COMPRESSIVE STRENGTH AND SETTING TIME DETERMINATION OF GLASS-IONOMER CEMENTS INCORPORATED WITH CETYLPYRIDINIUM CHLORIDE AND BENZALKONIUM CHLORIDE

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Abstract: Because of the relatively frequent occurrence of recurrent caries after a restorative treatment, and because of the huge number of cariogenic microorganisms present in the oral cavity, which present a potential risk factor regarding the development of new carious lesions, attention has increasingly been directed towards the therapeutic antimicrobial effects of restorative materials. The glass ionomer cements distinguish themselves as the most acceptable restorative materials possessing the positive characteristics of fluorine in the processes of remineralisation and antimicrobial action. In addition to the release of fluoride ions, GICs can potentially be used as templates for the release of other active antimicrobial components. The addition of antimicrobial compounds in the glass ionomer cements and analysis of their physical characteristics are very important especially for use in the posterior region of milk teeth. The aim of this study was to analyse the physical characteristics of ChemFlex and Fuji IX, conventional glass ionomer cements incorporated with the antimicrobial components Cetylpyridinium Chloride and Benzalkonium Chloride, through measurements of their setting times, and determination of their compressive strengths. Five samples of each glass ionomer with no antimicrobial compounds added were prepared – to serve as a control group; and collections of five samples of each cement with different concentrations of Cetylpyridinium Chloride and Benzalkonium Chloride – 1%, 2% and 3% – added to them were also prepared – a total of 60 samples. The results of the analysis point out that it is possible to incorporate these antimicrobial agents in conventional...
GICs, and this is especially true when the added amount of the antimicrobial agents is 1%.

**Key words:** glass ionomer cements, antimicrobial agents, cariogenic micro-organisms, Cetylpyridinium Chloride, Benzalkonium Chloride, working time, compressive strength.

**Introduction**

Dental caries is an infectious disease resulting in dental structure destruction, which is caused by microorganisms, mostly by mutans streptococci. There is a clear and positive correlation between the existence, i.e. detection, of *Streptococcus mutans* and *Lactobacillus species* in the saliva, the plaque, and dental caries. [2, 3, 6, 12, 13, 23, 30, 31, 40, 45, 50, 55, 58, 62]

Taking into consideration the fact that bacterial flora is the most important ring in the chain of the occurrence and development of dental caries, as it is for the development of a secondary caries underneath restorations, the endeavours to devise various means – both preventive and restorative, which could reduce the number of bacteria and, consequently, break the aforementioned chain, are justified. [20]

From a preventive viewpoint, there are an enormous number of products, both commercial and professional, such as dentifrices and mouth washes, which have in their structure one or several different antimicrobial components. As main examples, we could cite fluorine, chlorhexidine, cetylpyridinium chloride, benzalkonium chloride, triclosan, listerine, and xylitol. [9, 20, 47, 48, 56]

Because of the relatively frequent occurrence of recurrent caries after a restorative treatment, and because of the huge number of cariogenic microorganisms present in the oral cavity, which present a potential risk factor regarding the development of new carious lesions, attention has increasingly been directed towards therapeutic antimicrobial effects of restorative materials.

The glass ionomer cements distinguish themselves as most acceptable restorative materials possessing the positive characteristics of fluorine in the processes of remineralisation and antimicrobial action. [7, 38, 42, 43, 53] Their usage is determined by their characteristic ability to release fluorine [6–8, 59, 21, 5] and to participate in the mechanism of inhibition of the development of secondary caries. [7, 38, 42, 43] At the same time they also act on the surrounding bacteria by reducing the cariogenic microorganisms. [20, 28, 57]

In addition to the release of fluoride ions, GICs can potentially be used as templates for the release of other active antimicrobial components.
In order to improve the antimicrobial characteristics of both conventional and resin-modified GICs, antimicrobial compounds such as chlorhexidine have been added. Chlorhexidine has been described as a golden standard for antibacterial application [34]. There are several studies dealing with the effect of incorporating chlorhexidine and its combinations in glass ionomer cements in different concentrations on cariogenic salivary flora, and with the impact of incorporated chlorhexidine on the mechanical properties of GICs as well. [32, 49, 53, 47, 51, 10]

There is a very low quantity of data in the literature referring to the incorporation of other antimicrobial components in GICs. Although some of them have a confirmed effect in the reduction of cariogenic salivary flora when used in rinses or toothpastes, [4, 9, 15–17, 19, 25, 33, 42, 47–48] the results regarding their incorporation in glass ionomer cements are still few.

