Original Article

Microhardness of different esthetic restorative materials: Evaluation and comparison after exposure to acidic drink

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ABSTRACT

Background: Acidic beverages, such as soft drinks (orange juice and cola), can produce erosion of resin composites. The aim of this in vitro study was to evaluate the effect of immersion in acidic drink on the Vickers microhardness (VK) of different esthetic restorative materials (one nanohybrid Ormocer-based composite, one nanoceramic composite, one nanofilled composite, and one microfilled hybrid composite).

Materials and Methods: In this in vitro study, thirty specimens of each esthetic restorative material were divided into three subgroups (n = 10): specimens of group 1 were used as control, specimens of group 2 were immersed in 50 ml of acidic drink for 1 day, specimens of group 3 were immersed in 50 ml of acidic drink for 7 days. Data were analyzed by Shapiro–Wilk test to assess the normality of the distributions followed by nonparametric Kruskal–Wallis analysis of variance and Mann–Whitney U-test comparison test among groups. A significant level of \( \alpha = 0.05 \) was set for comparison between the groups.

Results: Mann–Whitney U-test showed that each material showed lower microhardness values after immersion in acidic solution \( (P < 0.05) \). Paired t-test confirmed that microhardness for each composite did not change after immersion in distilled water (Control group) \( (P > 0.05) \). Significant changes were registered for all restorative materials after immersion in acidic solution for 1 day and 7 days \( (P < 0.05) \).

Conclusion: The Filtek Supreme XTE, a nanofilled composite, and Admira Fusion, a nanohybrid ormocer-based composite, showed the best behavior. The Ceram X Universal (nanoceramic composite) although reached lower hardness values than the previous materials, but resisted well to the 1 week immersion in soft-drink. Finally, the Gradia Direct achieved the most disappointing results: Low microhardness values are justified by the nature of its filling (microfilled hybrid composite).

Key Words: Acidic, drink, erosion, hardness, restorative materials

INTRODUCTION

Resin-based composites are used worldwide in dentistry, mainly because of their esthetic quality and good physical properties. Since resin composites were first developed, many efforts have been made to improve the clinical behavior of this restorative material.

Resin composites have been classified according to various characteristics, such as size, content, and filler...
Correlations between bacterial adhesion and various surface characteristics (chemical composition, surface energy, surface roughness, and presence of functional groups on the surface) have been intensively investigated in an attempt to reduce bacterial adhesion through surface modification.\textsuperscript{[12,13]}

The aim of this \textit{in vitro} study was to evaluate the effect of immersion in acidic drink on the Vickers microhardness (VK) of different esthetic restorative materials (one nanohybrid Ormocer-based composite, one nanoceramic composite, one nanofilled composite, one microfilled hybrid composite).

\textbf{MATERIALS AND METHODS}

\textbf{Specimen preparation}

In this \textit{in vitro} study, one nanohybrid Ormocer-based composite (Admira Fusion, Voco, Cuxhaven, Germany), one nanoceramic composite (Ceram X Universal, Dentsply De Trey, Konstanz, Germany), one nanofilled composite (Filtek Supreme XTE, 3M ESPE, St Paul, MN, USA), and one microfilled hybrid composite (Gradia Direct, GC Corporation, Tokyo, Japan); for each brand, the A3 Vita shade was selected [Table 1]. All materials were polymerized according to manufacturers’ instructions into silicon rings (height 2 mm; internal diameter 6 mm; and external diameter 8 mm) to obtain specimens of identical size. Cavities of these rings were slightly overfilled with the material, covered with Mylar strip (Henry Schein, Melville, NY, USA), pressed between glass plates and polymerized for 40 s on each side using a curing unit (Celalux II, Voco, Cuxhaven, Germany). One light polymerization mode was used for each material standard: 1000 mW/cm\textsuperscript{2} for 40 s. The intensity of the light was verified with a radiometer (SDS Kerr, Orange, CA, USA). The light was placed perpendicular to the specimen surface, at distance of 1.5 mm. The upper surface of each specimen was then polished with fine and superfine polishing disks (Sof-Lex Pop On; 3M ESPE, St Paul, MN, USA) to simulate clinical conditions.

