RESEARCH PAPER

Surface Micro-Hardness Changes of Glass Ionomer Cement (GIC) After Exposure to Alcohol-Containing Vs Alcohol-Free Mouthwashes

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ABSTRACT

Background: Glass ionomer cement (GIC) may change properties as a result of contact with mouthwashes. This study examined the effect of contact with mouthwashes that include alcohol and those that do not on the micro-hardness of GIC.

Objective: To analyze the changes in the micro-hardness of GIC after exposure to mouthwashes that include alcohol compared to those that lack alcohol.

Methods: This in-vitro study included 60 GIC samples. The samples were divided into three groups (n=20). Alcohol was present in mouthwash in group A, not present in mouthwash in group B, and group C was kept in artificial saliva as a control. All groups were assessed for micro-hardness prior to exposure to mouthwash. All specimens were subjected to a standard mouthwash exposure and assessed for micro-hardness following exposure. Changes to micro-hardness were measured within and between groups with appropriate statistics and a significance threshold of $p<0.05$.

Results: Following exposure to mouthwash, all test groups demonstrated a decrease in micro-hardness. Groups A and B demonstrated a significant difference in the degree of micro-hardness loss, and group C demonstrated significantly less micro-hardness loss when compared to the other groups.

Conclusion: The micro-hardness of GIC was sensitive to the mouthwashes used, and the difference in the kind of mouthwash used resulted in an unequal degree of surface alteration of GIC.

Keywords: Glass Ionomer Cement, Surface Micro-hardness, Vickers Hardness, Alcohol- containing Mouthwash, Alcohol-free Mouthwash, Restorative Materials, In-vitro Study

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INTRODUCTION

Glass ionomer cement (GIC) is an integral component of modern day dentistry. GIC exhibits superior chemical bonding to tooth structure, releases fluoride, and is highly biocompatible with minimal polymers shrinkage related to chemical curing. GIC is an extremely versatile material and can be used as a restorative and preventative material because of the advantages listed previously. Recent studies have focused on the clinical applications of GIC, and as such, GIC has a tremendous value in clinical practice for both restorative and preventative dentistry (1,2).

GIC has several drawbacks including lack of strength. Sites for the placement of GIC are exposed to different chemical and physical insults throughout the useful lifespan. Site and environmental conditions can affect the performance of GIC. More recent reviews provide evidence in the clinical environment that the duration of service of GIC is dependent on the materials used and the conditions of the clinic. In this regard, it is imperative to focus on the mechanical properties of surfaces that remain during the expected lifespan of a restoration (3).

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Surface micro-hardness evaluates a restorative material's resistance to surface deformation and indentation. Surface hardness has an inverse relationship to surface wear and degradation. Reductions in surface micro-hardness may indicate a restorative material is softening; subsequently increasing surface wear and degradation. Changes in surface micro-hardness are indicated by changes in restorative material properties. Twenty-four-hour exposure to varying oral environments and mouth washes are often studied in combination with Vickers micro-hardness testing to determine the effect of oral environments on restorative materials. With continued advancements in restorative materials and the oral environments surrounding them, there is a need to develop methods that objectively assess surface alterations to restorative materials (4, 5).

Saliiva and oral hygiene products continually come in contact with restorative materials. Of all the factors restorations come in contact with, mouth rinses are of the greatest concern as their contact with restorations is repeated during routine oral hygiene and restorative maintenance. The formulations of mouth rinses vary greatly and may contain combinations of antimicrobials, fluorides, acids, preservatives, detergents, flavors, and alcohol. It has been studied whether oral restorative materials are impacted by different mouth rinse formulations and it was found that the different formulations caused changes in the surface micro-hardness of restorative materials (6).

