estatisticamente significativa após ciclagem térmica e de escovação. A rugosidade superficial aumentou em todas as resinas compostas testadas após a ciclagem térmica e de escovação, sendo que a resina microparticulada de partículas esféricas (Palfique Estelite) apresentou ao final dos testes a superfície mais lisa ( $p < 0.01$ ). **Conclusão** – De acordo com a metodologia aplicada nesta pesquisa, todas as resinas compostas estudadas apresentaram resistência à abrasão, no entanto, a Palfique Estelite apresentou ao final dos testes a maior lisura superficial.

**Descritores:** Resinas compostas; Nanotecnologia; Desgaste da restauração dentária

### Introduction

The quality of composite resins has improved greatly in recent years; however, its durability in the oral cavity depends on the characteristics inherent to the material. Superficial wear has been one of many obstacles facing composite resin restorations. Restorative material wear in the oral environment results from direct contact between the tooth and the restorations during mastication, oral parafunctions, toothbrushing with abrasive particles, as well as chemical effects caused by dietary factors<sup>1</sup>. The abrasion caused by toothbrushing can affect all surfaces of the teeth and therefore the restoration. Abrasion is an undesirable phenomenon, not only leading to an increase in surface roughness, but also resulting in the gradual removal of substance<sup>2</sup>. Restoration roughness increases the coefficient of friction and may increase the rate of wear<sup>3</sup>.

Despite the improvement in wear resistance of restorative materials, wear continues to be a problem<sup>4</sup>. Therefore, to achieve higher quality esthetics and longevity of composite resin restorations, new materials have been developed. Thus, manufacturers have recently used nanotechnology, also known as molecular nanotechnology or molecular engineering, which is the production of functional materials and structures in the range of 0.1 to 100 nanometers (nm)<sup>5</sup>. The new composite resin materials may contain nanometric particles (20 to 75 nm), nanoclusters (0.1 to 0.6  $\mu\text{m}$ ) and spherical submicron particles (0.1 to 0.3  $\mu\text{m}$ ). These innovations promise to optimize the physical properties of composite resins without compromising esthetics, in an endeavor to improve the clinical properties of these materials in the long term.

With the development of new composites is important to deter-

mine their performance after being subjected to abrasion. Therefore, this study was conducted to evaluate the effect of toothbrush abrasion and thermal cycling on the four composite resins.

### Methods

Four composites were tested: two nanofilled composites, Filtek Supreme (3M ESPE, St. Paul, Minn., USA) and Esthet-X (Dentsply-Caulk, Petrópolis, Rio de Janeiro, Brazil); one submicron composite with spherical filler, Palfique Estelite (Tokuyama Dental Corporation, Tokyo, Japan); and one microhybrid resin composite was used as control with irregular filler, Filtek Z250 (3M ESPE, St. Paul, Minn., USA). The technical profiles of the composites evaluated are shown in the Table 1.

Five cylindrical specimens were made of each material using a mold (3 mm thick and 10 mm in diameter). The mold was placed on Mylar matrix strips and a glass slide. The resin was inserted in bulk and another Mylar matrix strip and glass slide was placed on top of it to extrude the excess material. After removing the top glass slide, the samples were polymerized with a light-curing unit (Curing Light XL 3000, 3M ESPE, St. Paul, Minn.) for 60 seconds. The curing light unit was tested for light output using a curing radiometer (Model 100, Demetron Research Corp., Kerr), which showed an intensity of 600 mW/cm<sup>2</sup>. The specimens were removed from the molds and their opposite sides were polymerized for the same length of time. After light polymerization was completed, the specimens were stored in distilled water at 37°C for 24 hours.

The top surfaces to be evaluated were polished with a series of finishing disks (FGM-Diamond PRO, FGM, Joinville, Santa Catarina,

