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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

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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

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type, and the physical and mechanical properties of the materials.[1] Nanotechnology, known as molecular nanotechnology, is the production of functional materials and structures, at a range of 0.1–100 nm, by various physical and chemical methods.[2] A nanohybrid is a hybrid resin composite with nanofiller in a prepolymerized filler form, whereas nanofill is a composite resin that is composed of both nanomers and nanoclusters.[3] The term Ormocer is an abbreviation for Organically Modified Ceramics. Ormocers were initially used together with dimethacrylates, but a recent material formulated with a pure-Ormocer-based resin matrix has been developed.[4] An Ormocer is a hybrid molecular structure. This combines organic and inorganic components at nanoscopic scale through the sol-gel method, and the main characteristic of this type of material is the incorporation of organic groups linked to the inorganic backbone.[5] Ormocer materials contain inorganic-organic copolymers in addition to the inorganic silanated filler particles. Ormocers are described as three-dimensionally cross-linked copolymers. The Ormocer matrix is a polymer even before light curing. It consists of ceramic polysiloxane, which has low shrinkage as against the organic dimethacrylate monomer matrix seen in composites.[6]

Acidic beverages, such as soft drinks (orange juice and cola), can produce erosion of resin composites.[7,8] The surface degradation of resin materials is related to the content and distribution of the fillers, the composition of the matrix resin, and the effect of silane surface treatment on the fillers.[8,9] Although the mechanical properties of these materials have been improved substantially, their antibacterial properties are still limited.[10,11] The bacterial accumulation on the surfaces of restorative materials can provide the bacterial source leading to the development of secondary caries and periodontal diseases. Bacterial accumulation is highly dependent on the characteristics of the material surface, and the roughness of resin composites can influence the oral biofilm adherence.[11] Biofilm formation is a stepwise process initiated by adhesion of planktonic bacteria onto the surface of a tooth or other structures in the oral cavity.

This process progresses from colonization and coadhesion, through growth and maturation, to detachment and spread of the microorganisms from the biofilm.[12] The physical and chemical properties of the surface affect the feasibility of bacterial infection. 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.[12,13]

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, one microfilled hybrid composite).

MATERIALS AND METHODS

Specimen preparation

In this *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^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.

Immersion in acidic drink

Specimens of each esthetic restorative material were divided into three subgroups (n = 10): specimens

Table 1: Esthetic restorative materials used in this study

Material	Composition	Type	Filler content, percentage (w/w) (v/v)	Lot#
Admira Fusion (Voco, Cuxhaven, Germany)	Matrix: Resine Ormocer Filler: Silicon oxide nano filler, glass ceramics filler (1 μm)	Nanoybrid Ormocer-based composite	84 (w/w) 69 (v/v)	1508065
Ceram.X Universal (Dentsply De Trey, Konstanz, Germany)	Matrix: Methacrylate modified polysiloxane, dimethacrylateresin, fluorescent pigment, UV stabilizer, stabilizer, camphorquinone, ethyl-4 (dimethylamino) benzoate, iron oxide pigments, titanium oxide pigments, aluminum-sulfo-silicate pigments Filler: Barium-aluminum-borosilicate glass (1.1-1.5 μm) methacrylate functionalized silicon dioxide nano filler (10 nm)	Nanoceramic composite	76(w/w) 57 (v/v)	1407000927
Filtek supreme XTE (3M ESPE, St Paul, MN, USA)	Matrix: Bis-GMA, TEGDMA, UDMA, bisphenol A polyethylene glycol diether dimethacrylate Filler: silica nanofillers (5-75 nm) zirconia/silica nanoclusters (0.6-1.4 μm)	Nanofilled composite	78.5 (w/w) 59 (v/v)	N595296
Gradia direct (GC Corporation, Tokyo, Japan)	Matrix: UDMA, dimethacrylate camphorquinone Filler: Fluoroaluminosilicate glass silica powder	Microfilled hybrid composite	73 (w/w) 64 (v/v)	140127A

Bis-GMA: Bis-phenol A diglycidyl methacrylate; TEGDMA: Triethylene glycol dimethacrylate; UDMA: Urethane dimethacrylate; UV: ultra-violet light

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: $VK = 1.854\ P/d^2$, where VK is Vickers microhardness in kgf/mm^2, 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

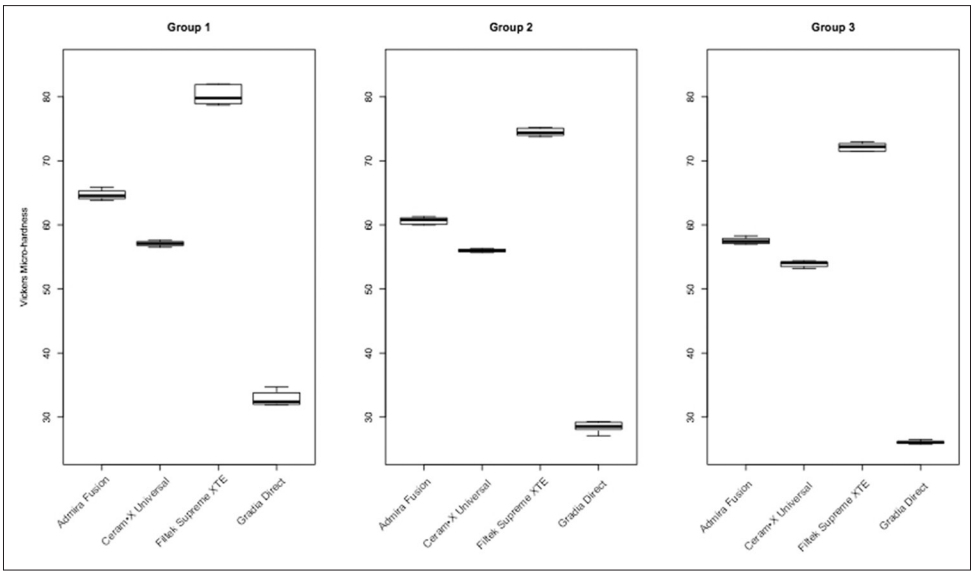

Figure 1: Box-plots for Vickers microhardness of material tested for each experimental group. Bold horizontal line represents median.