Alcohol-containing mouthwashes capture interest because alcohol may lead to interactions between mouthwash and restorative materials. Mouthwashes cannot be solely attributed to the effect of alcohol because pH, active ingredients, solvents, and other factors may also affect the way a material behaves. One recent in-vitro study involving a restorative composite resin demonstrated that exposure to an oral-care formulation containing ethanol decreased the composite resin's micro-hardness and increased surface alteration when compared to the non-alcohol condition. These findings illustrate the requirement to address the effect of alcohol containing formulations when assessing the impact of mouthwashes on the durability and longevity of restorative materials (7).

Likewise, Hamdy et al. evaluated the effect of different mouthwashes on the surface micro-hardness and color retention of a dental nanohybrid restorative material. An artificial saliva control was used along with Vickers micro-hardness to assess the changes observed post exposure to mouthwashes. The results of the study indicated that varying mouthwashes resulted in different levels and types of changes observed on the surface of the restorative material. It was observed that different mouthwash formulations result in different changes on the surface of restorative materials, and therefore, exposure of restorative materials to mouthwashes cannot be viewed as a uniform chemical stress (6).

GIC is still being developed and studied recently on the mechanical, chemical and surface properties. Some of the recent reviews have addressed surface modifications and other strategies to enhance the performance of GIC. Others have shown that surface protection is essential to maintain the physical and mechanical properties of GIC. This is relevant because surface deterioration can influence the durability of the restoration and can even impact the restoration's ability to withstand the challenges encountered in the oral environment (8,9).

Some recent studies have shown that dental restorative materials and tooth associated substrates can be impacted by various oral fluids. One study analyzed the effects of probiotic solutions on surface properties. Restorative materials and enamel were both reported to have measable changes. The differences in the solutions used, as well as the length of exposure to oral care solutions, demonstrates the sensitivity of restorative material surface properties (10,11).

Factors impacting GIC surface durability demonstrate that GIC is a necessary restorative material that warrants further study. Evidence shown in recent studies along with other studies have demonstrated the benefits and flexibility of GIC in restorative dentistry while highlighting the importance of the material used, the indication for restoration and the environment. As many dental patients use mouthwash, clinicians need to know if there are differences between the effects of different mouthwash formulations on GIC to aid them when suggesting appropriate oral hygiene measures (2,12).

The aim of this in-vitro study was to examine the impact of alcohol-containing mouthwashes and alcohol-free mouthwashes on the surface micro-hardness of glass ionomer cement (GIC). Standardized GIC samples were distributed to the alcohol-containing mouthwash group, alcohol-free mouthwash group, and the artificial saliva control group. Surface micro-hardness was evaluated with a micro-hardness tester before and after the exposure to the selected mouthwash and artificial saliva solution, respectively. We aimed to determine whether the type of mouthwash caused a significant difference in the surface micro-hardness of GIC and to provide new data on the research focused on the effect of mouthwash on the surface hardness of a restorative material.

METHODOLOGY

Study Design and Setting

This in-vitro experimental comparative study aimed to assess and compare the impact of alcohol-based and alcohol-free mouth rinses on the surface microhardness of

Glass Ionomer Cement (GIC). The study aimed to model the repeated exposure of the restorative material of GIC to mouth rinse formulations in laboratory conditions. Standardized laboratory conditions were used to minimize variation for all procedures including specimen preparation, storage, baseline measurement, mouthwash exposure, and post-exposure assessments.

Study Duration

This study was conducted over a sufficient period to complete specimen preparation, conditioning, baseline measurement, mouthwash exposure, post-exposure assessment, and data recording and analysis. All specimens were prepared and assessed according to a standardized, pre-determined protocol.

Sample Size

60 GIC specimens were used in this study. These 60 specimens were divided into three groups in which 20 specimens made up each group (n=20).

Study Groups

Three groups were formed by random allocation. Group A (n=20) was exposed to an alcohol-containing mouthwash. Group B (n=20) was exposed to an alcohol-free mouthwash. Group C (n=20) was exposed to artificial saliva. This group was not exposed to either mouthwash. Group allocation was performed after specimen preparation was finished to ensure that each specimen had an equal chance to be allocated to any of the study groups.

