

# Influence of different storage media on the microhardness of composite resins

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**ABSTRACT:** The longevity of composite resins is strongly influenced by the intrinsic characteristics of materials and by the environment to which they are exposed. The objective of this study was to evaluate the effect of different storage media on the microhardness of composite resins for direct restorations. The composite resins tested were: Durafill® VS – Heraeus Kulzer (microparticle), Filtek™ Z250 - 3M ESPE (microhybrid), and Filtek™ Supreme - 3M ESPE (nanoparticle). Twenty-five standard-format disc-shaped specimens were prepared for each material, and were randomly distributed into five groups (n=25). Specimens were kept in 5 different storage media (dry, 25%, 50%, and 100% alcohol solutions, and distilled water) at 37°C for one week. Knoop microhardness measurements were made with a microdurometer (10gf / 10sec) in two time intervals: immediately after specimen preparation, and again after seven-day storage in the different media. Data were analyzed using ANOVA and the Tukey test at 5% level of significance. The wet storage media had a distinct influence on the microhardness of composite resins evaluated. The nanoparticle resin showed greater loss of microhardness when submitted to alcoholic media in comparison with micro- and microhybrid resins, which was significant only when submitted to the 100% alcoholic medium. The composite resins evaluated showed good behavior in relation to the microhardness.

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## Introduction

At present, composite resins are the dental materials most commonly used in direct restorations, both in anterior and posterior teeth. Since their advent, and the development of etching techniques, composite resins have undergone several changes in their chemical composition to improve their mechanical properties, and consequently, their clinical performance (1).

Different types of composite resins are commercially available: microparticle, hybrid, microhybrid, and the more recent nanoparticulate resins, which combine the properties of earlier hybrid types with microparticle composites (2). Preference for composite resins is mainly due to four factors: high bond strength to the tooth structure; improved mechanical properties; their ability to mimic dental structures; and they require less wear of dental structure

precisely because of their good bond strength. (3)

Composite resins are comprised of an organic matrix (monomers, initiators, color modifiers, etc); an inorganic matrix (filler), and a bonding agent (silane) (4). Monomers are the main components of the organic matrix of composites (4). Among the most commonly used monomers are the bisphenol A-glycidyl methacrylate (BisGMA); urethane dimethacrylate (UDMA); triethylene glycol dimethacrylate (TEGMA), and ethylene glycol dimethacrylate (EGDMA) (5).

Water absorption by a resin composite may lead to its expansion and plasticization, leading to hydrolyzation of the silane (bonding agent), and causing microcracks in the restorative material (6). However, the addition of components to the resin matrix, such as UDMA that presents lower water sorption than BisGMA and TEGDMA, and has a protective effect by increasing the mechanical

strength of the restorative material, which may help to increase the longevity of the resin (7).

Therefore, the durability of composite resins is strongly influenced by the intrinsic characteristics of materials and by the environment to which they are exposed (8). Even in the absence of mechanical loads or abrasive forces, composite resin restorations are exposed to degradation resulting from chemical agents found in the saliva, food, beverages, and daily mouthwashes (9).

Alcoholic beverages and mouth rinses that have alcohol in their composition, have a low pH and can cause deterioration of the organic matrix of composite resins (10). According to Montenegro *et al.* (11), hardness can be defined as the ability of a material to resist wear and penetration. Understanding of these characteristics is essential for the indication and clinical longevity of restorative materials (11).

The microhardness of a composite resin is not only affected by the degree of conversion, but also by the filler particles present in the restorative material (12). Thus, Knoop hardness tests can contribute to evaluating the mechanical properties of resinous materials (13).

Therefore, to simulate the effect of different agents found in the oral cavity on resinous restorative materials, the objective of this *in vitro* study was to evaluate the effect of different storage media on the microhardness of three different types of composite resins with fillers consisting of inorganic particles of different sizes in their composition.