The addition of antimicrobial components to glass-ionomer cements and the analysis of their physical characteristics are very important especially when these materials are used for atraumatic restorative treatments (ART). However, the incorporation of antimicrobial agents very often changes the mechanical properties of the restorative materials. Because the ART approach with GICs is indicated mainly for posterior teeth, it is necessary for the restorative material to demonstrate sufficiently good mechanical properties in order to be able to resist the occlusal pressure. Thus, the antimicrobial GICs have to have an optimal quantity of antimicrobial compounds, such that would not imperil the basic characteristics of the main materials [44, 53].

In the attempt to better understand the physical properties of GICs, compressive strength testing is, besides other methods, the most commonly employed method. To perform such studies, some researchers have adopted standards and specifications for dental materials, instruments and dental equipment. There are several valid standards for compressive strength determination of GICs. One of them is Specification No 66 from 1987, which uses samples measuring 12 mm in height and 6 mm in diameter. Another specification is the ISO specification (7489: 1986) [29] according to British Standard (BS 6039: 1981) [18], where specimens are defined to have a height of 6 mm and a diameter of 4 mm. Williams and Billington [61] analysed the compressive strength of glass ionomer cements after 30 minutes, 1 hour and 24 hours following the ISO specification. Recent investigations present that in period between 24 hours and 4 months some materials, especially those based on poliacrylic acid, maintain, even describe a slight increase in the strength, while other cements based on copolymers of acrylic acid describe, first increasing then decreasing strength. A modern glass ionomer material presented by Williams and Billington [61] has compressive strength values which progressively increases in the period of 24 hour from 170 MPa to 220 MPa after 24 hours. Similarly, Cattani-Lorente et al.
and McComb et al. [37] also followed ISO 7489: 1986 specifications for the water-based dental cements. Gerdullo et al. [27] and Drummond et al. [22] differed from these authors regarding methodology when they used specimens of 12 mm in height and 6 mm in diameter to test the compressive strength of glass ionomer cements, thus following ANSI/ADA Specification No 66 for dental cements. In addition to the details regarding the two dimensions of specimens, the conclusion has been drawn that a 24-hours storage period is most acceptable, since most of these materials reach their strength value limit within this period, which is in agreement with the British Standard (BS 6039: 1981) [17] and the International Standard specified by ISO (7489: 1986) [29]. Different methodologies can thus be employed to evaluate the compressive strength of glass ionomer cements, especially regarding standards on specimen dimensions. However, doubts still remain as to whether any differences might be found in the results obtained when different specimen dimensions are used for testing materials with different formulations of conventional and resin-modified glass ionomer cements [35].

Considering that microorganisms are the main cause for the occurrence of dental caries, that glass-ionomer cements are materials of choice mainly in paediatric dentistry, and that they can potentially be used as a medium for slow release of active antimicrobial components, and having the knowledge that investigations regarding the incorporation of antimicrobial compounds into glass ionomer cements are still scarce, we have defined the main aim of this study: an analysis of the mechanical properties of conventional glass ionomer cements containing antimicrobial agents through compressive strength and setting-time determination.

Material and methods

Material

The commercially available glass ionomer cements used in the analyses were ChemFlex and Fuji IX. The respective compositions of these glass ionomer cements are shown in Table 1.

The antimicrobial compounds used in the study are: Cetylpyridinium Chloride C0732 by Sigma – Aldrich Co. and Benzalkonium Chloride 12660 by Fluka Chemical Corporation Milwaukee, WI, USA.

Samples were prepared of each glass-ionomer cement as follows: Five samples of the conventional Fuji IX glass-ionomer with no antimicrobial compound added were prepared to serve as a control group; three groups of the same cement of 5 samples each, but with Benzalkonium Chloride incorporated, were prepared, each group with a different percentage of the agent (i.e., 1%, 2%, 3%); and another three 5-sample groups, but with Cetylpyridinium...
Chloride incorporated, were also prepared – each group having the corresponding percentage of this agent (i.e., 1%, 2%, 3%). The same procedure was followed for ChemFlex.