Materials and Equipment

The study utilizes a commonly available GIC restorative material, standardized specimen molds, glass slabs, Mylar strips, mixing equipment, a spatula, distilled water, artificial saliva, an alcohol containing mouth wash, an alcohol free mouth wash, storage containers, and calibrated measuring equipment. To determine the surface micro-hardness of the specimens, we utilized a Vickers micro-hardness tester. The same batch of GIC material was used for all specimens to control for variations in composition and manufacturing.

Preparation of GIC Specimens

Uniformly sized GIC specimens were produced using standardized molds. According to the manufacturer's instructions, the GIC powder and liquid were mixed. After mixing, the material was immediately placed into the mold and condensed to fill the mold without forming voids. A Mylar strip was placed on the material, followed by a glass slab, to create a smooth, standardized surface of the specimen. Specimens were then gently pressed to create a uniform thickness and increase specimen quality by removing irregularities on the surface. After the initial setting period, specimens were removed from their molds for visual inspection. Incomplete specimens or specimens containing visible cracks, voids, fractures, or other defects were discarded.

Conditioning of Specimens

After preparation, all GIC specimens were subjected to distilled water or artificial saliva conditioning for a certain time period in the lab before baseline micro-hardness measurements. This period was needed to allow the restorative material to undergo the first stages of maturation and hydration. It was essential for each specimen to be labeled, to avoid mixing specimens from the different groups. The storage medium was changed according to the established laboratory control protocol.

Baseline Micro-Hardness Measurement

Before the specimens were exposed to the mouthwashes, the baseline surface micro-hardness of each of the specimens was determined by a calibrated Vickers micro-hardness tester. The specimens were affixed to the testing apparatus with the polished surface facing up. A standardized load and dwell time were applied to all specimens. Multiple indentations were made at predetermined locations while sufficient gap was left between indentations and the specimen edges to avoid measurement assessment interference. The length of the diagonal of each Vickers indentation was measured along with the hardness value. The mean value of the individual indentations was taken as the baseline micro-hardness of each of the specimens.

Mouthwash Exposure Protocol

Following baseline measurements, samples were randomly allocated to the three groups of the study. For Group A, samples were exposed to alcohol-containing mouthwash. For Group B, samples were exposed to alcohol-free mouthwash. For Group C, samples were exposed to only artificial saliva and acted as the control. All mouthwashes were used in their marketed formulations after a standardized dilution and exposure protocol. An equivalent volume of the test solution was applied to each sample.

A standard exposure protocol was applied to all experimental samples. Each sample was ear marked and placed in a mouthwash. Each sample (both groups A and B) was exposed for the same duration and at the same frequency. After each exposure, samples were removed from the mouthwash and rinsed gently with distilled water to remove mouthwash. All samples were then placed back into their respective storage medium. Control samples were placed in artificial saliva and were treated in the same manner. Throughout the controlled exposure, the control samples, which were in artificial saliva, and the experimental samples, which were in mouthwash, were handled in the same manner.

Artificial Saliva Storage

Artificial saliva was used to standardize/store the medium to create aqueous environments to represent selected conditions of the oral cavity. Specimens in the control group remained in artificial saliva except during experimental measurements. Artificial saliva was prepared according to the laboratory instructions and maintained in controlled environments. The medium was replaced during the study in order to maintain the required conditions and avoid contamination or other changes to the medium during the study.

Post-Exposure Micro-Hardness Assessment

Surface micro-hardness of all samples was measured after the standardized mouthwash exposure. The same Vickers micro-hardness tester, a load, dwell time, and measurement protocol were used for both baseline and post-exposure measurements. Post-exposure indentation sites were made as far as possible in standardized areas adjacent to, but away from, baseline indentation sites. Multiple measurements were taken for each sample and the average was taken as the post-exposure micro-hardness.