**Table 1. Resin-based composites tested**

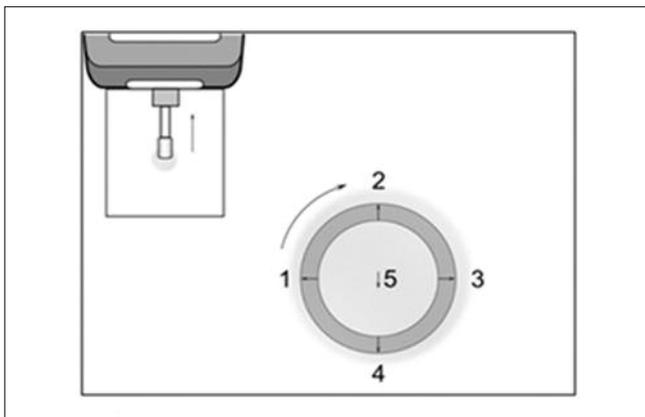
Material	Batch No.	Manufacturer	Filler type and particle range ( $\mu\text{m}$ )	Polymer	Filler volume (%)
Filtek Supreme	#2AB	3M Dental Products St Paul, MN USA	Nanomeric Zirconia/silica 20-75nm clusters 0,6–1.4 $\mu\text{m}$	Bis-GMA, Bis-EMA, UDMA, TEGDMA	59,5%
Esthet-X	#106161	Dentsply, Petrópolis, RJ, Brazil	Nanomeric Barium boron Fluoroaluminum Silicate glass Silica 0,04 – 1 $\mu\text{m}$	Bis-GMA, Urethane modified, EMA, TEGDMA	60%
Palfique Estelite	#E7191	Tokuyama Dental, Tokyo, Japan	Submicron Spherical Zirconia/ silica 0,1 – 0,3 $\mu\text{m}$	Bis-EMA, TEGDMA	71%
Filtek Z250	#2MH	3M Dental Products St Paul, MN USA	Microhybrid Zirconia/ silica 0.6 – 4.5 $\mu\text{m}$	Bis-GMA, Bis-EMA, UDMA, TEGDMA	60%

Bis-GMA: Bisphenylglycidylmethacrylate  
 Bis-EMA: Ethoxylated bisphenol dimethacrylate  
 UDMA: Urethane dimethacrylate  
 TEGDMA: Triethylene glycol dimethacrylate

Brazil) in a sequence from the highest to the lowest abrasiveness. Between abrasive disks, the specimens were rinsed with water to remove any loose particles resulting from use of the previous the finishing disk.

The specimens were transferred to a desiccator containing silica and maintained at 37°C for 24 hours and at  $\pm 23^\circ\text{C}$  for 1 hour. Then they were weighed to a precision of 0.001g using an analytical balance (AB204, Mettler Toledo, Switzerland). Three measurements of each specimen were taken and the mean was recorded as the initial weight.

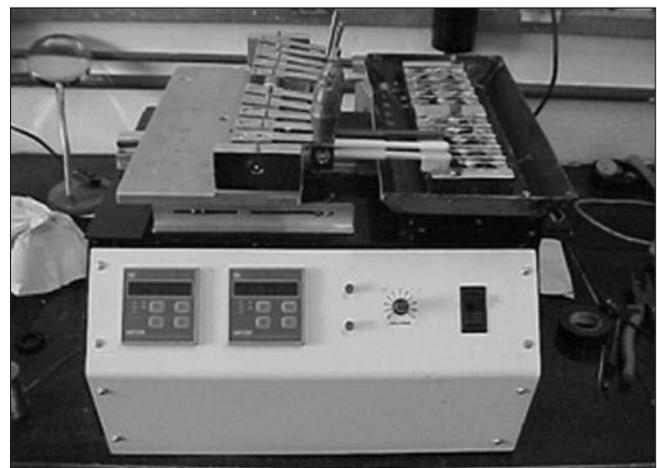
Surface roughness was assessed using a measuring instrument equipped with a 2  $\mu\text{m}$  radius diamond probe (SurfTest SJ 201-P, Mitutoyo Corp., Kawasaki, Japan), employing 0.25 mm cut-off length, and 1.25 mm measuring length. The surface roughness was characterized by Ra parameter (the mean value of the height of the surface profile above and below a center line throughout a prescribed sampling length roughness). Five tracings were performed at different locations for each specimen (Figure 1) and their mean corresponded to the initial surface roughness.



**Figure 1. Five tracings were performed at different locations for each specimen by profilometer**

Before the toothbrushing test, all specimens were thermal cycled 300 times at temperatures of 5 and 55°C. A dwell time of 60s in each water bath was used. Brushing was performed in a customized

toothbrushing machine (Figure 2). This produced horizontal back-and-forth movements of a soft nylon toothbrush (Colgate Palmolive – Division of Kolynos do Brasil Ltda, Osasco, São Paulo, Brazil) in water/dentifrice slurry at room temperature (23°C). The slurry consisted of a dentifrice (Colgate/calcio, Colgate Palmolive – Kolynos Ltda, Osasco, São Paulo, Brazil) and distilled water, in a ratio of 1:2 by weight. The brush load was 200g, stroke length 8 cm at a rate of the 100 cycles/minute. The test was performed for the duration of 10,000 cycles. A fresh slurry and toothbrush was used for every test.