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/mm²
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[F]
Filtek Supreme XTE	1 day	Distilled water	80.2±1.5[G]
	1 day	Coca Cola	74.5±0.6[H]
	1 week	Coca Cola	72.2±0.6[I]
Gradia Direct	1 day	Distilled water	32.9±1.1[L]
	1 day	Coca Cola	28.5±0.8[M]
	1 week	Coca Cola	26.1±0.2[N]

Within each material group for each solution the same capital letters in the same column represent statistical insignificance

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.

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.[14-16] To be clinically successful, restorative materials are required to have long-term continuousness,[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.[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.[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.[16] In addition to erosion of tooth hard tissues, the erosion of restorative materials has also received attention from researchers.[19-21] It has been shown that erosion induced substance loss, surface degradation, and reduced abrasive-resistance of restorative materials.[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.[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.[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[25] with regard to these acidic substances, the lactic, propionic, and acetic acids are commonly

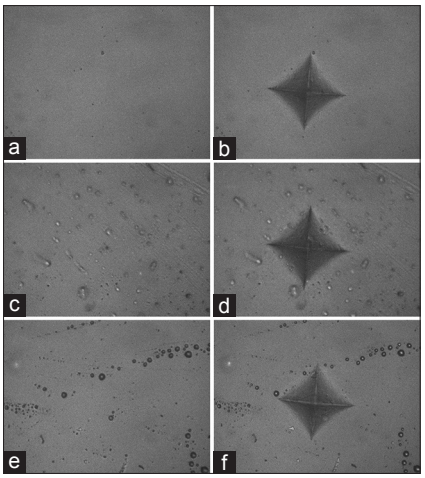

Figure 2: Photomicrographs of Admira Fusion before and after immersion in acidic drink for 1 and 7 days (×40). (a and b) Before immersion; (c and d) after immersion in acidic drink for 1 day, (e and f) after immersion in acidic drink for 7 days.

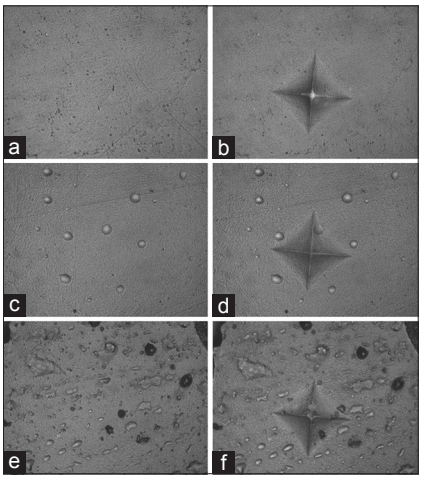

Figure 3: Photomicrographs of Ceram X Universal before and after immersion in acidic drink for 1 and 7 days (×40). (a and b) Before immersion; (c and d) after immersion in acidic drink for 1 day, (e and f) after immersion in acidic drink for 7 days.

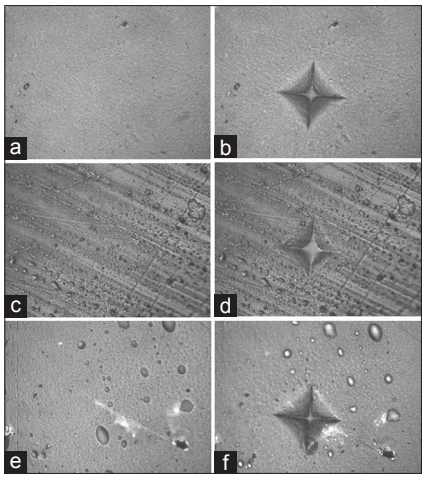

Figure 4: Photomicrographs of Filtek Supreme XTE before and after immersion in acidic drink for 1 and 7 days (×40). (a and b) Before immersion; (c and d) after immersion in acidic drink for 1 day, (e and f) after immersion in acidic drink for 7 days.

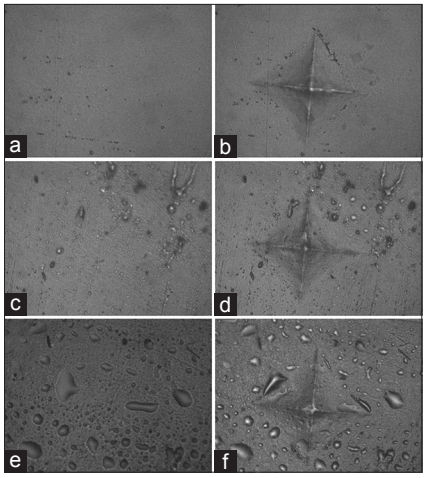

Figure 5: Photomicrographs of Gradia Direct before and after immersion in acidic drink for 1 and 7 days (×40). (a and b) Before immersion; (c and d) after immersion in acidic drink for 1 day, (e and f) after immersion in acidic drink for 7 days.

found in the oral environment and they are used as storage solutions for screening accelerated hydrolysis phenomena of composite resins[26] 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.[24]

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.[14,27] A low surface hardness value is largely related to inadequate wear resistance[28] and proclivity to scratching, which can compromise fatigue strength and lead to failure of the restoration.[15]

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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