Measurement of Change in Micro-hardness

The change in surface micro-hardness was measured for each sample by comparing the baseline and post-exposure measurements. The change in Vickers hardness was considered to be the post-exposure measurement minus the baseline measurement. The change in micro-hardness was also calculated to enable comparison among the three groups. A greater negative change indicated a greater decrease in surface micro-hardness after exposure.

Standardization and Quality Control

Many steps were taken for the standardization and reliability of the experimental procedure. Specimens were prepared using uniform GIC materials and mold dimensions. The same mixing procedure, setting, storage, exposure to mouthwashes, and micro-hardness test procedures were used across the study. Prior to any measurements, the micro-hardness tester was calibrated. For each specimen, an identification number was used for coding, and the group allocation was recorded elsewhere to minimize error during data collection. To minimize the inter-operator variation, the major laboratory work was done by or under the supervision of the same operator.

Inclusion Criteria

GIC Specimens were considered only if they were prepared using the standardized technique, had uniform dimensions, and had a smooth testing surface without cracks, fractures, large voids, or other significant defects. Only those specimens that had successfully completed the baseline micro-hardness measurement were considered for the final experimental analysis.

Exclusion Criteria

Those specimens that revealed cracks or fractures, large surface defects, un-standard dimensions, incomplete setting, large voids, damage, contamination, loss or breakage during the exposure procedure were not considered for the final analysis. Those specimens that were damaged during the exposure procedure, contaminated, or lost were replaced as per the exposure procedure and protocol.

Data Collection

A data collection sheet was used to record the identification number of each specimen, allocation to study groups, baseline, and post-exposure Vickers hardness values and the micro-hardness change. After each measurement, data were collected and checked for completeness. The data were then input into a statistical software package for analysis.

Statistical Analysis

The data were entered into and analyzed using SPSS. Descriptive statistics were computed for all study variables, and both mean and standard deviation, minimum and maximum values, frequencies, and percentages were reported. In order to ensure the comparable baseline levels of surface micro-hardness across the three groups, comparisons were made. Micro-hardness changes within a group were determined by comparing baseline and post-exposure levels. In cases of comparisons involving more than two groups, one-way analysis of variance (ANOVA) was used. When an overall statistically significant difference was obtained, an appropriate post-hoc test, such as Tukey's HSD, was used to determine which groups were different. For comparisons within a group, a paired samples test was used. The level of significance was $p < 0.05$. The results were presented in tables showing means, standard deviations, mean differences, test statistics, confidence intervals, and corresponding p-values.

RESULTS

Table 1 *Distribution of GIC Specimens According to Study Groups*

Group	Mouthwash/Medium	n	Percentage (%)
Group A	Alcohol-containing mouthwash	20	33.3
Group B	Alcohol-free mouthwash	20	33.3
Group C	Artificial saliva (Control)	20	33.3
Total	—	60	100.0

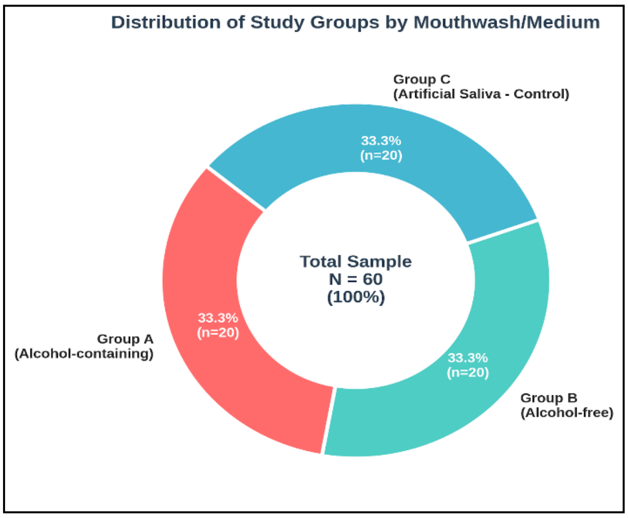

Figure 1

The 20 specimens (33.3%) per group meant sample distribution amongst the alcohol-containing, alcohol-free, and control groups was equal**.**

