EFFECT OF DIFFERENT BEVERAGES ON MICROHARDNESS OF ZIRCONIA REINFORCED GLASS Ionomer AND MICRO-HYBRID RESIN COMPOSITE

Heba Bahgat* and Hadeel Farouk**

ABSTRACT

Objective: This study aimed to evaluate the influence of commonly used beverages, on surface microhardness of Zirconia reinforced glass ionomer (Zirconomer improved) and microhybrid resin-based composite (Filtek Z250).

Materials and methods: A total of 40 disk-shaped specimens were fabricated (20 discs from each type of material). Ten Specimens from each material were immersed in Instant Coffee and the other ten Specimens were immersed in Cola. After initial microhardness recording, each group was subject to thermocycling and then microhardness was re-assessed (n= 10).

Results: It was observed that each of restorative material type, thermocycling, and immersion solution, had a statistically significant effect on mean microhardness of both tested materials irrespective of the other tested variables.

Conclusion: Resin composite showed significant higher hardness values when compared to Zirconia reinforced glass ionomer. There was a significant decrease in hardness in both materials after thermocycling, with a significant influence of soft drinks more than instant coffee.

KEY WORDS: Microhardness, Zirconia reinforced glass ionomer, micro-hybrid resin composite, daily beverages

INTRODUCTION

The goal of using any restorative material is to substitute the biological, functional and esthetic harmony of the lost tooth structure [1]. Advancement of restorative materials is imperative for better delivery of treatment. Thus the newer materials should provide significantly better properties than its predecessors [2].

Glass ionomer cement (GIC) was one of the first tooth-colored restorative materials introduced in the
dental field by Wilson and Kent way back in 1972[3]. In the last decade, there has been several innovative additions to enhance the properties of GIC. Unlike the early glass ionomers, these newer systems are easy and more practical to use [4].

Addition of fillers like silver, gold, and stainless steel powders to conventional glass ionomer has been investigated [5, 6]. These reinforced glass-ionomers exhibit reduced abrasion, but they have poor aesthetics. Zirconium and its oxide were used to improve the strength of glass-ionomers due to their good dimensional stability and toughness [7]. It was reported that many mechanical properties of glass-ionomer improved by mixing the powder with bioceramics [8, 9]. It can enhance the flexural strength of the demineralized dentin by remineralization [10].

Zirconia (ZrO2) reinforced GIC (Zirconomer) is one of the recent additions to the GIC family [11]. Their structural integrity has been attributed to the inclusion of zirconia fillers in the glass component thereby imparting better mechanical strength and dimensional stability [12,13]. Zirconomer Improved also developed as a reliable and durable self-adhesive tooth-colored zirconia reinforced posterior bulk fill restorative material comprises of nano-sized zirconia fillers to enhance aesthetic properties and superior handling characteristics [14].

The success of any material is assessed by its longevity and biocompatibility in oral environment [15,16]. Despite the development in the composition and characteristics of these restorative materials, restorations in the oral cavity are subject to a number of conditions that may cause changes in the physical and mechanical properties of these restorations, such as color and microhardness per se. Thereby, undermining the quality of the restoration and eventually necessitating replacement [17]. One of the factors that may affect the quality of the restorations is the consumption of certain beverages such as coffee, tea, soft drinks, alcoholic beverages and even fluoridated water [18]. The effect of these beverages on microhardness of resin composite materials varies depending on the intrinsic features of the composite, such as their chemical composition [19, 20].

Therefore, the purpose of the current study was to evaluate effect of two daily consumed beverages on the durability of surface microhardness of zirconomer improved GI and micro-hybrid resin composite.

<table>
<thead>
<tr>
<th>Material</th>
<th>Chemical Composition</th>
<th>Type</th>
<th>#Lot</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filtek Z250 XT 3M ESPE, St. Paul, MN. (USA)</td>
<td>The Filler System: Surface-modified zirconia/silica with a median particle size of approximately 3 microns or less, Non-agglomerated/non-aggregated 20 nanometer surface-modified silica particles, filler loading is (82% by weight (68% by volume The Resin System: BIS-GMA, UDMA, BIS-EMA, PEGDMA, TEGDMA</td>
<td>Micro-hybrid resin composite</td>
<td>N721306</td>
</tr>
<tr>
<td>Zirconomer IMPROVED (Shofu INC, Japan)</td>
<td>Powder: aluminofluoro-silicate glass, zirconium oxide, tartaric acid Liquid: polyacrylic acid, deionized water</td>
<td>Zirconia-reinforced glass ionomer</td>
<td>03171480</td>
</tr>
</tbody>
</table>
MATERIALS AND METHODS

In this experimental study two different tooth-colored restorative materials were used; a Zirconia reinforced glass-ionomer; ZIRCONOMER IMPROVED (Universal shade) (Zr) and a microhybrid resin composite; Filtek Z250 (Shade A1) (RC). The materials’ brand names, chemical compositions, types and lot number are shown in table (1).