## Material and method

The restorative materials used in this study and their compositions are presented in Table 1. Twenty-five specimens were fabricated for each tested material (75 in total) and randomly allocated to 5 groups (N=5) according to the storage media (dry, 25%, 50% and 100% alcohol solutions, and distilled water - used as control).

## Specimen preparation

A 2 mm thick acrylic matrix with a central orifice 5 mm in diameter, 2 polyester strips and 2 glass microscope slides were used to fabricate the specimens (Fig. 1). The matrix was placed on a polyester strip on a glass slide, and filled with the composite resin in a single increment (Fig. 2). Then, another strip and slide were placed on top of the matrix, and the assembly was pressed for 5 sec to ensure a flat surface, with the necessary smoothness and parallelism for microhardness measurements (Fig. 3). The top slide and strip were then removed and the composite resin was polymerized for 20 sec with a halogen light device (Optilux® - Demetron Kerr, Orange, CA, USA) with irradiance of 460 mW/cm<sup>2</sup> (Fig. 4), according to the respective manufacturers' specifications.

After this, the specimens were polished in a metallographic polisher fitted with abrasive papers in a sequence of 320, 600, 1200 and 2000 grit (Extect Corp.) and a felt disc with Diamond solution (Buehler). Between one abrasive paper and the next, the specimens were cleaned in an ultrasound device.

## Microhardness measurement

After polymerization, the specimens were randomly allocated to their groups, and the bottom surface of each specimen (opposite to light-exposed surface) was marked with a high-speed carbide bur under air/water cooling, to facilitate identification during the microhardness readouts. Microhardness measurements, performed with a Knoop indenter, 10 gf/ 10 sec, fixed to a microhardness tester (HMV - 2000® - Shimadzu - Japan), were measured before the specimens were immersed in the different storage media. Five measurements were taken (one in the center, two above and two below) equally spaced 1 mm apart (Fig. 5). After the initial measurement procedure, specimens were stored in properly identified darkened plastic containers filled with their respective storage media and placed in an

**Table 1** – Restorative materials evaluated.

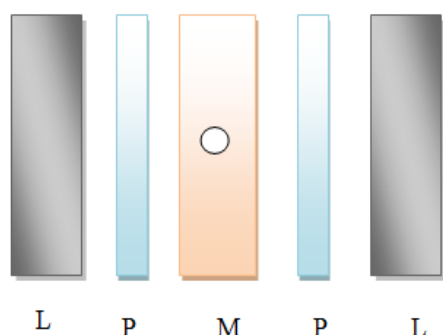
| Restorative Material | Manufacturer              | Composition                                                                             | Mean particle size                                                            | Color |
|----------------------|---------------------------|-----------------------------------------------------------------------------------------|-------------------------------------------------------------------------------|-------|
| Filtek™ Z250         | 3M ESPE, St Paul, MN, USA | <b>Organic matrix:</b><br>TEGDMA<br>UDMA<br>BisEMA                                      | Microhybrid<br>(0.60 μm)                                                      | A3    |
|                      |                           | <b>Inorganic particles:</b><br>Zirconia; Silica                                         |                                                                               |       |
| Filtek™ Supreme      | 3M ESPE, St Paul, MN, USA | <b>Organic matrix:</b><br>BisGMA<br>BisEMA<br>UDMA<br>TEGDMA                            | Silica nanoparticles<br>(5 – 20 μm)<br><br>Nanoagglomerates<br>(0.6 – 1.4 μm) | A3B   |
|                      |                           | <b>Inorganic particles:</b><br>Silica nanoparticles<br>Silica/zirconia nanoagglomerates |                                                                               |       |
| Durafill® VS         | Heraeus Kulzer            | <b>Organic matrix:</b><br>BisGMA<br>TEGDMA<br>UDMA                                      | Microparticles<br>(0.02 – 0.04 μm)                                            | A3    |
|                      |                           | <b>Inorganic particles:</b> Colloidal silica                                            |                                                                               |       |

incubator at  $37^{\circ}\text{C} \pm 2^{\circ}\text{C}$  for 7 days, when the second microhardness measurements were taken.