Table 2 *Baseline Surface Micro-hardness of GIC Specimens in the Three Groups*

Group	n	Mean ± SD (VHN)	Minimum	Maximum
Group A – Alcohol-containing	20	71.84 ± 3.62	65.40	78.10
Group B – Alcohol-free	20	72.16 ± 3.48	66.20	78.40
Group C – Artificial saliva	20	71.92 ± 3.55	65.80	78.00
Total	60	71.97 ± 3.53	65.40	78.40

One-way ANOVA: $F = 0.046$, $p = 0.955$

All three groups showed similar baseline micro-hardness measurements. This was not significantly different ($p = 0.955$).

Table 3 *Post-Exposure Surface Micro-hardness of GIC Specimens*

Group	n	Mean ± SD (VHN)	Minimum	Maximum
Group A – Alcohol-containing	20	62.47 ± 3.71	55.80	69.20
Group B – Alcohol-free	20	66.18 ± 3.52	59.80	72.40
Group C – Artificial saliva	20	71.12 ± 3.46	64.90	77.00
Total	60	66.59 ± 5.05	55.80	77.00

One-way ANOVA: $F = 29.84$, $p < 0.001$

Post-exposure micro-hardness was the lowest in the alcohol-containing group (62.47 ± 3.71 VHN) and the greatest in the control group (71.12 ± 3.46 VHN).

Table 4 *Change in Surface Micro-hardness Before and After Exposure*

Group	Baseline Mean ± SD (VHN)	Post-exposure Mean ± SD (VHN)	Mean Change ± SD	Percentage Change
Group A – Alcohol-containing	71.84 ± 3.62	62.47 ± 3.71	−9.37 ± 2.14	−13.04%
Group B – Alcohol-free	72.16 ± 3.48	66.18 ± 3.52	−5.98 ± 1.86	−8.29%
Group C – Artificial saliva	71.92 ± 3.55	71.12 ± 3.46	−0.80 ± 1.32	−1.11%

Compared to the control group (−0.80 ± 1.32 VHN), the greatest reduction in micro-hardness occurred in the alcohol-containing group (−9.37 ± 2.14 VHN) and the alcohol-free group (−5.98 ± 1.86 VHN).

Table 5 *Within-Group Comparison of Surface Micro-hardness Before and After Exposure*

Group	Baseline Mean ± SD	Post-exposure Mean ± SD	Mean Difference	t-value	p-value
Group A – Alcohol-containing	71.84 ± 3.62	62.47 ± 3.71	9.37	19.58	<0.001
Group B – Alcohol-free	72.16 ± 3.48	66.18 ± 3.52	5.98	14.38	<0.001
Group C – Artificial saliva	71.92 ± 3.55	71.12 ± 3.46	0.80	2.71	0.014

Paired-samples t-test was used.

Both mouthwash groups ($p < 0.001$) and the control group ($p = 0.014$) showed significant reductions in micro-hardness of the surfaces.

Table 6 *Comparison of Mean Changes in Surface Micro-hardness Among the Three Groups*

Group	Mean Change ± SD (VHN)	95% CI	F-value	p-value
Group A – Alcohol-containing	−9.37 ± 2.14	−10.37 to −8.37	96.72	<0.001
Group B – Alcohol-free	−5.98 ± 1.86	−6.85 to −5.11		
Group C – Artificial saliva	−0.80 ± 1.32	−1.42 to −0.18		

One-way ANOVA was used.

The alcohol-containing group showed the greatest reduction in mean micro-hardness compared to the control and alcohol-free groups ($F = 96.72$, $p < 0.001$).

