

Effect of chlorhexidine gluconate on porosity and compressive strength of a glass ionomer cement

Efeito da adição de gluconato de clorexidina na porosidade e resistência à compressão de um cimento de ionômero de vidro

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Resumo

Introdução: Por apresentar ampla atividade antibacteriana, a clorexidina (CHX) tem sido amplamente utilizada em odontologia, podendo ser facilmente incorporada ao cimento de ionômero de vidro (CIV) e liberada consequentemente na cavidade bucal. **Objetivo:** O objetivo neste estudo foi avaliar a porosidade e resistência à compressão de um CIV, ao qual foi adicionado diferentes concentrações de CHX. **Material e método:** Os espécimes foram preparados com CIV (Ketac Molar Esaymix) e divididos em 4 grupos de acordo com a concentração de CHX: controle, 0,5% e 1% e 2% (n=10). Para análise dos poros os espécimes foram fraturados com auxílio de martelo e cinzel cirúrgicos, de modo que a fratura era realizada no centro do corpo de prova, dividindo-o ao meio e as imagens obtidas no microscópio eletrônico de varredura (MEV) analisadas no software Image J. O teste de resistência à compressão foi realizado na máquina de ensaios mecânicos (EMIC - Equipamentos e Sistemas de Ensaios Ltda, São José dos Pinhais, PR, Brazil). A análise estatística foi realizada por ANOVA, complementada pelo teste de Tukey. Nível de significância adotado de 5%. **Resultado:** Não se observou alteração estatisticamente significativa entre os grupos estudados tanto para o número de poros quanto para a resistência à compressão. **Conclusão:** O uso de CIV associado ao gluconato de CLX a 1% e 2% é a melhor opção para ser utilizada na clínica odontológica.

Descritores: Cimentos de ionômeros de vidro; clorexidina; porosidade.

Abstract

Introduction: For presenting wide antibacterial activity, chlorhexidine (CHX) has been extensively used in dentistry and can be easily incorporated into the glass ionomer cement (GIC) and consequently released into the oral cavity. **Aim:** The aim of this study was porosity and compression strength of a GIC, that was added to different concentrations of CHX. **Material and method:** Specimens were prepared with GIC (Ketac Molar Esaymix) and divided into 4 groups according to the concentration of CHX: control, 0.5% and 1% and 2% (n = 10). For analysis of pores specimens were fractured with the aid of hammer and chisel surgical, so that the fracture was performed in the center of the specimens, dividing it in half and images were obtained from a scanning electron microscope (SEM) analyzed in Image J software. The compressive strength test was conducted in a mechanical testing machine (EMIC - Equipment and Testing Systems Ltd., Joseph of the Pines, PR, Brazil). Statistical analysis was performed by ANOVA, Tukey test. Significance level of 5%. **Result:** No statistically significant changes between the study groups was observed both for the number of pores as well as for the compressive strength. **Conclusion:** The use of GIC associated with CHX gluconate 1% and 2% is the best option to be used in dental practice.

Descriptors: Glass ionomer cements; chlorhexidine; porosity.

INTRODUCTION

Glass ionomer cements (GICs) have the potential of preventing caries disease and possess important properties, such as biocompatibility, chemical adhesiveness to dental structures, fluoride-releasing capacity, reabsorption of fluoride from oral environment¹⁻³. They also have the capacity of re-organising dental tissues and inhibiting bacterial growth⁴.

It is known that chlorhexidine (CHX) can be released into the oral cavity⁵, thus being incorporated to GICs in order to potentialise the antibacterial effect of this material^{5,6}. CHX stands out for its proven effectiveness against wide spectrum of pathogens oral cavity^{7,8} and low toxicity⁹.

Adding CHX to GIC can result in changes in the material's mechanical properties because its micro-structure is affected⁵. Factors such as integrity of the interface between glass particles and polymeric matrix, size of the particles, amount and size of the pores in the material all play an important role in determining the mechanical properties^{7,10} such as compressive strength¹⁰.

Studies have been carried out in order to incorporate antibacterial agents to GICs^{3,5,6,7,11-14}, such as CHX at different concentrations, showing effective results for certain microorganisms under in vitro conditions (*Streptococcus mutans*)^{3,6,12}. Nevertheless, the incorporation of antibacterial agents into restorative materials can result in changes in their physical properties depending the concentration used^{3,5,6}.