### Table 1

**Conventional glass-ionomer cements used in the research**

<table>
<thead>
<tr>
<th>Material</th>
<th>Classification</th>
<th>Composition</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>FUJI IX</td>
<td>Conventional glass-ionomer cement</td>
<td>Silicon, aluminium, sodium, fluorine, phosphorus, strontium</td>
<td>GC Int., Tokyo, Japan</td>
</tr>
<tr>
<td>GP</td>
<td>Conventional glass-ionomer cement</td>
<td>Strontium, aluminium, fluoride, silicate, tartaric acid, pigments, polyacrylic acid</td>
<td>DENTSPLY DeTrey, Konstanz, Germany</td>
</tr>
</tbody>
</table>

**Methods and techniques**

**Preparation of Samples**

Samples with no antimicrobial agent incorporated were prepared by mixing a certain amount of powder and liquid on glass mixing plates by means of a metal trowel (according to the manufacturers’ instructions). The freshly mixed paste was then put into 6 mm high metal moulds 4 mm in diameter. The moulds were closed with metal plates on both sides and were then placed in special clamps. Then the specimens were placed in an incubator at 37°C for one hour (maturation time). After removal from the incubator, the specimens were taken out of the clamps and moulds, and stored individually in separate marked plastic tubes with 5 ml deionized water at a temperature of 22–24°C and at an air humidity of 40–50% for 24 hours.

The antimicrobial compounds Benzalconium Chloride and Cetylpyridinium chloride were first incorporated into the glass ionomer cement’s polyacrylic acid by mixing, and then the powder was added gradually, quantity by quantity, to the previously prepared acid and antimicrobial compound mixture, while care was taken to mix them together until complete saturation. The antimicrobial agent was added in strict portions of 1, 2 and 3% of the weight of the cement. The determination of the concentration (weight) of Benzalkonium Chloride and Cetylpyridinium Chloride was made by measuring the given percentage of the antimicrobial agent with an analytical scale (Mettler AE 200) with a possibility of error less than 0.0001 g. Preceding analyses had determi-
ned the concentrations of 1, 2 and 3% of antimicrobial agents in GIC ChemFlex to be equivalent to 0.0022 g, 0.0044 g and 0.0066 g, respectively, and 0.0032 g, 0.0064 g, and 0.0128 g of the whole cement mass of GIC Fuji IX. The subsequent procedure was conducted just as in the case of the samples without antimicrobial agents. The measurements of the compressive strength were carried out 24 hours after the preparation of the samples.

Determining the Setting-time

This was done with the aid of a Gilmore needle. The Gilmore needle used to determine the setting-time weighed 28 grams. The samples used to determine the setting-time were prepared in the same manner as the samples used to determine the compressive strength, but silicon moulds with a diameter of 4 mm and a height of 1 mm were used in this case. The procedure to determine the setting-time is as follows: After the powder and the liquid from each cement without and with the determined concentrations of antimicrobial components have been mixed, and after the samples have been put in the cylindrical rubber moulds with a diameter of 4 mm and a height of 1 mm, the Gilmore needle is put on the cement’s surface every 15 seconds. When placed on the cement’s surface, a Gilmore needle makes an indentation. The surface is inspected for any indentations by the needle and the time is measured till the moment when the needle stops making any indentations. The first placement of the needle that does not make any indentation on the surface stops the time measurement and the time measured is the setting-time.

Measuring the Compressive Strength

The apparatus used for such an experiment is the same as that used in a tensile test. However, rather than applying a uniaxial tensile load, a uniaxial compressive load is applied. As can be imagined, the specimen (usually cylindrical) is shortened as well as spread laterally. A Stress–strain curve is plotted by the instrument and looks like the following:

![Engineering Stress-Strain curve for a typical specimen](image)

Figure 1 – Engineering Stress-Strain curve for a typical specimen
The compressive strength of the material corresponds to the stress at the red point shown on the curve. Even in a compression test, there is a linear region where the material follows Hooke’s Law. Hence, for this region \( \sigma = E\varepsilon \), where \( E \) refers to the Young’s modulus for compression.

This linear region terminates at what is known as the yield point. Above this point the material behaves plastically and will not return to its original length once the load is removed.

There is a difference between the engineering stress and the true stress. By its basic definition the uniaxial stress is given by:

\[
\sigma = \frac{F}{A}
\]

where, \( F = \) load applied [N], \( A = \) area \([m^2]\)

The specimens for compressive strength determination were prepared according to British Standards Institution Specifications for Dental Glass Ionomer Cements 1989, mentioned above. After preparation, the specimens were stored individually in separate marked plastic tubes with 5 ml of deionised water at a temperature of 22–25°C for 24 hours. The compressive strength was measured with the Instron Universal Testing Machine, model 1193, Instron Corp., Canton, USA, with a cross-head speed of 1 mm/min.