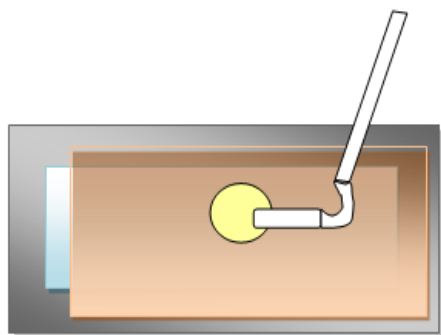
Taking into consideration that major changes in hardness tend to occur in the first 7 days (10) the period of 7 days was selected for this experiment in an attempt to simulate the brief and occasional contact of restorations with mouthwashes and beverages containing alcohol until the teeth are cleaned again (14).

### Statistical analysis

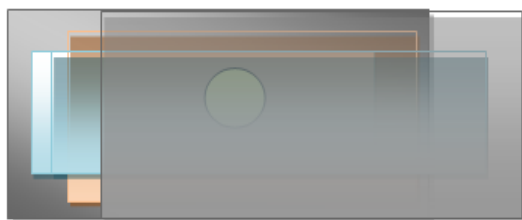
The data were submitted to two-way analysis of variance (ANOVA), followed by the Tukey test for comparison among groups at a 5% level of significance.



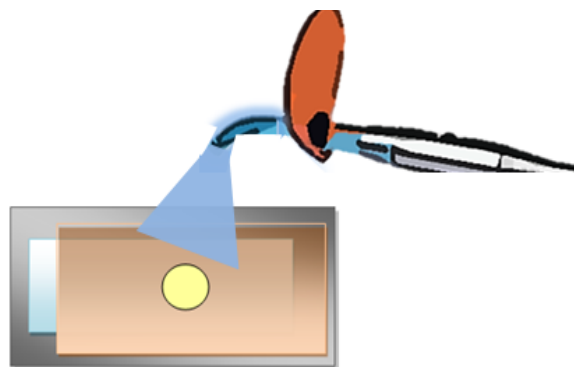
**Figure 1** – (M) 2 mm thick acrylic matrix with a central hole 5 mm in diameter; (G) glass slides; and (P) polyester strips.



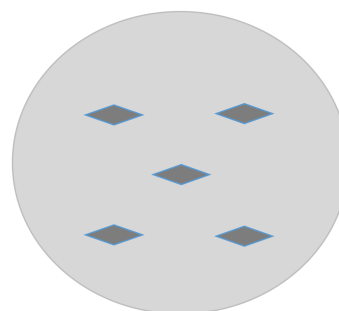
**Figure 2** – Acrylic matrix assembled on the polyester strip on the glass slide. Composite resin placement in the matrix in a single increment.



**Figure 3** – After resin placement, another polyester strip and glass slide were placed on top of the matrix and pressed for 5 sec.



**Figure 4** – After removing the top polyester strip and glass slide, the composite resin was polymerized for 20 sec.



**Figure 5** –Microhardness markings.

### Results

Mean Knoop microhardness values and standard deviations obtained immediately after fabrication (baseline); after 7-day storage, and the difference between the two assessments are presented in Table 2. In the comparison intragroup, the microhardness values of specimens immersed in the alcoholic solutions were lower than those of the specimens stored in distilled water. Except for all Durafill® VS (microparticle) and Filtek™ Z250 (microhybrid) specimens immersed in the 25% alcohol solution, all the other specimens stored in the alcoholic solutions lost over 20% of their baseline microhardness values (maximum acceptable microhardness loss) (15). Two-way ANOVA showed statistically significant differences among groups (Table 3).