Despite the scientific evidence demonstrating changes in some mechanical properties of GICs associated to CHX, the literature shows no study reporting this effect on the porosity of this material. Therefore, the objective of this study was to assess the area and number of pores as well as the compressive strength of a GIC to which different concentrations of CHX were added. The hypothesis to be tested is that the addition of different concentrations of CLX not promote changes in the number and size of pores, as well as the strength of compression resistance.

MATERIAL AND METHOD

Forty specimens, for each testing being conducted, were prepared (3mm height x 6mm diameter) for four experimental groups divided according to the following CHX concentrations: control (no CHX); GIC + 0.5% CHX; GIC + 1% CHX; and GIC + 2% CHX. The material used was a Ketac Molar Easymix cement (3M Espe, Seefeld, Germany) manipulated according to the manufacturer's instructions by using plastic spatula and paper pad at controlled room temperature (23±1°C), air relative humidity of 50±5%, and then inserted into the matrix by using a Centrix syringe (DFL, Rio de Janeiro, RJ, Brazil).

The different concentrations of CHX were obtained from a 20% CHX solution (Sigma Aldrid, Steinheim, Germany) and then added to GIC during their manipulation by using a micro-pipette. After filling the matrices with one increment, the material was covered with polyester tape and a glass plate, on which a 100 gram weight was placed during 40 seconds for accommodation and extravasation of excess material. The

specimens of all groups were prepared by only one person, thus assuring the same characteristics for manipulation and insertion of the material.

Prior to evaluation of the porosity, the samples were stored in an oven for 1 hour at 37°C and 90% relative humidity, then fractured aided surgical chisel and hammer, so that the fracture was performed in percent of the test body, dividing it in half and submitted to scanning electron microscopy (SEM). The resulting images (100x magnification) were analysed by using a Image J software (Rasband WS, Image J; US National Institutes of Health, Bethesda, MD, USA). In order to standardise the area to be analysed, the image was divided into quadrants and the upper left quadrant of each one was selected by using the software tools. Next, pores were delimited and calculated^{15,16}.

For assessment of the compressive strength, the specimens (n=10) were stored in oven for 24 hours at 37°C and 90% relative humidity and then submitted to mechanical testing in a universal testing machine (EMIC - Equipamentos e Sistemas de Ensaios Ltda, São José dos Pinhais, PR, Brazil) with load cell of 5 kN and operating at a cross-speed of 0.5 mm/min.

The values of area and number of pores and compressive strength were analysed in terms of data distribution and variance homogeneity by using the Shapiro-Wilk, Levene, and ANOVA tests. The SPSS version 13.0 software was used for statistics tests at significance level of 5% (P < 0.05).

RESULT

Data on area occupied by pores and number of them as well as on compressive strength of the material are listed in Table 1.

The values of area occupied by pores in the GIC had normal distribution (Shapiro-Wilk's test, p = 0.062) and variance homogeneity (Levene's test, p = 0.366). Adding CHX at different concentrations resulted in no statistically significant change in the area of pores (ANOVA, p = 0.083).

Analysis of the number of pores revealed normal distribution (Shapiro-Wilk's test, p = 0.08) but no variance homogeneity (Levene's test, p = 0.002). Adding CHX at a concentration of 0.5% (Figure 1) decreased significantly the number of pores in the material (ANOVA with Tamhane's correction, p = 0.001). At concentrations of 1% and 2% (Figure 1), no statistically significant differences were observed in the number of pores compared to the control group (Figure 1).

The values of compressive strength had normal distribution (Shapiro-Wilk's test, p=0,776) and variance homogeneity (Levene's test, p = 0,081). Addition of CHX at different concentrations did not change the compressive strength of GIC significantly (ANOVA, p = 0.627)

DISCUSSION

Studies have been conducted to better understand the properties of GICs, with compressive strength test being the most common method used for assessing the resistance of these

