Evaluation of Surface Roughness, Water Sorption, and Solubility of Glass Ionomer Restorative Cement Incorporated with Different Antibiotics

Wafaa S. Ahmed1, Essam E. Al–Wakeel2, Enas T. Enan3 Noha A. El–wassefy4

Abstract:
Objective: To evaluate the influence of adding different antibiotics to glass ionomer cement (GIC) on its surface roughness, water sorption, and solubility. Materials and Methods: A two-component GIC system (Fuji IX) and commercially available antibiotic tablets including ciprofloxacin and amoxicillin /clavulinate were used. The two types of powdered antibiotics were incorporated and stirred with the GIC powder at a ratio of 2 wt%. A total number of 120 Specimens were prepared using a split Teflon mold and grouped as follows; group I: Conventional GIC as a control group, group II: Ciprofloxacin-modified GIC, group III: (Amoxicillin/Clavulanate)-modified GIC, group IV: a combination of the two antibiotics-modified GIC. Each group was tested for surface roughness using a profilometer. Water sorption and solubility were evaluated after 7 days of immersion in distilled water. The collected data of each test were statistically analyzed using a two-way analysis of variance (ANOVA) followed by post-hoc Tukey multiple comparisons. The level of significance was set at P ≤ 0.05.

Results: For water sorption, the highest mean value was recorded in group II while the lowest value was reported in group III. For the solubility test, the greatest median value was recorded in group IV with the least value recorded in group III. For surface roughness, the highest mean Ra value was found in group II while the least value was reported in the control group (group I). Conclusions: The present in vitro study demonstrated that the incorporation of 2% of antibiotics into glass ionomer cement leads to increase solubility, water sorption, and surface roughness of GIC.

Introduction:
The demand for an optimal restoration became a challenge for researchers in restorative dentistry and the current era of preventive and conservative dentistry. The aim of restorative material development should be to produce bioactive materials that have therapeutic benefits.1 GIC is a common biomaterial used in dentistry.2 It has unique characteristics like biocompatibility, anti-cariogenic effect, antibacterial activity, chemical adhesion to hard tooth structures, the release of fluoride ions, and a low coefficient of thermal expansion that is similar to the tooth structure.3 It is commonly utilized as a restoration, luting cement, and cavity base; also it is known as the main restoration for deciduous teeth in addition to various therapeutic uses in dentistry, including Atraumatic Restorative Treatment (ART).4 Numerous modifications have been made to GICs to improve their physical, mechanical, and biological properties. Therapeutic effects can be obtained by incorporating GICs with other antibacterial agents,5 as zinc ions, silver ions, iodine, and chlorhexidine.6-8 Unfortunately, the inclusion of antibacterial agents in restorative materials results in alterations in their mechanical and physical characteristics.8

The durability of restorative materials is influenced by many variables, such as hardness, water sorption, and solubility. Water sorption can change the volume of a material and can cause deterioration of the matrix structure by acting as a plasticizer.9 The rate of dissolution of cement is not only affected by the testing conditions, but also by the specimen shape and thickness, powder/liquid ratio of the cement, pH, dissolution time, and concentration of the solute.7 Surface roughness also is one of the most important factors in determining the performance and durability of restorative materials.10 It is defined as the fine irregularities or deviations from the optimum form of restorative materials' natural surface texture.11 The surface roughness of restorative materials has a significant clinical value since it indicates the deposition of plaque, discoloration, and wear and abrasion resistance.12 Smooth surface enhances the patient's comfort, because a change in surface roughness of 0.3 μm may be noticed by the tip of the tongue.13 Rough or irregular surface texture may raise the risk of dental diseases and may lead to dental plaque collection, gingival irritation, biofilm retention, discoloration, and unsatisfactory aesthetic appearance of restorations.12,14 So, modification of restorative materials, like glass ionomer cement by introducing antimicrobials should not damage inherent characteristics Hence, the present study aimed to evaluate the influence of adding different antibiotics to

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Table: Comparison of control and modified groups for water sorption, solubility, and surface roughness