Although Durafill® VS specimens stored in wet media showed a drop in hardness values, no statistically significant differences were observed among the different media investigated. Filtek™ Z250 specimens stored in the alcoholic solutions and dry showed no statistically significant differences when compared with specimens stored in distilled water.

Filtek™ Supreme (nanoparticulate) specimens stored in the alcoholic solutions showed no statistically significant differences among them; or when specimens kept in 25% and 50% alcoholic solutions and dry were compared with specimens stored in distilled water, however, specimens stored in 100% alcoholic solution showed significant difference when compared with specimens immersed in

**Table 2** – Mean Knoop microhardness (standard deviations) of tested specimens.

| Material                   | Medium          | Baseline          | After 7 days     | Difference       |
|----------------------------|-----------------|-------------------|------------------|------------------|
| <b>Filtek™<br/>Z250</b>    | A 100%          | 1124.700 (18.464) | 84.880 (4.925)   | -39.820 (18.266) |
|                            | A 50%           | 130.800 (4.549)   | 90.460 (21.670)  | -40.340 (22.634) |
|                            | A 25%           | 128.600 (7.469)   | 115.000 (8.396)  | -13.600 (7.503)  |
|                            | Distilled water | 1141.600 (14.707) | 129.600 (15.757) | -12.000 (24.124) |
|                            | Dry             | 1155.000 (19.506) | 160.200 (9.257)  | 5.200 (25.153)   |
| <b>Filtek™<br/>Supreme</b> | A 100%          | 1144.600 (13.030) | 84.740 (3.902)   | -59.860 (14.198) |
|                            | A 50%           | 142.200 (6.978)   | 104.900 (8.919)  | -37.300 (12.920) |
|                            | A 25%           | 136.000 (6.519)   | 86.700 (5.556)   | -49.300 (11.618) |
|                            | Distilled water | 140.000 (7.483)   | 123.000 (7.968)  | -17.000 (12.668) |
|                            | Dry             | 1138.200 (13.479) | 145.800 (15.690) | 7.600 (21.161)   |
| <b>Durafill® VS</b>        | A 100%          | 52.280 (0.443)    | 42.420 (1.522)   | -9.860 (1.346)   |
|                            | A 50%           | 51.700 (0.620)    | 44.160 (0.630)   | -7.540 (0.814)   |
|                            | A 25%           | 54.120 (2.301)    | 44.100 (1.394)   | -10.020 (2.633)  |
|                            | Distilled water | 52.200 (1.454)    | 42.480 (2.505)   | -9.720 (1.616)   |
|                            | Dry             | 52.720 (3.141)    | 54.860 (1.045)   | 2.140 (3.648)    |

A: Alcoholic solution.

**Table 3** – Two-way analysis of variance (ANOVA).

| Sources of Variation | Sum of squares    | Degree of freedom | Mean Square | “F”     | p        |
|----------------------|-------------------|-------------------|-------------|---------|----------|
| Resin                | 7321.1144         | 2                 | 3660.5572   | 16.7835 | 0.000000 |
| Medium               | 15515.4819        | 4                 | 3878.8705   | 17.7844 | 0.000000 |
| Resin x Medium       | 7226.0509         | 8                 | 903.2564    | 4.1414  | 0.000549 |
| <b>Total</b>         | <b>43148.9512</b> | <b>74</b>         |             |         |          |

distilled water, which showed a considerably higher mean microhardness value (Table 4).

Intergroup comparison showed no statistically significant differences between specimens stored in distilled water and those stored dry. (Table 5).

## Discussion

Composite matrices are susceptible to softening by organic acids and the various constituents of foods and beverages (16). Moreover, it is important to consider that there are various types of commercially available alcohol-containing mouthwashes with different compositions and indications, which are increasingly being used for oral hygiene (17).

As many patients routinely use these products, it is essential to conduct studies to investigate their likely effect on the mechanical properties of direct composite resin restorations (9).