**Figure 2. Toothbrushing abrasion testing machine**

The above-mentioned treatment was repeated four times, totalizing 1,200 cycles of thermal cycling and 40,000 cycles of toothbrushing. The thermal cycling simulated the exposure to the oral cavity<sup>6</sup>. After that, all specimens were rinsed in distilled water and transferred to a desiccator, following the same process described above, to determine final weight and surface roughness.

Data were analyzed using two-way ANOVA and the Tukey test to determine whether there were significant differences ( $p < 0.01$ ).

## Results

The mean weight and roughness values at baseline and after the toothbrushing abrasion are shown in Table 2 and 3, respectively.

No statistically significant difference was found in the weight between baseline and after toothbrushing. The resin composites Esthet X and Palfique Estelite presented decrease in weight after toothbrushing, while the Filtek Supreme and Filtek Z250 showed increase in weight.

Brushing significantly increased roughness ( $p < 0.01$ ) for all composites. Palfique Estelite, after brushing, was significantly smoother than the other resins.

**Table 2. Mean weight (grams  $\pm$ SD<sup>‡</sup>) in baseline and after cycles to different resins**

Material	Baseline	After cycles
Filtek Supreme	0.5790 $\pm$ 0.0053 <sup>Ab*</sup>	0.5801 $\pm$ 0.0054 <sup>Ab</sup>
Esthet X	0.6475 $\pm$ 0.0162 <sup>Aa</sup>	0.6425 $\pm$ 0.0161 <sup>Aa</sup>
Palfique Estelite	0.5476 $\pm$ 0.0114 <sup>Ab</sup>	0.5407 $\pm$ 0.0124 <sup>Ab</sup>
Filtek Z250	0.6398 $\pm$ 0.0218 <sup>Aa</sup>	0.6411 $\pm$ 0.0217 <sup>Aa</sup>

<sup>‡</sup> SD: Standard deviation

\* Superscript uppercase letters in each column indicate statistically significant differences ( $p < 0.01$ )

Superscript lowercase letters in rows indicate statistically significant differences ( $p < 0.01$ )

**Table 3. Mean surface roughness (grams  $\mu\text{m}^{\dagger} \pm$  SD<sup>‡</sup>) in baseline and after cycles to different resins**

Material	Baseline	After cycles
Filtek Supreme	0.15 $\pm$ 0.05 <sup>Aa</sup>	0.44 $\pm$ 0.03 <sup>Bb</sup>
Esthet X	0.18 $\pm$ 0.03 <sup>Aa</sup>	0.45 $\pm$ 0.07 <sup>Bb</sup>
Palfique Estelite	0.13 $\pm$ 0.03 <sup>Aa</sup>	0.28 $\pm$ 0.06 <sup>Ba</sup>
Filtek Z250	0.15 $\pm$ 0.03 <sup>Aa</sup>	0.57 $\pm$ 0.09 <sup>Bb</sup>

<sup>†</sup>  $\mu\text{m}$ : Micrometers

<sup>‡</sup> SD: Standard deviation

\* Superscript uppercase letters in each column indicate statistically significant differences ( $p < 0.01$ )

Superscript lowercase letters in rows indicate statistically significant differences ( $p < 0.01$ )

## Discussion

In the oral cavity, restorative material surfaces are subjected to a variety of factors that may alter the quality of the surface. Among others, oral hygiene procedures play a significant role. While toothbrushing with fluoridated toothpaste helps decrease the incidence of caries, the frequent use of prophylactic home procedures may have undesirable side effects, such as roughening the restorative material surfaces and dental hard tissues, thereby enhancing bacterial growth, increasing wear and discoloration of restorations, which may eventually impair their esthetic appearance<sup>7</sup>.

Both resin matrix and filler particle type or content are thought to affect surface conditions after toothbrushing, owing to the selective abrasion of the resin matrix and the dislodgment of filler particles after long-term exposure<sup>8</sup>.

Resin composites with different classifications have different wear mechanisms that are influenced mostly by the filler systems in the materials. In an effort to eliminate the problems of surface roughness, new tooth-colored materials have been produced, combining the proven composite and the innovative nanotechnology. Nanotechnology offers the possibility of designing materials with completely new characteristics<sup>9</sup>.