Specimens Preparation

Forty tooth-colored restorative material specimens were divided into two groups (20 specimens each), where group I were made of Zirconomer disks (Zr) and group II were made of resin composite discs (RC). A specially fabricated flat two halves split Teflon ring mold (7x7mm diameter and 4mm in thickness) was used for specimen’s fabrication of identical size. An external metal ring was used surrounding the two halves of the Teflon to keep the mold assembly.

In order to prepare Zirconia reinforced GI disks (Zr), the powder and the liquid were mixed with the spatula for 30 seconds (ratio 8.0:1.0g) on the mixing pad according to manufacturer’s instructions. A mylar strip was situated on a glass slab with the mold placed over. The material was then introduced into the mold until it is overfilled, then covered with another strip. The top of specimen was pressed with a light pressure to extrude the excess material. Finally, Zirconomer disc is left undisturbed for 3 minutes.

In order to prepare micro-hybrid resin composite discs (RC), the material was applied in two increments of 2mm each using Teflon tipped instrument which was used to adapt the material to all of the internal mold aspects. Each increment was cured with LED light curing unit (Litex 680A light curing unit) at 450-700mW/cm2 as manufacturer’s instructions, with holding the light guide tip resting on the upper surface of the mold for standardization of the distance of the curing light from the adhesive in all tested specimens. A celluloid matrix strip was pressed over the final increment in order to have non-porous highly finished composite restorations.

After polymerization of resin composite specimens and preparation of Zirconomer, specimens were stored in tap water at room temperature for about 24 hours to allow for maximum degree of conversion of the former and undisturbed setting reaction of the latter.

Immersion solutions

Ten specimens of each material; Zr and RC, were immersed in one of both solutions; Zr-Cola and RC-Cola (10 each) immersed in 50ml of acidic drink (Coca-Cola/Coca-Cola Company, Giza, Egypt), and Zr-Coffee and RC-Coffee (10 each) were immersed in 50ml of instant coffee which was prepared by adding 15gm of instant coffee powder (Nescafe/ Nestle Company, Giza, Egypt) into 500ml of boiling tap water. All the immersions were refreshed every 24 hours.

Surface microhardness testing

All specimens were subjected to surface microhardness (VHN) assessment (baseline). Each specimens’ top surface was indented at 3 points by a microhardness testing machine (Wilson (Beuhler) micro hardness tester, Germany) by using a 100-gr load with a dwell time of 10 seconds. Each indentation point was at least 1 mm away from each other or disc border. The mean of three points was calculated and reported as the surface hardness of each sample.

Thermocycling

For each material, the 20 specimens (10 Cola and 10 Coffee) were subject to microhardness testing (baseline groups; 10 Zr-Cola, 10 RC-Cola, 10 Zr-Coffee, and 10 RC-Coffee), then subjected to thermocycling for 1200 cycles. They were immersed at 5°C followed by 55°C for 20 seconds each, with an intermediary 5 seconds resting time (Thermocycling apparatus Mechatronic, Germany). All specimens were then subject to surface microhardness testing again in the same manner (thermocycled groups; 10 Zr-Cola, 10 RC-Cola, 10 Zr-Coffee, and 10 RC-Coffee), in an area away from the initial indentations.

Statistical analysis

Statistical analysis of the data was performed using (IBM® SPSS® Statistics Version 20 for Windows). Three-way ANOVA was used to test interaction effects. Independent specimens t-test was applied to compare between the means in non-related specimens at a significance level of p<0.05.

RESULTS

Data in Table (2) present mean Vickers surface hardness values and standard deviations of tested restorative materials (Zr) and (RC) after immersion in (Cola and instant Coffee) with and without thermocycling. There were significant interactions between Zr and RC (F=10296.630, P<0.001), between Cola and Coffee (F=1707.878, P<0.001), and between Control and thermocycled classes (F=189.764, P<0.001). Again, there was significant interaction between all tested variables (F=6.113, P=0.019) according to the Three-way ANOVA (Table 3).

In this study, the means of microhardness of RC were significantly higher than Zr, with the RC-Coffee (without thermocycling) had the hardest surface among all groups. For each material, thermocycling had a significant effect where the control group (without thermocycling) was significantly harder than the other group. Regarding immersion solution, Cola had a significant effect on the microhardness of all tested groups when compared with instant coffee. Zr-Cola, subject to thermocycling represented the least significant value among all tested group (p>0.05).