The aqueous ethanol solutions used in the present study, simulated the effect of resin composite coming into contact with beverages or mouthwashes containing alcohol, and distilled water simulated the humid oral medium provided by saliva and water (14).

The results of the present research were in agreement with the findings of other studies that showed loss of microhardness of different composites when immersed in alcohol solutions (14,18). In this study, the microhardness values of nanoparticulate resin were reduced when the samples were submitted to alcoholic media in comparison with the values of microparticle and microhybrid resins.

According to Brandrup *et al.* (19), BisGMA-based resin polymers can undergo plasticization by chemical products and other solvents that present solubility parameters ranging between 18.2 and 29.7 MPa, such as ethanol (22 Mpa) (19). Vouvoudi and Sideridou (20) indicated that the plasticizing effect of alcohol gradually decreased with the presence of UDMA. In this study, the samples made of Durafill® VS were not severely affected by the different media, probably due to the larger quantity

**Table 4** – The Tukey test at a 5% significance level - individual comparisons between the different media and the distilled water used as control.

| Restorative Materials | Alcoholic solution 100%<br>X<br>Distilled water | Alcoholic solution 50%<br>X<br>Distilled water | Alcoholic solution 25%<br>X<br>Distilled water | Dry<br>X<br>Distilled water |
|-----------------------|-------------------------------------------------|------------------------------------------------|------------------------------------------------|-----------------------------|
| Filtek™ Z250          | 27.820                                          | 28.340                                         | 1.600                                          | 17.200                      |
| Filtek™ Supreme       | *42.860                                         | 20.300                                         | 32.300                                         | 24.600                      |
| Durafill® VS          | 0.140                                           | -2.180                                         | 0.300                                          | 11.860                      |

\*Above the critical value of 33.0354358. A: Alcoholic solution.

**Table 5** – The Tukey test results for multiple comparisons.

| Medium                  | Materials    |              |                 |
|-------------------------|--------------|--------------|-----------------|
|                         | Durafill® VS | Filtek™ Z250 | Filtek™ Supreme |
| Alcoholic solution 100% |              |              |                 |
| Alcoholic solution 50%  |              |              |                 |
| Alcoholic solution 25%  |              |              |                 |
| Distilled water         |              |              |                 |
| Dry                     |              |              |                 |

The bars represent the group.

of UDMA in Durafill® VS composition, necessary to ensure its adequate viscosity (21).

For Almeida *et al.* (22), the TEGDMA solubility parameter is about 15% lower than that of BisGMA. Thus, it may be speculated that increased TEGDMA content in the resin matrix would interfere in the softening effect promoted by ethanol. This may explain the significant loss of hardness observed in the Filtek™ Z250 and Filtek™ Supreme specimens stored in the alcoholic solutions. It is important to point out that several distilled spirits and brands of mouthwashes have alcohol concentrations of about 50% and 25%, respectively, which could lead to the degradation of resin materials, (22) as shown in the present study.

Previous reports have shown greater change in the hardness values of composites in the first 7 days of exposure to media, and for this reason, specimens were kept in the alcoholic media for one week in the present study (14).

Yesilyurt *et al.* (14) showed significant loss of microhardness in methacrylate-based composites, among them Supreme and Z250 resins, when submitted to 75% alcohol solutions for 7 days. Whereas, in the present study, significant reduction in microhardness values was shown after exposure to 100% alcohol solutions in the same 7-day time interval. It is important to point out that beverages such as Vodka may contain a minimum alcohol content of 96% (23).

An *in vitro* study by Jyothi *et al.* (22) on the effect of mouthwashes on a nanoparticulate resin composite demonstrated that two brands (Listerine® and Periogard®) containing 21.6% and 11.6% of alcohol in water (w/v), respectively, promoted statistically significant reductions in hardness when compared with another brand (Colgate Plax®) with 7.2% w/v. This absence of difference was probably due to the low percentage of alcohol in Colgate Plax®.