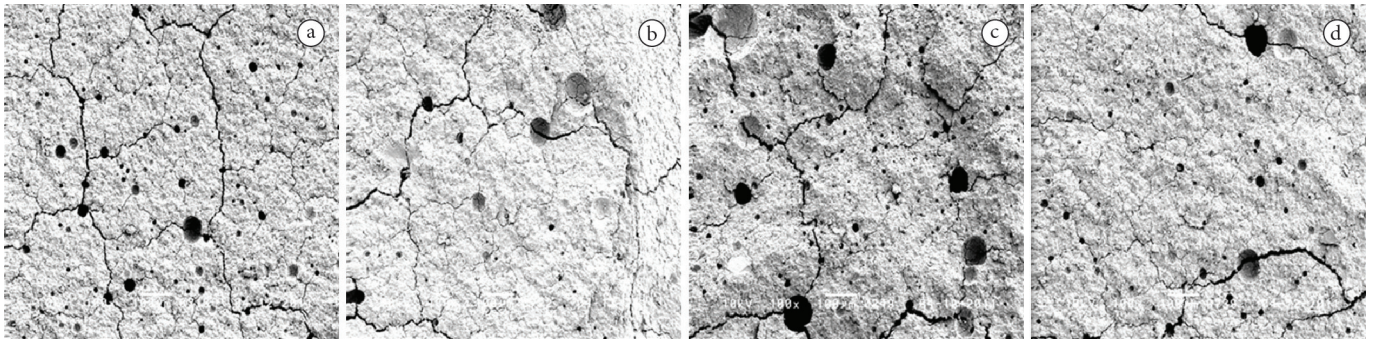


Figure 1. Porosity of the glass-ionomer cement in the control group A. group control; B. group GIC + 0.5% CHX, C. group GIC + 1.0% CHX, D. group GIC+ 2.0%.

Table 1. Means and standard deviations for area occupied by pores (μm^2), number of pores, and compressive strength (mPa) of the glass-ionomer cement (GIC) depending on the concentrations of CHX

Groupos	Pore area (μm^2)	Number of pores ⁺	Compressive Strength (mPa)
GIC	8650,48 ^a ± (2683,08)	55 ^a ± (22)	7,98 ^a ± (1,78)
GIC + 0,5% CHX	6140,96 ^a ± (2018,89)	31 ^b ± (6)	8,31 ^a ± (1,47)
GIC + 1% CHX	8783,36 ^a ± (4021,23)	44 ^a ± (7)	8,15 ^a ± (3,25)
GIC + 2% CHX	9880,15 ^a ± (3743,61)	53 ^a ± (11)	7,12 ^a ± (1,76)

Same letters mean no statistically significant difference ($p > 0.05$). *ANOVA followed by post hoc Tamhane's ($p = 0.001$).

materials¹⁷. In fact, this test allows mechanical integrity of these materials to be assessed¹⁸.

The resistance of GICs is affected by the internal porosity depending on the method of manipulation as well, since hand-mixed cements have bubbles whose diameter is greater than that of the bubbles observed in encapsulated ones¹⁹. The generalised and inherent formation of pores within the GIC reduces its strength to flexion and cohesion^{20,11}. These pores are regions of high concentration of stress, which increased the likelihood of fracture of the material²².

The capacity of the restorative materials to resist to functional forces is an important requisite for its long-term clinical performance. To be clinically accepted, the modified materials must have superior antimicrobial activity and show properties comparable to those of the conventional ones²².

In order to improve the antibacterial properties of the GICs, the addition of CHX to this material has been tested and showed excellent results^{3,10,17,23}. According to Palmer et al.³, GICs have the potential of slowly releasing active agents, such as fluoride²⁴, and the CHX incorporated into GICs can be similarly released into the oral cavity. This association is aimed at reducing severity and frequency of secondary caries by decreasing failures and unsuccessful use of these restorative materials^{6,7,12,25}. Based on the observation of the presence of viable bacteria in the remaining dentin after removal of the infected layer and proper sealing of the cavity researchers observed that under in vitro conditions the addition of CHX to the GIC showed favorable antibacterial effect against microorganisms such as *Streptococcus mutans*, *Lactobacillus* spp., *Candida albicans* and *Actinomyces naeslundii*,

acts on the biofilm act on the biofilm, which begins to show less pathogenic composition compared to acidogenic biofilm with a predominance of *S. mutans*, which would be formed without the presence of the antibacterial agent^{3,9,11-14,23,25}

Türkün et al.³ demonstrated that GIC and CHX at a concentration of 0.5% have a long-term antibacterial effect without compromising the physical properties of the restorative material. However, concentrations at 1.25 and 2% weaken the material by compromising its physical properties, such as resistance to erosion, compression, diametrical traction, flexion, setting time, and surface hardness³. According to Jedrychowski et al.⁷, CHX digluconate increases the antibacterial activity when added to GIC, since it diffuses more rapidly than the cement as the latter is in powder form (CHX diacetate). However, this can change the physical and mechanical properties of the cement as the volume of liquid in the mixture is increased⁷.

The present study demonstrated that although GIC mixed with CHX at a concentration of 0.5% had resulted in no change in the area occupied by pores, the number of pores was decreased. This fact indicates that pores are likely to be larger at this concentration, which causes restorations to be more susceptible to failures or alterations²⁶.