Nanocomposites have small primary filler particles, so that the filler and resin matrix are abraded off together during wear. For microhybrid composites whose average filler particle sizes are approximately 1  $\mu\text{m}$ , the relatively soft resin matrix is worn first, and the inorganic filler stands out above the surface<sup>10</sup>. Although the average cluster size of the nanocomposites is similar to that in microhybrid composite fillers, nanosized primary particles in the nanocomposites wear by breaking off individual primary particles. Thus, the resulting wear surfaces have smaller defects and better gloss retention<sup>5</sup>. This could explain the greatest surface roughness found for

Filtek Z250, which contains filler particles around 0.6 to 4.5  $\mu\text{m}$ . However, Heintze and Forjanic<sup>7</sup> (2005) found no correlation between the mean particle size and roughness after simulated toothbrushing in a regression analysis including 16 resin composites. On the other hand, better roughness values were observed for Z250 than for Filtek Supreme after 50,000 and 100,000 cycles of toothbrushing using profilometry, although the AFM analysis showed a smoother surface for Filtek Supreme<sup>11</sup>.

When there is not sufficient resin around the filler during the wear in resin composite with an irregular shaped filler, the individual filler particle is plucked out, leaving a void on the surface and a new wear cycle begins<sup>5</sup>. Whereas, a spherical filler might keep the surface smoother than an irregular shaped filler after it is exposed to the surface, justifying the smoother surface after toothbrushing abrasion found for Palfique Estelite, which has a spherical submicron filler. This is in agreement with results of previous study where the lowest surface roughness values, recorded from both AFM and stylus profilometry were found on Palfique Estelite compared with Filtek Supreme and Filtek Z250<sup>12</sup>. Besides, of the materials tested in another study, Palfique Estelite maintained the highest gloss during wear<sup>13</sup>.

Although the particle size given for Filtek Supreme is lower than Palfique Estelite fillers, it seems that the undefined volume fraction of Filtek Supreme nanoclusters, with the significantly greater size range, may affect the Supreme roughness values<sup>14</sup>. This suggests the significant benefit of submicron filler particles present on Palfique Estelite. The round shape of the fillers in Palfique Estelite may be an additional contributor factor for the lowest surface roughness observed<sup>12</sup>. These microspheres are responsible for the integrity and aesthetics of this composite, which offers the clinician high resistance to abrasion<sup>15</sup>.

Although the weight did not change significantly after toothbrushing, Palfique Estelite and Esthet X showed loss of weight after toothbrushing abrasion, whereas Filtek Supreme and Filtek Z250 showed a higher final weight than at baseline. Composite restoratives are not stable after polymerization and constantly interact with their environment. The principle interaction occurs with water which diffuses into the matrix causing two opposing phenomena. In some composites, water will leach out free, unreacted monomers and ions<sup>16</sup>. Elution of leachable components contributes to further shrinkage and loss in weight of the material. Conversely, hygroscopic absorption of water leads to swelling of the material and an increase in weight. The second option is what probably happened with Filtek Supreme and Filtek Z250 in the present study. Thermal cycling can benefit this phenomenon. Yap and Wee<sup>17</sup> (2002) found that cyclic temperature changes increase the water sorption of some composites.

A previous study examining the degree of hygroscopic expansion in resin-based composites showed an inverse relationship between filler-loading and water sorption<sup>18</sup>. As the volume of filler increases, the amount of water absorbed into the matrix is reduced. Filtek Supreme and Filtek Z250 have filler loading of approximately 59% by volume while Esthet X and Palfique Estelite have more than 60%. Moreover, for a fixed-volume fraction of filler, a finer particle size in the composite results in less interparticle spacing, more protection of the softer resin matrix, and less filler plucking, all of which lead to enhanced wear resistance of the material<sup>19</sup>.

In recent years, manufacturers have improved resin based composites by reducing particle size, increasing filler quantity, improving adhesion between the filler and the organic matrix, and using low-molecular-weight monomers to improve handling and polymerization<sup>20</sup>. Furthermore, particles have become spherical and smaller. The shape of prepolymerized filler particles has become more diverse, and various types of composites containing both prepolymerized and irregular-shaped filler particles have been developed<sup>21</sup>.

Since the influence of particles on the wear and consequently on the roughness of dental composites is complex, systematic studies are necessary for ascertaining whether nano-structured and spherical submicron composites truly offer the potential for improving the quality of composites.

## Conclusion

The toothbrushing and thermal cycling did not significantly influence the weight of composite resins. The microhybrid composite showed the highest surface roughness and spherical submicron filler composite the smoothest surface.

Therefore, the quantity of particles and their size and morphology influence the performance of composite resin after toothbrushing and thermal cycling.

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