<table>
<thead>
<tr>
<th></th>
<th>Group I</th>
<th>Group II</th>
<th>Group III</th>
<th>Group IV</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water Sorption (Mean±SD)</td>
<td>0.66±0.08</td>
<td>0.69±0.10</td>
<td>0.60±0.18</td>
<td>0.66±0.13</td>
<td>0.68</td>
</tr>
<tr>
<td>Solubility (Median-(range))</td>
<td>-0.12 (-0.22 – 0.04)</td>
<td>-0.16 (-0.25 – (-0.13))</td>
<td>-0.08 (-0.16 – 0.03)</td>
<td>-0.20 (-0.32 – (-0.06))</td>
<td>0.55</td>
</tr>
<tr>
<td>Surface roughness (mean±SD)</td>
<td>0.48±0.07</td>
<td>0.84±0.30</td>
<td>0.62±0.18</td>
<td>0.68±0.21</td>
<td>0.046*</td>
</tr>
</tbody>
</table>

* P significant at ≤0.05

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GIC on selected properties of it, including water sorption, solubility, and surface roughness.

Material and methods:

Materials: A two-component glass ionomer cement system (Fuji IX high strength posterior restorative GC; GC International, Tokyo, Japan) was used as a control. This material was modified by adding different antibiotics including ciprofloxacin and amoxicillin/clavulanate.

Methods: Sample size calculation: The sample size for this study was calculated initially before any work using G*Power program (G*Power Ver. 3.0.10, Kiel, Germany). The total sample size of 120 specimens achieved 80% power (equal to type II error), type I error (α) was 0.05.

Cement modification and grouping: To prepare the modified glass ionomer cement, commercially available antibiotic tablets were ground into a fine powder using a mortar and pestle. The grounded antibiotics were weighed using a four-digit scale and the ratio was adjusted to be 2 wt%. The powdered antibiotics were incorporated and stirred with the glass ionomer powder.

A total number of 120 Specimens were grouped according to the type of modification as follows: (n = 30 for each group)
- Group I: Conventional glass ionomer cement as a control group.
- Group II: Ciprofloxacin-modified glass ionomer cement.
- Group III: (Amoxicillin/Clavulanate)-modified glass ionomer cement.
- Group IV: Combination of the two antibiotics-modified glass ionomer cement.

Water Sorption and Solubility measurement

Specimens’ preparation: To determine the water sorption and solubility, 20 disc-shaped specimens were used (five specimens for each group).

Water Sorption and Solubility measurement: For measurement of Solubility, the specimens were kept in a desiccator for 24 hours with fresh silica gel and then weighed to give the initial mass (m1). The thickness and diameter of each specimen were measured. Each sample's volume was determined: V = πr²h [mm³], where π = 3.14, r represents the medium radius, h for the thickness, and V for the volume. After that, each specimen was stored individually and vertically in a glass bottle containing 10 ml of distilled water. The bottles with the specimens were kept at 37 ± 1 °C for 7 days in an incubator. After this period, each specimen was removed from the water and dried gently. Then each specimen was weighed, and this value is termed (m2). Then put the specimens in a desiccator with fresh silica gel to record the final mass after dehydration (m3). The water sorption (WS) and the solubility (SL) were determined with the following equations:15

\[ W_S = m_2 - m_1 / m_1 \]
\[ S_L = m_1 - m_3 / m_1 \]

Where (m1) is the initial mass (mg) before water immersion, (m2) is the mass after 7 days of immersion, (m3) is the final mass after the specimen has been dried in the desiccator, and (V) is the specimen's volume.16

Surface roughness measurement:

Specimens’ preparation: Twenty disc-shaped specimens were prepared (5 specimens for each group). The average surface roughness of the specimens (Ra, μm) was measured with a contact profilometer (Surftest SJ210, Mitutoyo Corp, Kawasaki, Japan) as shown in Figure, following ISO 4287-1997 with the stylus traversing distance of 4 mm, the cut-off value for surface roughness was of 0.8 mm, and a measuring speed of 0.5 mm/s.

Figure: Surface roughness measured by a profilometer.

The mean surface roughness was estimated after recording three subsequent measurements in various directions for all specimens in each group.2, 17 During the experimental period, a calibration block was used periodically to evaluate the performance of the profilometer.
Statistical analysis: Values were presented as mean and standard deviation (SD). Data were explored for normality using Kolmogorov-Smirnov test of normality. For parametric data, a one-way analysis of variance (ANOVA) test was used for comparison between groups, followed by Tukey's post hoc test for pairwise comparison. Solubility test was non-parametric data and was compared using Kruskall-Wallis test between groups; followed by post-hoc Dunn's whenever the difference between groups was statistically significant. The level of significance was set at $P \leq 0.05$.