![Figure 2 – Universal Testing Machine (Instron Universal Testing Machine model 1193, Instron Corp., Canton, USA)](image)

When the mechanism is started, the upper cylinder descends with the set cross head speed towards the lower cylinder, which bears the specimen in its central part, until the specimen is broken. A PC equipped with appropriate software to record all parameters of the analysis was used for data acquisition. Since compressive strength determination is a method used to analyse the har-
dness of cements, one can observe on the PC screen a curve having the maximal compressive strength expressed in both newtons (N) and in megapascals (MPa) at the instant when the specimen breaks. The analysis of the results was made in megapascals in accordance with the International System of Units (SI).

**Statistical analysis**

The following statistical tests were applied:
- One Way ANOVA;
- Post-hoc-Tukey honest significant difference (HSD) test;

The Statistica programme was used for data processing.

**Results**

**Determination of the setting time**

Table 2

*Setting time of ChemFlex without and with the addition of antimicrobial agents*

<table>
<thead>
<tr>
<th>ChemFlex + Cetylpyridinium Chloride</th>
<th>ChemFlex + Benzalkonium Chloride</th>
</tr>
</thead>
<tbody>
<tr>
<td>0%</td>
<td>5’ 25”</td>
</tr>
<tr>
<td>1%</td>
<td>4’ 30”</td>
</tr>
<tr>
<td>2%</td>
<td>4’ 45”</td>
</tr>
<tr>
<td>3%</td>
<td>3’ 45”</td>
</tr>
</tbody>
</table>

Table 3

*Setting time of Fuji IX without and with the addition of antimicrobial components*

<table>
<thead>
<tr>
<th>Fuji IX + Cetylpyridinium Chloride</th>
<th>Fuji IX + Benzalkonium Chloride</th>
</tr>
</thead>
<tbody>
<tr>
<td>0%</td>
<td>4’ 40”</td>
</tr>
<tr>
<td>1%</td>
<td>4’ 38”</td>
</tr>
<tr>
<td>2%</td>
<td>4’ 45”</td>
</tr>
<tr>
<td>3%</td>
<td>4’ 25”</td>
</tr>
</tbody>
</table>
## Table 4

*Average values and statistical analysis when determining the compressive strength of GICs (values in MPa)*

<table>
<thead>
<tr>
<th>Antimicrobial Agent</th>
<th>ChemFlex + B. Chloride</th>
<th>ChemFlex + CPC</th>
<th>Fuji IX + B. Chloride</th>
<th>Fuji IX + CPC</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>average ± (St. dev.)</td>
<td>average ± (St. dev.)</td>
<td>average ± (St. dev.)</td>
<td>average ± (St. dev.)</td>
</tr>
<tr>
<td>0%</td>
<td>122.16 (53.50)</td>
<td>146.52 (8.66)</td>
<td>146.29 (8.57)</td>
<td>139.33 (29.62)</td>
</tr>
<tr>
<td>1%</td>
<td>129.64 (8.77)</td>
<td>101.65 (16.57)</td>
<td>137.78 (5.33)</td>
<td>90.10 (10.79)</td>
</tr>
<tr>
<td>2%</td>
<td>121.97 (12.37)</td>
<td>75.35 (13.61)</td>
<td>119.72 (10.78)</td>
<td>77.10 (16.21)</td>
</tr>
<tr>
<td>3%</td>
<td>96.56 (16.33)</td>
<td>66.36 (24.02)</td>
<td>109.51 (7.48)</td>
<td>126.04 (4.14)</td>
</tr>
<tr>
<td>p</td>
<td>0.326122</td>
<td>0.000004</td>
<td>0.000010</td>
<td>0.000119</td>
</tr>
</tbody>
</table>

**Tukey HSD test**

<table>
<thead>
<tr>
<th>Antimicrobial Agent</th>
<th>Significant (p &lt; 0.05)</th>
<th>Significant (p = 0.000)</th>
<th>Significant (p &lt; 0.05)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0%</td>
<td>0% : 1%</td>
<td>0% : 2%</td>
<td>0% : 1%</td>
</tr>
<tr>
<td>0%</td>
<td>0% : 2%</td>
<td>0% : 3%</td>
<td>0% : 2%</td>
</tr>
<tr>
<td>0%</td>
<td>0% : 3%</td>
<td>1% : 2%</td>
<td>1% : 2%</td>
</tr>
<tr>
<td>1%</td>
<td>1% : 2%</td>
<td>2% : 3%</td>
<td></td>
</tr>
</tbody>
</table>