In a longitudinal study of restorations using the ART technique, Frencken et al.²⁴ observed that half of the restorations presented failures. Of these, half of the failures were related to the physical properties of the cement used and its manipulation by the operators. The presence of pores also makes the material more friable^{26,27}.

In order to minimise the effects of porosity, one can use the ultrasonic excitement during the initial setting of the material, thus decreasing number, size and amount of pores. However, the internal porosity of the material is not completely eliminated¹⁵.

With regard to the compressive strength, it was observed in the present study that different concentrations of CHX added to GIC did not change the results of the tests applied, thus corroborating studies conducted by Farret et al.²³. However, Türkün et al.³ observed GIC mixed with CHX at concentrations of 0.5% and 2% presented significant changes in the compressive strength. Jedrychowski et al.⁷ demonstrated that adding CHX

at concentrations higher than 5% had significantly affected the compressive strength of the GIC by decreasing it by 5%.

According to Naasan, Watson¹⁸, testing the compressive strength allows us to know the mechanical integrity of the materials, including the GICs.

CONCLUSION

The use of GIC associated with CHX gluconate 1% and 2% is the best option to be used in dental practice.

REFERENCES

- Mhaville R, Van Amerongen WE, Mandari G. Residual caries and marginal integrity in relation to Class II glass ionomer restorations in primary molars. *Eur Arch Paediatr Dent*. 2006; 7: 81-4. PMID:17140532. <http://dx.doi.org/10.1007/BF03320819>
- Pellegrinetti MB, Imparato JCP, Bressan MC, Pinheiro SL, Echeverria SR. Avaliação da retenção do cimento de ionômero de vidro em cavidades atípicas restauradas pela técnica restauradora atraumática. *Pesqui Bras Odontopediatria Integr*. 2005; 5: 209.
- Türkün LSB, Türkün M, ErtugruL F, Mustafa A, Brugger S. Long-term antibacterial effects and physical properties of a chlorhexidine-containing glass ionomer cement. *J Esthet Restor Dent*. 2008; 20: 29-45. PMID:18237338. <http://dx.doi.org/10.1111/j.1708-8240.2008.00146.x>
- Maltz M, Oliveira E, Fontanella V, Bianchi R. A clinical, microbiologic, and radiographic study of deep caries lesions after incomplete caries removal. *Quintessence Int*. 2002; 33: 151-9. PMID:11890029.
- Palmer G, Jones FH, Billington RW, Pearson GJ. Chlorhexidine release from an experimental glass ionomer cement. *Biomaterials*. 2004; 25: 5423-31. PMID:15130727. <http://dx.doi.org/10.1016/j.biomaterials.2003.12.051>
- Emilson CG. Susceptibility of various microorganisms to chlorhexidine. *Scand J Dent Res*. 1977; 85: 255-65. PMID:266752.
- Jedrychowski J, Caputo A, Kerper S. Antibacterial and mechanical properties of restorative materials combined with chlorhexidines. *J Oral Rehabil*. 1983; 10: 373-81. PMID:6355413. <http://dx.doi.org/10.1111/j.1365-2842.1983.tb00133.x>
- Pucher JJ, Daniel JC. The effects of chlorhexidine digluconate on human fibroblasts in vitro. *J Periodontol*. 1992; 63: 526-32. PMID:1625152. <http://dx.doi.org/10.1902/jop.1992.63.6.526>
- Takahashi Y, Imazato S, Kaneshiro AV, Ebisu S, Frencken JE, Tay FR. Antibacterial effects and physical properties of glass-ionomer cements containing chlorhexidine for the ART approach. *Dent Mater*. 2006; 22: 647-52. PMID:16226806. <http://dx.doi.org/10.1016/j.dental.2005.08.003>
- Xie D, Brantley WA, Culbertson BM, Wang G. Mechanical properties and microstructures of glass-ionomer cements. *Dent Mater*. 2000; 16: 129-38. [http://dx.doi.org/10.1016/S0109-5641\(99\)00093-7](http://dx.doi.org/10.1016/S0109-5641(99)00093-7)
- Botelho MG. Inhibitory effects on selected oral bacteria of antibacterial agents incorporated in a glass ionomer cement. *Caries Res*. 2003; 7: 108-14. <http://dx.doi.org/10.1159/000069019>
- Hoszek A, Ericson D. In vitro fluoride release and the antibacterial effect of glass ionomers containing chlorhexidine gluconate. *Oper Dent*. 2008; 33: 696-701. PMID:19051864. <http://dx.doi.org/10.2341/08-20>
- Ribeiro J e Ericson D. In vitro antibacterial effect of chlorhexidine added to glass-ionomer cements. *Scand J Dent Res*. 1991; 99: 533-40. PMID:1763290.
- Sanders BJ, Gregory RL, Moore K, Avery DR. Antibacterial and physical properties of resin modified glass-ionomers combined with chlorhexidine. *J Oral Rehabil*. 2002; 29: 553-8. PMID:12071924. <http://dx.doi.org/10.1046/j.1365-2842.2002.00876.x>
- Coldebella CR, Santos-Pinto L, Zuanon AC. Effect of ultrasonic excitation on the porosity of glass ionomer cement: A scanning electron microscope evaluation. *Microsc Res Tech*. 2011; 74: 54-7. PMID:21181710. <http://dx.doi.org/10.1002/jemt.20873>
- da Mata M, Santos-Pinto L, Zuanon ACC. Influences of the insertion method in glass ionomer cement porosity. *Microsc Res Tech*. 2012; 75: 667-70. PMID:22298315. <http://dx.doi.org/10.1002/jemt.21109>
- Mallmann A, Ataíde JCO, Amoedo R, Rocha PV, Jacques LB. Compressive strength of glass ionomer cements using different specimen dimensions. *Braz Oral Res*. 2007; 21: 204-8. PMID:17710284. <http://dx.doi.org/10.1590/S1806-83242007000300003>
- Naasan MA, Watson TF. Conventional glass ionomers as posterior restorations: a status report for the American Journal of Dentistry. *Am J Dent*. 1998; 11: 36-45. PMID:9823085.
- Mitchell CA, Douglas WH. Comparison of the porosity of handmixed and capsulated glass-ionomer luting cements. *Biomaterials*. 1997; 18: 1127-31. [http://dx.doi.org/10.1016/S0142-9612\(97\)00038-0](http://dx.doi.org/10.1016/S0142-9612(97)00038-0)
- Arcoria CJ, Butler JR, Wagner MJ, Vitasek BA. Bending strength of Fuji and Ketac glass ionomers after sonication. *J Oral Rehabil*. 1992; 19: 607-13. PMID:1469496. <http://dx.doi.org/10.1111/j.1365-2842.1992.tb01490.x>