TABLE (2) Mean surface microhardness value (VHN) and standard deviations (khf/mm²) in each subgroup

<table>
<thead>
<tr>
<th></th>
<th>Zirconomer IMPROVED (Zr)</th>
<th></th>
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<tbody>
<tr>
<td></td>
<td>Cola</td>
<td>Coffee</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Control</td>
<td>Thermocycled</td>
<td>Control</td>
</tr>
<tr>
<td></td>
<td>46.95±0.40a</td>
<td>44.15±0.060b</td>
<td>55.45±0.71c</td>
</tr>
<tr>
<td></td>
<td>Filtek Z250 XT (RC)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Cola</td>
<td>Coffee</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Control</td>
<td>Thermocycled</td>
<td>Control</td>
</tr>
<tr>
<td></td>
<td>67.00±0.73a</td>
<td>64.34±0.60b</td>
<td>75.34±0.60c</td>
</tr>
</tbody>
</table>

In each row; within same materials, values with same lowercase letter were not significant statistically (p>0.05)
INFLUENCE OF BEVERAGES ON MICROHARDNESS OF ZIRCONIA REINFORCED GI

DISCUSSION

The use of glass ionomer restorative materials in dentistry has substantially increased over the past period. During that, they faced a lot of obstacles such as difficulty in handling properties, poor resistance to surface wear and poor resistance to fracture [21].

Manufacturers are trying to develop improved products with these disadvantages eliminated or reduced to an acceptable level [22,23,24,25]. Researches on metal free restorations were undertaken in the past 20 years [26], to investigate the properties of such materials. Zirconomer was introduced as a new class of glass ionomers with the incorporation of Zirconia fillers to enhance its fracture resistance, hardness, and strength. It combines an amalgam strength and durability together with the benefits of glass ionomers. However, these restorative materials have to sustain against a lot of oral environmental factors to which they are exposed such as water, saliva, acids, bases, and alcohols. All these factors have been related to the reduction of hardness [27, 28].

Therefore, this in vitro study was conducted to evaluate surface microhardness of this new material; Zirconomer (Zr) in comparison with a micro-hybrid resin composite; Filtek Z250 (RC) after exposure to two of the most commonly consumed beverages; Instant Coffee and soft drink (Cola pH=2.73). Besides, thermocycling was used as a simulation of the oral environment.

Hardness can be defined as a physical property that compromise fatigue strength of the material resulting from premature failure. The materials’ hardness correlates with the degree of conversion, and compressive strength [23, 29]. A direct relationship was found between low surface hardness and inadequate wear resistance together with tendency to scratching that compromises fatigue strength. This finally may lead to failure of restoration [24, 30].

Thermocycling is considered a common in vitro aging technique simulating the intake of hot and cold beverages and food in the daily life. In the current study, thermocycling was combined by storage in different pH solutions to try to generate the tension in the oral cavity to try to mimic what happens intraorally to a great extent. Since, 10,000 cycles are equivalent to 1 year of intra oral performance [39]. Therefore, specimens in the present study were subject to 1,200 cycles, for 20 seconds dwell time, and 5 seconds resting time representing approximately 1.5 months of in vivo activity.

The differences in the results of the present study were consistent with the few studies conducted during the past few years regarding this new material.

### TABLE (3) Results of Three-way ANOVA for the effect of different variables on mean microhardness.

<table>
<thead>
<tr>
<th>Source of variation</th>
<th>Type III sum of Squares</th>
<th>df</th>
<th>Mean Square</th>
<th>F-value</th>
<th>P-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermocycling</td>
<td>77,757</td>
<td>1</td>
<td>77,757</td>
<td>189.764</td>
<td>&lt;.001*</td>
</tr>
<tr>
<td>Solution</td>
<td>699.816</td>
<td>1</td>
<td>699.816</td>
<td>1707.878</td>
<td>&lt;.001*</td>
</tr>
<tr>
<td>Material</td>
<td>4219.121</td>
<td>1</td>
<td>4219.121</td>
<td>10296.630</td>
<td>&lt;.001*</td>
</tr>
</tbody>
</table>

Thermocycling x Solution x Material 2.505 1 2.505 6.113 .019*

df: degrees of freedom = (n-1), *Significant at P ≤ 0.05
They showed that there are several factors such as the storage time, storage condition, as well as the incorporation of hydroxyapatites had significant influences on the material hardness, compressive strength, surface roughness, sorption and solubility. In the present study, a statistically significant interaction between all tested variables was recorded. This goes well with Nuran et al., 2009 who found a significant interaction between time, immersion solution and material (p<0.001).