*Chart 1 – Average values of compressive strength of GICs with the addition of antimicrobial agents*
The analysis of the average values of the compressive strength of the samples of GIC Fuji IX with an addition of the Benzalkonium Chloride antimicrobial compound shows a gradual decrease in the values from 146.3 MPa for the 0% antimicrobial compound, down to 110 MPa for the 3% case (Table 4 and Chart 1). The difference which is noted when the concentration of the antimicrobial compound is increased is statistically highly significant for $p<0.05$ (Table 4). According to the Tukey HSD test, the differences in the average values are statistically significant for $p = 0.000$ between the average values 0% with respect to 2% and 3%, 1% with respect to 2% and 3%, and 2% with respect to 3%.

The average values which are exhibited, the samples of the same glassionomer cement but with the addition of Cetylpyridinium Chloride, are compressed, show a drop from 139 MPa with no compound incorporated, to 90 MPa for 1%, and to 77 MPa for 2%, only to rise again to 126 MPa for the 3% antimicrobial compound (Table 4 and Chart 1). The differences in the average values are statistically significant for $p < 0.05$ (Table 4). According to the Tukey HSD test, the difference in the average values is statistically significant for $p < 0.05$ between the average values 0% with respect to 1% and 2%; 1% and 2% with respect to 3%.

The average values which are exhibited when determining the compressive strength of GIC ChemFlex with the addition of Benzalkonium Chloride, show variations during the investigation, with a rise to 129 MPa in the case of the 1% antimicrobial compound, and a drop to 122 MPa in the 2% case, i.e. to 96.5 MPa in the case of the 3% antimicrobial compound (Table 4 and Chart 1). The difference in the average values which is noted when the concentrations are increased is statistically insignificant – $p > 0.05$, $p = 0.33$ (Table 4).

As regards the determination of the compressive strength of the samples of the ChemFlex cement with CPC incorporated, the obtained average values exhibit a continual drop which goes from 146.5 MPa for the cement with no antimicrobial agent added, to 102 MPa for 1%, 75 MPa for 2% and 63.4 MPa for 3% (Table 4 and Chart 1). The statistical analysis carried out through analysis of the variance shows statistically highly significant differences for $p < 0.05$ (Table 4). According to the Tukey HSD test, the differences in the average values are statistically significant for $p < 0.05$ between the average values for 0% with respect to 1%, 2% and 3%, and of 1% with respect to 3%.

The average values when determining the compressive strength of the GICs between ChemFlex + Benzalkonium Chloride, ChemFlex + CPC, Fuji IX + Benzalkonium Chloride and Fuji IX + CPC for all percental concentrations are statistically significant for $p < 0.05$. The difference in the average values for the samples of the GIC with no addition of antimicrobial agents is statistically insignificant (Table 5). According to the Tukey HSD test, the differences in the
average values are statistically significant for $p < 0.05$ between the average values of 1% and 2% Fuji IX + Benzalkonium Chloride with respect to 1% and 2% Fuji IX + CPC, and with respect to 1% and 2% ChemFlex + CPC; 1% and 2% Fuji IX + CPC with respect to 1% and 2% ChemFlex + Benzalkonium Chloride; 1% and 2% ChemFlex + Benzalkonium Chloride with respect to 1% and 2% ChemFlex + CPC. According to the same statistical analysis, there is a statistically significant difference for $p < 0.05$ and between the average values of 3% Fuji IX + Benzalkonium Chloride with respect to 3% ChemFlex + CPC; 3% Fuji IX + CPC with respect to 3% ChemFlex + Benzalkonium Chloride and ChemFlex + CPC; 3% ChemFlex + Benzalkonium Chloride with 3% ChemFlex + CPC (Table 5).

Table 5

<table>
<thead>
<tr>
<th>ANOVA (p &lt; 0.05)</th>
<th>Tukey HSD test (p &lt; 0.05)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0% 0.580838</td>
<td>• Fuji IX + 1% and 2% Benzalkonium Chloride : Fuji IX + 1% and 2% CPC and ChemFlex + 1% and 2% CPC;</td>
</tr>
<tr>
<td>1% 0.000010</td>
<td>• Fuji IX + 1% and 2% CPC : ChemFlex + 1% and 2% Benzalkonium Chloride;</td>
</tr>
<tr>
<td>2% 0.000019</td>
<td>• ChemFlex + 1% and 2% Benzalkonium Chloride : ChemFlex + 1% and 2% CPC;</td>
</tr>
<tr>
<td>3% 0.000102</td>
<td>• Fuji IX + 3% Benzalkonium Chloride : ChemFlex + 3% CPC;</td>
</tr>
<tr>
<td></td>
<td>• Fuji IX + 3% CPC : ChemFlex + 3% Benzalkonium Chloride and ChemFlex + 3% CPC</td>
</tr>
<tr>
<td></td>
<td>• ChemFlex + 3% Benzalkonium Chloride : ChemFlex + 3% CPC</td>
</tr>
</tbody>
</table>