21. Nomoto R, McCabe JF. Effect of mixing methods on the compressive strength of glass ionomer cements. *J Dent.* 2001; 29: 205–10. [http://dx.doi.org/10.1016/S0300-5712\(01\)00010-0](http://dx.doi.org/10.1016/S0300-5712(01)00010-0)
22. Geirsson J, Thompson JY, Bayne SC. Porosity evaluation and pore size distribution of a novel directly placed ceramic restorative material. *Dent Mater.* 2004; 20: 987–95. PMID:15501328. <http://dx.doi.org/10.1016/j.dental.2004.07.003>
23. Farret MM, Lima EM, Mota EG, Hugo MS, Oshima HMS, Barth V, de Oliveira SD. Can we add chlorhexidine into glass ionomer cements for band cementation? *Angle Orthod.* 2011; 81: 498-502. PMID:21299380. <http://dx.doi.org/10.2319/090310-518.1>
24. Frencken JE, Makoni F, Sithole WD. ART restorations and glass ionomer sealants in Zimbabwe: Survival after 3 years. *Community Dent Oral Epidemiol.* 1998; 26: 372–81. PMID:9870536. <http://dx.doi.org/10.1111/j.1600-0528.1998.tb01975.x>
25. Frencken JE, Imazato S, Toi C, Mulder J, Mickenausch S, Takahashi Y, Ebisu S. Antibacterial effect of chlorhexidine-containing glass ionomer cement in vivo: a pilot study. *Caries Res.* 2007; 41: 102-07. PMID:17284910. <http://dx.doi.org/10.1159/000098042>
26. Nomoto R, Komoriyama M, McCabe JF, Hirano S. Effect of mixing method on the porosity of encapsulated glass ionomer cement. *Dent Mater.* 2004; 20: 972–78. PMID:15501326. <http://dx.doi.org/10.1016/j.dental.2004.03.001>
27. Fleming GJ, Kenny SM, Barralet JE. The optimisation of the initial viscosity of an encapsulated glass-ionomer restorative following different mechanical mixing regimes. *J Dent.* 2006; 34: 155–63. PMID:16085350. <http://dx.doi.org/10.1016/j.jdent.2005.05.008>

CONFLICTS OF INTERESTS

The authors declare no conflicts of interest.

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