Delaviz *et al.* (24) suggested that saliva could modify the effect of mouthwashes on restorative materials, by

diluting them, causing a reduction in the softening effect, and by promoting a protective effect. Considering that the absence of saliva was a limitation of this *in vitro* study, further *in situ* researches are necessary to direct investigations into the tendency of the effect of the conditions proposed in the present study.

Weak acids (acetic, lactic and propionic), produced by bacterial metabolism, have a solubility parameter close to that of alcohol, which can affect the durability of direct resin restorative materials. This is a problem, however, that can be circumvented by routine oral hygiene procedures performed by patients (14,24).

The use of distilled water in the present study, was an attempt to simulate the moist oral environment provided by the saliva. However, water sorption has been shown to be the main reason for the onset and propagation of cracks, surface flaws, debonding of filler particles, and interfacial displacement over time (25). The reduction in surface hardness of specimens stored in distilled water may be attributed to the hydrophilic characteristics of BisGMA, UDMA and BisEMA (25).

Microhardness is related to the capacity of composite resins to resist abrasive wear and surface degradation, which are influenced by surface smoothness. According to Kumar *et al.* (26), microhybrid composites with round particles could show improved hardness when compared with those containing irregular fillers due to improved surface smoothness.

It is important to remember that when hardness decreases and surface roughness increases, microorganisms such as *S. mutans*, *S. oralis* and *A. naeslundii*, are capable of adhering more easily and firmly to resinous materials, leading to a more acidic environment (27). Apart from affecting the longevity of composite restorations, the buildup of acid plaque can also result in color change, secondary caries, and periodontal diseases (27).

Knowledge about the vulnerability of esthetic restorative materials in the oral environment is fundamental to enable professionals to be aware when selecting materials, to base their choice on obtaining the best clinical performance of restorations.

## Conclusion

The results of this study showed that contact with alcohol solutions of various concentrations and low pH reduced the microhardness of the resins tested and this loss was proportional to the concentration of alcohol used.

## Resumo

NUNES, Margareth Calvo Pessutti; PORCELLI, Ilma Carla de Souza; FRANCO, Eduardo Batista. **Influência de diferentes meios de armazenagem na microdureza superficial de resinas compostas.** Oral Sci., jan/dez. 2015, vol. 7, nº 1, p. 7-13.

A durabilidade das resinas compostas é fortemente influenciada pelas características intrínsecas dos materiais e ambiente a que estão expostas. O objetivo deste estudo foi avaliar o efeito de diferentes meios de armazenagem na microdureza superficial de resinas compostas de uso direto. Foram testadas uma resina de micropartícula (Durafill® VS - Heraeus Kulzer), outra microhíbrida (Filtek™ Z250 - 3M ESPE) e a de nanopartícula (Filtek™ Supreme - 3M ESPE). Confeccionaram-se 25 corpos de prova para cada material, em formato de discos padronizados, distribuídos em cinco grupos de forma aleatória (n = 25). As amostras foram mantidas em 5 diferentes meios de armazenagem (seco, 25%, 50% e 100% de soluções de álcool, e água destilada) a 37°C durante uma semana. As mensurações de microdureza Knoop, utilizando o aparelho microdurômetro (10gf/10seg), foram realizadas em dois momentos: imediatamente após a preparação das amostras e após sete dias de armazenagem. Os dados foram analisados usando ANOVA e teste de Tukey em nível de significância de 5%. Os meios de armazenagem úmidos influenciaram de forma distinta na microdureza das resinas compostas avaliadas. Houve maior perda de microdureza na resina nanoparticulada quando submetida aos meios alcoólicos em comparação às resinas de micropartícula e microhíbrida, sendo significativa apenas quando submetida ao meio alcoólico de 100%. As resinas compostas avaliadas mostraram bom comportamento em relação à microdureza.

**PALAVRAS-CHAVE:** Resinas Compostas, Álcool, Armazenamento, Teste de Dureza.

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