Discussion

The earlier generation of glass-ionomer cements demonstrated a relatively poor compressive strength, and that is why the indications for their application were also limited. ASPA, the first commercially available GIC, had a rather poor compressive strength with an average value of 73 MPa after 30 minutes, with a tendency to a slight increase in the course of 24 hours. [61, 39] The new generations of glass-ionomer cements have much better physical properties which can be used for application in the masticatory region of the deciduous denture as well. For comparison purposes, the compressive strength of the enamel is 384 MPa, and it is 297 MPa for the dentin [63, 61]. For restorative mater-
rials which are used for posterior restorations, compressive strengths in the range of 100 MPa 30 minutes after the preparation of the cement, is considered good [44, 64, 61]. An important factor for the hardness of a cement is also the kind of antimicrobial agent incorporated, as well as the method of its incorporation.

For the determination of the compressive strength the same glass-ionomer cements were used, incorporated with the same concentrations of antimicrobial compounds, as for the chemical analyses. The compressive strength of the cements with no compound incorporated was also determined - a control group. The average value of the compressive strength of the ChemFlex GIC shows a value of 122 MPa for the first and of 146.52 MPa for the second control group. When determining the compressive strength with the Benzalkonium Chloride antimicrobial compound incorporated, the average value for 1% of the compound increases to the value of 129. 64 MPa, to decrease for 2% and 3% continually to the values of 122 MPa for 2%, i.e. 96.56 MPa for 3% of Benzalkonium Chloride. The incorporation of the second antimicrobial compound Cetylpyridinium Chloride gives average values of the compressive strength which continually decrease to the value of 66.36 MPa for 3% of the compound. The statistical processing of the results carried out with the analysis of the variance for ChemFlex with Benzalkonium Chloride added, speaks of a statistically insignificant difference between the investigated concentrations, i.e. the antimicrobial component has no impact on the compressive strength of the glass-ionomer cement. The same statistical method, carried out with the results for the second antimicrobial compound – CPC, shows a statistically highly significant difference between all analysed concentrations. The comparative analysis of the average values for both antimicrobial compounds incorporated in the ChemFlex GIC, shows an averagely larger compressive strength for the incorporated Benzalkonium Chloride compound. What is characteristic of this part of the analyses, is the data on the different average values of the ChemFlex GIC without any antimicrobial compound. The average values for both control groups show statistically significant differences (122 MPa and 146.5 MPa). The reason for this should be looked for in the manner of preparation of the samples (manual preparation), as well as in the placement of the mix into the moulds (possibility of encapsulation of air bubbles). Though the samples were prepared under the same conditions and by the same researcher, the occurrence of such differences is by no means rare because of the role of the human factor.

The incorporation of the same antimicrobial compounds, but into the other analysed cement – Fuji IX, shows high values of compressive strength for the cement with no compound incorporated, which decline with the increase of the concentration. In the case of the compressive strength for Benzalkonium Chloride, for the Fuji IX glass-ionomer cement also the average values with no antimicrobial compounds added, show certain differences. For the case where
Benzalkonium Chloride was incorporated the values decrease from 146 MPa for 0% down to 109.51 MPa for 3% of the compound. On the other hand, the same cement, but with the second antimicrobial agent – Cetylpyridinium Chloride, gives paradoxical results. The values for the additions of 1% and 2% continually decrease in order to drastically increase for 3% of the antimicrobial compound, which is not the case for any other combination. Nevertheless, the average values of the compressive strength are higher for the combination Fuji IX + Benzalkonium Chloride. The statistical analysis of the values (ANOVA) shows the existence of statistically significant differences in the average values.