Thirty cylindrical specimens of each material were prepared in this manner. After polymerization and during the experimentation, the specimens were stored in distilled water at 37°C and 100% humidity before performing the Vickers hardness test.

\textbf{Immersion in acidic drink}

Specimens of each esthetic restorative material were divided into three subgroups (\(n = 10\)): specimens
of group 1 were used as control, specimens of group 2 were immersed in 50 ml of acidic drink (Coca-Cola/Coca-Cola Company, Milano, Italy) for 1 day, specimens of group 3 were immersed in 50 ml of acidic drink (Coca-Cola/Coca-Cola Company, Milano, Italy) for 7 days.

Surface microhardness measurements
The VK of the enamel surface was determined with a microhardness tester (Isoscan HV 1OD, LTF SpA, Antegnate, BG, Italy) using a Vickers diamond indenter and a 100 g load applied for 20 s and a 40x objective lens at the baseline time and after the experimental stage. Five VK readings were recorded for each sample surface. Five indentations equally placed over a circle and each no closer than 0.5 mm to the adjacent indentations were made on the surface of each specimen.

For a given specimen, the five hardness values for each surface were averaged and reported as a single value. The diagonals' length of the indentations was measured by a built-in scaled microscope, and a Vickers values were converted into microhardness values. Microhardness was obtained using the following equation: \( VK = 1.854 \frac{P}{d^2} \), where VK is Vickers microhardness in kgf/mm², \( P \) is the load in kgf and \( d \) is the length of the diagonals in mm.

Statistical analysis
The data were analyzed using Stata 12 software (Stata, College Station, Texas, USA). Descriptive statistics including the mean, standard error of mean, and minimum and maximum values were calculated for all groups. Statistical analysis of the results of microhardness testing included Shapiro-Wilk test to assess the normality of the distributions followed by nonparametric Kruskal-Wallis analysis of variance (ANOVA) and Mann-Whitney U comparison test among groups. A significant level of \( \alpha = 0.05 \) was set for comparison between the groups. For each specimen microhardness before immersion was compared with a paired \( t \)-test with microhardness after immersion to define the amount of erosion.

RESULTS
Descriptive statistics of the microhardness values are reported in Table 2 and displayed in Figure 1. Baseline values are significantly different for each composite (\( P < 0.05 \)). Data are not normally distributed as confirmed by Shapiro-Wilk test (\( P < 0.05 \)). Kruskal–Wallis ANOVA confirmed significant differences in microhardness Vickers values among the three experimental groups (\( P < 0.05 \)). Mann–Whitney U test showed that, except for control group, each composite showed lower microhardness values after immersion in acidic solution (\( P < 0.05 \)). The lowest values were registered after immersion in acidic solution for 1 week (\( P < 0.05 \)). Paired \( t \)-test confirmed that microhardness for each composite did not change after immersion in distilled water (control group) (\( P > 0.05 \)). Significant changes were registered for all restorative materials after immersion.
DISCUSSION

The use of resin-based restorative materials in dentistry has substantially increased over the past few years because of their good esthetic appearance, improvements in formulations, ease of handling, and ability to establish a bond to dental hard tissues.\textsuperscript{[14-16]} To be clinically successful, restorative materials are required to have long-term continuousness,\textsuperscript{[17]} a quality which is strongly influenced not only by the intrinsic characteristics of the materials but also by the environment to which they are exposed to.\textsuperscript{[15,18]} However, the oral cavity is a complex, aqueous environment where the restorative material is in contact with saliva. In addition, other factors such as low pH due to acidic foods and drinks may influence the material’s mechanical and physical characteristics.\textsuperscript{[16]}

The consumption of sports and energy drinks has gained high popularity among the young population in recent years, but they are being widely consumed by the general population.\textsuperscript{[16]}

In addition to erosion of tooth hard tissues, the erosion of restorative materials has also received attention from researchers.\textsuperscript{[19-21]}

It has been shown that erosion induced substance loss, surface degradation, and reduced abrasive-resistance of restorative materials.\textsuperscript{[22]}

Although restorative materials are less susceptible to erosive attacks compared to enamel, the erosive attack can induce, at least to some extent, the degradation of the matrix and fillers of restorative materials.\textsuperscript{[23]}

Even though a great variety of substances may be present at the oral environment, water, saliva, acids, bases, salts, and alcohols have been related to the reduction of hardness, flexural strength, and flexural modulus properties.\textsuperscript{[23,24]}

In addition, the biofilm accumulated over the restoration can produce acidic substances that may imply surface degradation, leading to the material’s softening and surface roughening\textsuperscript{[22]} with regard to these acidic substances, the lactic, propionic, and acetic acids are commonly present in acidic solution for 1 day and 7 days ($P < 0.05$). Figures 2-5 show the gradual surface changes and indentations of the different esthetic restorative materials tested.