In the current study, RC-Coffee registered the highest values of microhardness when compared to Zr-Coffee before thermocycling. This goes well with Sharafeddin et al. in 2017, who stated that the lower hardness of Zr could be due to the heterogeneous phases present within this material.

On the contrary and taking into consideration the linear relationship between compressive strength and microhardness [Subaer et al. 2017], Zr had the greatest compressive strength among tested materials as reported by Bhatia at al. in 2017. They explained this by the homogenous incorporation of the microsized zirconia particles in the glass component, which further reinforces the material with high strength and lasting durability.

While after thermocycling both showed a significant reduction in surface hardness. This goes well with Sharafeddin et al. in 2017 who stated that Zr showed reduction in hardness after one day of water storage. This was explained by that Zr is self-curing and therefore chemically similar to glass ionomers, resulting in more dissolution in water. However, this contradicts a study conducted by Gu et al. in 2005 stating that Zr is resistant to dissolution as soon as it sets completely, and this phenomenon explains the lower surface hardness reduction of Zr throughout the study [31]. This again contradicts Nuran et al., 2009 where Resin composites showed modest increase in hardness after one month (in the present study a significant decrease was reported after approximately 1.5 month) followed by a significant decrease after one year.

The current study results are in accordance with Poggio et al., 2018 who stated that nanofilled and micro hybrid resin composites registered significant loss of microhardness after 1 week immersion in soft-drink while nanoceramic and nanohybrid composites resisted acid attack successfully and did not show significant loss of microhardness. They explained this by stating that resin composites might be exposed either continuously or discontinuously to chemical agents found in saliva, food, and beverages which might have different deleterious effect on the polymeric network and thus modifying its structure physically and chemically [33,34]. Besides, the erosion of resin composites produced from Cola might be the reason for the reported decrease in hardness as reported by Fatima et al. in 2013.

This contradicts Ilday et al. 2010 who found that Cola didn’t reduce surface hardness of microhybrid RC after immersion for 1 week. They stated that there was a gradual increase in microhardness irrespective of the type of beverages used. The free radicals within composites formed cross links after light curing. This usually occurs while the samples were stored in water just after curing and till the time of testing. In the present study, the significant decrease within both materials might be due to the more storage period the specimens were subject to, which is more than one week, that gave the solution enough time to cause changes within the material.

Somayaji et al in 2016, reported significant decrease in microhardness of micro hybrid composites after 21 days of immersion in acidic beverages with non-significant reduction after a shorter period of 7 and 14 days. They explained by stating that the acidic beverages containing phosphoric and carbonic erosive acids [40] with low pH might require a long period of time to dissolve the material [36].

In the present study, the Zr-Coffee and RC-Coffee groups (before and after thermocycling) showed significant higher hardness than Zr-Cola and RC-
Cola groups (before and after thermocycling). This may be explained by the fact that the immersion in coffee did not affect the microhardness to a greater extent as Cola (with low pH) did. This is due its less acidity, however, the significant decrease occurred after thermocycling in both groups. This may be addressed to the longer period of aqueous exposure with thermocycling which simulates the different temperature fluctuations like the oral conditions. This results in more water sorption and polymer degradation. Also, the presence of zirconia/silica fillers and zinc and glass fillers made the material to be more susceptible to hydrous attack [37]. Concerning contradictions of the results of the present study with others, it might be due to difference in specimen preparation method.

Finally, surface microhardness is considered as only one of many other factors representing a good restorative material. Therefore, future studies are needed to investigate other mechanical properties of this newly introduced material. Moreover, SEM photomicrographs may be needed in future studies to show if there is a direct relationship between the VHN values recorded and the structure of this tested material.

**CONCLUSIONS**

**Despite of the limitations of this in vitro study**, Zirconomer showed a good, so far not an excellent, behavior after thermocycling (to simulate the oral environment) together with being exposed to an acidic attack of Cola and a higher pH of Instant Coffee. It can be pointed out that Zirconomer is a promising esthetic restorative material that can be used in stress bearing areas due its high strength and low solubility based on previous studies although it showed a disappointing behavior when compared with microhybrid resin composite in this study.

**REFERENCES**


32. Claudio Poggio, Matteo Viola, Maria Miranda, Marco Chiesa, Riccardo Beltrami, and Marco Colombo. Microhardness of different esthetic restorative materials: Evaluation and comparison after exposure to acidic drink


35. Sultana N, Khan TH. Water absorption and diffusion characteristics of nanohydroxyapatite (nHA) and poly(hydroxybutyrate-co-hydroxyvalerate-) based composite tissue engineering scaffolds and nonporous thin films. J Nanomater, 2013; 479109.


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