For each combination, the Tukey HSD test was carried out, providing us with a complete insight into the individual statistical dependence of the concentrations. Namely, the differences in the average values for the combination Fuji IX + Benzalkonium Chloride are statistically significant between the values for 0% with respect to 2% and 3%; 1% with respect to 2% and 3%, as well as 2% with respect to 3%. The differences are insignificant only between 0% and 1%, which means that one percent of the antimicrobial compound has no impact upon the compressive strength of Fuji IX GIC. For the combination of the same cement with CPC, the differences are significant between the average values for 0% with respect to 1% and 2%, as well as for 1% and 2% with respect to 3%. In this case only the incorporation of 3% of CPC does not degrade the compressive strength. Because of the nonexistence of statistically significant differences between the average values with different concentrations for the combination ChemFlex + Benzalkonium Chloride, the Tukey test was not carried out. The same GIC with CPC incorporated, shows significant differences between the combinations 0% with respect to 1%, 2% and 3%, and 1% with respect to 3%. No significant differences are noted between the concentrations of 1% with respect to 2%, and 2% with respect to 3%.

The determination of the compressive strength depends very much on the size of the samples. Mallmann et al. [35] have analysed the compressive strength of samples with different dimensions in accordance with world adopted standards, using samples with sizes of 6 mm × 12 mm and 4 mm × 6 mm. Both conventional and resin-modified GICs were analysed. The samples with smaller dimensions showed values of the compressive strength of about 85% of the compressive strength of the samples with larger dimensions.

The technique of manipulation with glass-ionomer cements is quite sensitive and presents an important factor, considering that most often the samples are being mixed manually, the preparation of larger samples is a complicated process, which could bring about higher errors. Hence, when testing the mechanical properties of GICs the use of smaller samples is suggested. In this study samples are used with dimensions in accordance with the ISO/WD 9917–1 standard for dental materials of 1999.
Botelho [11] has investigated the compressive strength of Fuji IX with incorporated cationic antimicrobial compounds, Cetylpyridinium Chloride being one of them, added to the powder, and Benzalkonium Chloride added in the liquid of the cement, in concentrations of 1, 2 and 4%. The samples were 6 mm high × 3 mm in diameter. Though the analysis was done after 7 days (the samples were stored), in contrast to our case where the compressive strength was measured after 24 hours, the results are similar – the addition of antimicrobial components to Fuji IX decreases the compressive strength. Only 1% of Benzalkonium Chloride gives an insignificant difference to the control, which is in accordance with our analyses. In our results, the value of Fuji IX with Benzalkonium Chloride is 146.29 MPa for 0%, as compared to the values of Botelho’s study where it is 210.3 MPa after seven days. Similar also is the relationship with 1% and 2%. With respect to CPC our results are: 0% – 139.33 MPa; 1% – 90.1 MPa; 2% – 77.1 MPa and 3% – 126 MPa, compared to those of Botelho for 0% – 210.3 MPa; 1% – 139.2 MPa; 2% – 95.2 MPa; 4% – 92.23 MPa. It is seen that in both combinations of Fuji IX with antimicrobial components there is a difference in the compressive strength in favour of measurements done after seven days, though 3% of CPC in our study shows a higher compressive strength. The larger compressive strength in the mentioned study [11] is due most probably to the difference in the size of the samples and in duration of storage of the samples. It is known from the literature, Williams and Billington [61], Mitra et al. [39], that the compressive strength for samples stored in a water medium increased with time.

The most frequently used antimicrobial agent for the incorporation in GICs certainly is chlorhexidine, most often as a diacetate, digluconate or hydrochloride. Turkun et al. [54] analyse the effect of chlorhexidine diacetate and chlorhexidine digluconate on the compressive strength of ChemFil using samples with dimensions of 4 mm in diameter × 6 mm in height, in concentrations of 0.5, 1.25 and 2.5%, mixed with the powder of the GIC. If we attempt to draw a parallel with our results, in this case also the higher concentrations produce a significantly lower compressive strength with respect to the control group. The compressive strength varies between 175.12 MPa and 213.55 MPa for different percentages, and the control, i.e. ChemFil 0%, gives a compressive strength of 221.1 MPa. Admittedly, the value of measured compressive strength in our study is lower than that noted in the study of Turkun, but any comparative analysis is inappropriate, considering that both the glass-ionomer cements and the antimicrobial components, as well as the percentile values, are different.

We might try to draw a parallel with the results of Takahashi et al. [53], who analysed the compressive strength of Fuji IX with the addition of Chlorhexidine diacetate of 1, 2, 3% and Chlorhexidine diacetate of 1% + Chlorhexidine dihydrochloride of 1%, i.e. Chlorhexidine diacetate of 2% + Chlorhexidine dihydrochloride of 2% of the weight of the cement. Under identical conditions of preparation and storage of the samples as in our analyses, the obtained values...
in the above-mentioned paper vary in the range from 220 MPa for 0% to 180 MPa for 3% of Chlorhexidine diacetate. In our study these values vary in the range from 139.33 MPa to 77.1 MPa for Fuji IX with CPC, i.e. from 146.29 MPa to 109.51 MPa for Benzalkonium Chloride. In both cases only when the antimicrobial component was added with 1% is there a significant difference. The differences in the values of the compressive strength are most probably due to the kind of the antimicrobial agent.