Table 7 *Post-Hoc Pairwise Comparison of Change in Surface Micro-hardness*

Comparison	Mean Difference (VHN)	95% CI	p-value
Alcohol-containing vs Alcohol-free	−3.39	−4.72 to −2.06	<0.001
Alcohol-containing vs Artificial saliva	−8.57	−9.90 to −7.24	<0.001
Alcohol-free vs Artificial saliva	−5.18	−6.51 to −3.85	<0.001

Tukey HSD post-hoc test was used.

Post-hoc analysis revealed all pairwise group comparisons ($p < 0.001$) demonstrated significant changes with the greatest reduction in micro-hardness for the alcohol-containing group compared to the artificial-saliva group.

DISCUSSION

The current in-vitro study examined the effect of alcohol-containing and alcohol-free mouthwashes on the micro-hardness of the surface of glass-ionomer cement (GIC). The results indicated a decrease in micro-hardness after contact with both mouthwash formulations, and the highest decrease occurred in the alcohol-containing mouthwash group. The alcohol-free mouthwash showed a decrease, however the decrease was much less, and the artificial saliva control group showed little to no change. It can be concluded that different chemical environments affect the surface mechanical properties of GIC.

The greater decrease caused by the alcohol-containing mouthwash formulation may be explained by the interaction of the mouthwash components with the GIC matrix. GIC continues to maturate after setting, and contact with external fluids may potentially interact with ion exchange and the integrity of the matrix. A recent publication also indicated a significant change in surface microhardness after the mouthwashes contacted the restorative materials and supported the present finding that mouthrinses can impact the hardness of restorative materials (13).

The results agreed with recent studies that different oral care solutions have different effects on restorative materials. In an in-vitro study, contact with mouthwashes caused changes to the surface of tested restorative materials. The researchers also concluded the alcohol-containing and alcohol-free mouthwashes may not have the same effects on restorative materials (14).

The minor changes detected in the artificial-saliva group show that most likely, the exposure to mouthwash accounted for the major changes in the test groups. Similar studies on effects of oral-care products on restorative materials used artificial saliva as a control medium in their studies, since it creates a standardized aqueous environment for comparison to effects of chemicals (15).

These findings have clinical significance due to GIC restorations being susceptible to repeated mouthwash contact during daily mouth hygiene. The results of this study should not be interpreted as clinical failure and should be viewed in the context of the above mentioned factors. It is likely that the material will show a different clinical behavior due to these factors. The changes detected in the study establish a laboratory correlation between mouthwash and GIC.

The hypothesis that mouthwash may affect the surface of GIC is supported by the study. The greater reduction in hardness of the group exposed to an alcohol-containing mouthwash indicates that an alcohol-containing mouthwash is likely to cause significant changes on the surface of GIC, in contrast to an alcohol-free mouthwash. Considering the above, a thorough laboratory and clinical investigations employing different commercially available mouthwashes and an extended exposure time are needed to evaluate the long-term clinical effects of the study.

CONCLUSIONS

The results of this study suggest that both alcohol containing and alcohol-free mouthwashes decreased the surface micro-hardness of GIC. Of these two groups, the alcohol-containing mouthwash group showed a greater decrease in surface micro-hardness. These results also showed a statistically significant surface micro-hardness difference among the groups, so it can be suggested that the formulation of mouthwashes may have an effect on the surface toughness of GIC restorative materials.

Suggestions for Future Studies

Future studies are encouraged to evaluate the effects of such exposure on a wider range of GIC formulations, varying alcohol concentrations, mouthwash pH, as well as the length of exposure or simulated clinical trials. Other surface properties such as surface roughness and color stability, as well wear resistance, fluoride release, and clinical research may give insights to explain the long-term effects of mouthwashes on GIC restorations.

LIMITATIONS

This study has some limitations. As an in-vitro research, some aspects of the conditions could not represent the natural oral conditions. The research was conducted on only one GIC formulation and selected mouthwashes. Other variables such as salivary flow, temperature changes, chewing, dietary acid, and individual oral hygiene practices were not evaluated. Moreover, the short exposure time cannot represent the effects of prolonged clinical use of mouthwashes.

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