**Results:**

For water sorption, the highest mean value was recorded in group II with the lowest value reported in group III. For the solubility test, the greatest median value was recorded in group IV while the least value was recorded in group III. For surface roughness, the highest mean Ra was found in group II while the least was reported in the control group (group I), Table.

**Discussion:**

Glass ionomer cement has the advantage of being able to change its biological and physical characteristics simply by changing the powder-to-liquid ratio or by chemical formulation. Several changes have been made in glass ionomer cement to enhance its physical, mechanical, and biological characteristics.

Regarding Water sorption and solubility, they are important factors to consider when evaluating bonding materials since they are linked to the cement's durability and the longevity of restorative materials. Water sorption can affect the volume of material and induce damage to the matrix structure. Water sorption tests assess a specimen's net weight increase as a result of water molecule diffusion and elution.

Initially, the water sorption process transfers Ca$^{2+}$ and Al$^{3+}$ into glass ionomer cement, where they react with polyacrylic acid. But, excessive water absorption leads to cement degradation and deterioration over time, causing a compromise of structural and mechanical features.

In this study, the water sorption and solubility were measured after immersion of the specimens for 7 days, since it has been mentioned in previous studies that the largest quantity of water uptake occurs in most hydrophilic materials within the first week. For water sorption, all of the examined groups gained water at the end of the immersion time, there were no statistically significant variations in mean values between the control and the three modified groups, a probable explanation is that there is no difference in their chemical composition, as the studied groups are all conventional glass ionomer cement and as a result, there is no significant variations in their water sorption capacity measurements were found. A material's solubility refers to its capacity to dissolve in another material.

For solubility, all of the examined groups had negative mean values. Negative solubility measurements may be due to incomplete dehydration of these materials, negative readings were also found by Toledano et al. in 2006, and Keyf et al. It's possible that the acid-base interaction was extended and water molecules were constantly bonded into their structures. As a result, the materials gained weight and expanded. As the acid-base interaction progresses, the glass ionomer cement absorbs water as an essential part of its structure; as a result, the higher the rate of reaction, the higher the water absorption into the cement structure and vice versa. The greatest median value was recorded in the combination group while the least value was recorded in the amoxicillin-modified GIC group. These variations might indicate that the water molecules did not bond to the structure equally in all groups after the acid-base reaction ended. As a result, some of the absorbed water molecules were either only trapped in the space of the matrix, filler, or matrix–filler interface. Then, this loosely bonded water was vaporized out of the specimens after drying in the desiccator.

One of the most critical surface features in the clinic is surface roughness, it is one of the most important factors in determining the performance and durability of restorative materials. It is defined as the fine irregularities or deviations from the optimum form of restorative materials' natural surface texture. The surface roughness of restorative materials has a significant clinical value since it indicates the deposal of plaque, discoloration, wear, and abrasion resistance. Smooth surface enhances the patient's comfort, because a change in surface roughness of 0.3 μm may be noticed by the tip of the tongue. Rough or irregular surface texture may raise the risk of dental diseases as it leads to dental plaque collection, gingival irritation, biofilm retention, discoloration and unsatisfactory aesthetic appearance of restorations.

Surface roughness was measured with a profilometer in several in vitro studies. Likewise, profilometric analysis was used to measure Ra levels in this study because it is accurate and simple to use. Evaluation of surface roughness is determined by measuring the small-scale differences in the height of a physical surface. The average of heights (Ra) is the most widely used measure for assessing surface roughness and was used to express the surface roughness in μm. The lower the Ra value, the smoother the specimen's surface. Bollen et al. and Silva et al. determined that 0.2 μm was the essential surface roughness (Ra) for bacterial colonization of numerous dental materials. Surface roughness values of more than 0.2 μm are likely to promote bacterial adhesion, dental plaque development, and acidity.

In the current study, all experimental groups exhibited values higher than 0.2 μm including the control group.

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incorporating antibiotics, has high surface roughness values, the modified GICs have a bactericidal effect and reduce recurrent caires.

**Conclusion:**

The incorporation of 2 wt% of antibiotics into glass ionomer cement leads to an increase in its solubility, water sorption, and surface roughness.

**References:**