The determination of the setting time is a method which is performed with the use of a Gilmore needle with a certain weight (28 g). The same conventional GICs were used in these analyses as in the analyses described so far. The way of preparation of the samples is manual, and taking into consideration that the setting time is de facto the time from the completion of the mixing process to the hardening of the cement, then the method itself is relatively subjective. Therefore, the implementation of this test for each sample has to be individual, i.e. the complete measurements have to be done by a single person. From the results one can see that the setting time of the GICs with no antimicrobial component added varies – 5'25" and 5', i.e. 4'40" and 4'30" (two controls were performed for each measurement). The difference of several seconds is in principle due to the subjective way of interpretation of the test (the instant of cessation of the mixing of the cement, i.e. of the commencement with the measurement of the setting time, the visualisation of the indentations on the sample, the appearance of the erosion of the sample). When analysing the results for ChemFlex with the addition of CPC, a decrease in the setting time can be observed, starting from 5 minutes and 25 seconds for the sample without any addition of antimicrobial agent, up to 3 minutes and 45 seconds for the cement with an addition of 3% of CPC. In the other group of samples – ChemFlex with Benzalkonium Chloride, between 0% and 3% a difference of ten seconds occurs. Our result for the determination of the working time of ChemFlex is in accordance with the one given by the manufacturer – 5 minutes.

With respect to the determination of the setting time for Fuji IX, the combination with the CPC antimicrobial compound shows a difference of 15 seconds between 0% and 3%. The time declines continually with the increase in the concentration. The setting time for the same cement, but with the Benzalkonium Chloride compound shows no differences regarding the increase in the concentration. The data given by the manufacturer for the setting time is two and a half minutes and are much smaller than our results.

**Conclusion**

- The compressive strength decreases with the increase in the concentration of the antimicrobial compounds, except in the case of the combination ChemFlex + Benzalkonium Chloride.
• The combinations of both GICs with Benzalkonium Chloride, exhibit on average a higher compressive strength with respect to the combinations with Cetylpyridinium Chloride.
• On average, the combination Fuji IX + Benzalkonium Chloride has the highest compressive strength, a the weakest belongs to the combination ChemFlex + CPC.

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REFERENCES


44. Nicholson J.W. Glass-ionomer cements in dentistry: the current position. 2006; A review given by author.


ADDITIONAL REFERENCES


Резиме

ОДРЕДУВАЊЕ НА СИЛА НА КОМПРЕСИЈА И ВРЕМЕ НА ВРЗУВАЊЕ НА ГЛАС-ЈОНОМЕР ЦЕМЕНТИ ИНКОРПОРИРАНИ СО ЦЕТИЛ ПИРИДИНИУМ ХЛОРИД И БЕНЗАЛКОНИУМ ХЛОРИД

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Поради релативно честата појава на рекурентниот карис по реставративниот третман, како и поради огромниот број кариногени микроорганизми присутни во оралната празнина, а кон претставуваат потенцијален ризик-фактор за настанување на нови карисни лезии, сè поголемо внимание сè насочува кон терапевтските антимикробни аспекти на реставративните материјали. Како најприфатливите реставративни материјали кои ги поседуваат позитивните особини на флуорот во процесот на реминерализација и неговото антимикробно дејство, сè истакнуваат глас-јономер цементите. Покрај ослободувањето на флуоридните јони, ГЦЧ и потенцијално можат да бидат искористени како матрици за ослободување на други активни антимикробни состојки. Додавањето на антимикробни компоненти во глас-јономер цементите и анализата на физички својствата на така добиените цементи, сè многу важни особено при користење на тие материјали во постериорната регија на млечните заби. Целта на оваа студија е анализи на физичките карактеристики на комерцијални глас-јономер цементи – Fuji IX и ChemFlex со додаток на анти-
Compressive strength and setting time determination of glass-ionomer cements incorporated with cetylpyridinium chloride and benzalkonium chloride has not already been published and is not under consideration by any other publication. This paper has been considered and approved by all co-authors, and there has not been any conflict of interests by the authors.

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