### Table 2: Mean±standard deviation surface microhardness values of tested restorative materials before and after immersion in solutions

| Material          | Test period | Solution    | Micro-hardness Kgf/mm$^2$ |
|-------------------|-------------|-------------|---------------------------|
| Admira Fusion     | 1 day       | Distilled water | 64.7±0.9$^a$              |
|                   | 1 day       | Coca Cola   | 60.7±0.5$^b$             |
|                   | 1 week      | Coca Cola   | 57.3±0.5$^c$             |
| Ceram.X Universal | 1 day       | Distilled water | 57.1±0.4$^d$             |
|                   | 1 day       | Coca Cola   | 56±0.2$^e$               |
|                   | 1 week      | Coca Cola   | 53.9±0.5$^e$             |
| Filtek Supreme XTE| 1 day     | Distilled water | 80.2±1.5$^f$             |
|                   | 1 day       | Coca Cola   | 74.5±0.6$^f$             |
|                   | 1 week      | Coca Cola   | 72.2±0.6$^f$             |
| Gradia Direct     | 1 day       | Distilled water | 32.9±1.1$^g$             |
|                   | 1 day       | Coca Cola   | 28.5±0.8$^g$             |
|                   | 1 week      | Coca Cola   | 26.1±0.2$^{2h}$          |

Within each material group for each solution the same capital letters in the same column represent statistical insignificance.
found in the oral environment and they are used as storage solutions for screening accelerated hydrolysis phenomena of composite resins and increase of hygroscopic expansion of Bis-GMA-based materials. Chemical substances may affect more actively the organic matrix of composites, but the type, size, and concentration of fillers may also influence the material’s resistance to degradation.

This in vitro study focused on Vickers microhardness (VK) of different esthetic restorative materials after exposure to acidic drink. The material’s hardness is one of the most important properties and correlates well with compressive strength, resistance to intraoral softening, and degree of conversion. A low surface hardness value is largely related to inadequate wear resistance and proclivity to scratching, which can compromise fatigue strength and lead to failure of the restoration.

In the current study, Filtek Supreme XTE registered the highest values of microhardness but respectively showed quite higher percentage loss of microhardness after 1 week immersion in soft-drink. Conversely Gradia Direct registered the lowest values and after a week immersion in the acidic drink lost about 20% of its initial hardness. The nanoceramic composite (Ceram X Universal) presented initial
hardness values which amount to 57 HV (less than the initial values of Admira and Filtek Supreme) but resisted acid attack successfully. Finally, the nanohybrid Ormocer-based composite (Admira Fusion), offered good initial values of microhardness and did not show a significant loss of microhardness after 1 week immersion in soft drink.

In a clinical environment, a material’s decrease of hardness may contribute to its deterioration.[29] Under in vivo conditions, composite resin materials may be exposed either discontinuously or continually to chemical agents found in saliva, food, and beverages.[18] Consequently, in the short- or long-term, these conditions may have a different deleterious effect on the polymeric network, modifying its structure physically and chemically.

**CONCLUSION**

Despite the limitations of this in vitro study, the composite that showed the best behavior both initial and after the acid attack is the Filtek Supreme XTE (nanofilled composite), followed by Admira Fusion (nanohybrid Ormocer-based composite). The Ceram X Universal (nanoceramic composite) although reached lower hardness values than the previous materials, but resisted well to the 1 week immersion in soft-drink. Finally, the product that achieved the most disappointing results is the Gradia Direct: Low microhardness values are justified by the nature of its filling (microfilled hybrid composite). Further investigations may be required to evaluate the effect of acidic solutions on mechanical and surface properties of esthetic restorative materials containing different types, sizes, and content of fillers.

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**Conflicts of interest**

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or nonfinancial in this article.

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