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Original Article

Characterization of Mechanical Properties, Fluoride Release and Colour Stability of Dental Sealants

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Abstract

Objective: To compare the mechanical properties, fluoride release, colour stability and spreading characteristics of glass ionomer cement (GIC), compomer (CO), chemical (CR) and light-cured resin based (LR) fissure sealants. **Material and Methods:** Cylinders were prepared to determine mechanical properties by diametral tensile strength tests (stored for 24h in distilled water at 37°C) and fluoride release (24 hours, 3, 7 and 15 days; deionised water). Disk shaped specimens were prepared to determine colour stability (1 week in pigmenting solution; 37°C; CIE L*a*b*; ΔE). A simplified method of a spreading test was proposed to predict viscosity characteristics of the tested materials. Data were analysed using one-way ANOVA followed by T-test ($p<0.05$). **Results:** Results showed differences between materials regarding all tested properties. CO was statistically more resistant than the other materials ($p<0.05$), but no difference was observed between CR and LR. The fluoride release of GICs was significantly higher than the other materials ($p<0.05$). CO showed lower ΔE than the other materials in the following order: CO<LR<GIC<CR. LR spread more than the other materials. **Conclusion:** All the materials developed to provide adequate preventive sealing tested in this study have particular physicochemical characteristics and should be recommended considering the particularities of each clinical case.

Keywords: Pit and fissure sealants, Physical Properties, Chemical Properties.

Introduction

Dental caries is still the major public health problem in dentistry and a recent publication even shows an increase in global dental caries prevalence [1]. Public health initiatives are well known strategies to revert this scenario and among other actions include the application of pit and fissure sealants. Associated with other strategies, such as fluoridated water and dental health education, sealants are a cost-effective action in preventing caries [2]. This application was developed in the 60's with the purpose to seal deeper parts of pits and fissures, regions that are more vulnerable to develop caries. Within the first two years after molar teeth start erupting, they're still immature and if the biofilm control isn't efficient yet, sealants are then recommended. There are basically two types of pit and fissure sealants: water and polymer based materials. Both types of materials work equally as a mechanical obstacle to cariogenic microorganisms, consequently avoiding dental caries progression.

Conventional glass ionomer sealants have the advantage of releasing fluoride into oral cavity, offering a cariostatic action on adjacent enamel. In addition, even in sealant lost situations, cement remains were still found on the deeper parts of pits and fissures [3], preserving an effective protection against the development of caries. However, glass ionomer cements have inferior retention to enamel surface than resin based fissure sealants [4].

Resin based sealants are applied in combination with acid etching and show excellent penetration [5] and retention characteristics, that can be even improved by the application of a bonding agent prior to the application of the sealing material [6]. In vitro studies show successful marginal sealing ability and microtensile bond strength to enamel of unfilled resin based sealants [4]. On the other hand, glass ionomer cements release active fluoride ions to the surrounding enamel and are less technique sensitive. However, there is no clear evidence [7] about the superiority of glass ionomer cements or resin based sealants in clinical situation. Despite this fact, Frencken [8] observes that glass ionomer cements are chemically more hydrophilic than resin-based materials and are therefore less sensitive to handler's skills in the clinical situation. Thus, based on clinical experience, glass-ionomer cements should rather be used in sealing pits and fissures that cannot be kept absolutely moisture-free, such as in erupting molars or in children with behaviour problems.

Aiming to compare materials with dental sealing indication, this study evaluated the mechanical properties, fluoride release and colour stability of glass ionomer cement (GIC), compomer (CO), chemical cured resin based sealant (CR) and light cured resin based sealant (LR). Study's null hypothesis is that the materials tested do not differ with regards to their properties.

Material and Methods

Four materials recommended as dental sealants were used in this study. Commercial name, composition and activation source of each sealant are outlined on Table 1. Both resin-based sealants used in this study were not filled.

Table 1. List of materials used in this study.

Material	Product	Composition	Activation
Glass ionomer cement (GIC)	VITRO FIL (DFL, Brazil)	Glass powder and aqueous solution of poly (acrylic acid)	Chemical
Chemical cured resin (CR)	ALPHA SEAL AUTO (DFL, Brazil)	A: BisGMA, TEGDMA, activator and stabilizers K: BisGMA, TEGDMA, initiator and stabilizers	Chemical
Light cured resin (LR)	ALPHA SEAL LIGHT (DFL, Brazil)	BisGMA, TEGDMA, canphorquinone and stabilizers	Light
Compomer (CO)	VITRO SEAL ALPHA (DFL, Brazil)	BisGMA, TEGDMA, *poly* (*acrylic* acid-co-*maleic* acid), catalyst, stabilizers, pigments, glass ionomer, silica and canphorquinone	Light

All samples were prepared according to manufacturer instructions. GIC was prepared with 1.2:1 powder:liquid ratio. CR was prepared mixing equal parts of pastes A and K. LR and CO were light activated using a Light Emitting Diode (LED) device (Radii-plus, SDI, Australia) during 20 seconds for each sample size. All samples were included in clean moulds and covered with polyester strips on both sides for standardised surfaces, no polishing was performed.

Diametral Tensile Test

Cylindrical samples (n=6) with 4mm diameter and 6mm height were stored in distilled water for 24 hours prior to the test. A universal testing machine (DL-500, EMIC, Brazil) equipped with a 5 kN load cell was used. The samples were tested at 0,75mm/min rate and the maximum load attained was registered. The tensile strength was calculated using the following formula: $\sigma_{strength}$ = (2P/πdl)$\sigma_{\text{strength}} = \left(\frac{2P}{\pi dl}\right)$. Where: $\sigma_{strength}$ is the diametral tensile strength (in MPa), P is the maximum load (in N), d is the sample diameter (in mm) and l is the sample length (in mm).

Fluoride Release

Cylinder samples (n=4) with 4mm diameter and 6mm height (surface area equal to 100,5mm^2) of each material were stored in 10ml of deionised water at 37°C for 24 hours, 3, 7 and 15 days. The amount of fluoride in water was measured using an ion meter (HI 253, HANNA, USA) with a fluorine selective electrode (Digimed, Brazil). Measurements were performed at ambient temperature. For each analysis, water was collected, measured, discarded and new deionised water was added. Each water sample was analysed in triplicate to assure the reliability of the procedure.

Colour Stability

Five discs with 2mm thickness and 10mm diameter for each material were prepared using a metallic mould. Baseline colour was measured according with the CIE L*a*b* using a calibrated spectrophotometer (SP62, X Rite). The discs were arranged vertically in an acrylic support, immersed in 5 ml of a solution of equal parts in weight of soft drink (Coca-cola, Cia Fluminense de refrigerantes, Lot 50012P200411), grape juice (SuFresh, Wow Ind. e Com., Lot 7438-6), chocolate powder (Nescau, Nestlé Brasil, Lot 1040121031), concentrated tea (Mate Real, Moinhos Unidos Brasil, Lot 02/11-02) and concentrated guarana (Guaracamp, Rio Mix Ind. de Bebidas, Lote:004) for one week at 37°C in a dark container. At the end of one week, each disc was removed from the solution, washed with distilled water, dried and the CIE L*a*b* was again recorded. ΔE was calculated as: $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$

Spreading

The consistency of sealants was measured by a modified version of miniature slump test, widely used to characterize the workability of fresh concrete [9]. Two flat and optically clear virgin acrylic plates were used to measure the capacity of the material to flow, indirectly measuring the viscosity. A small amount of material (0,5g) was placed over the centre of the lower plate, on which the diagonals and medians are traced. After 30" from the beginning of homogenization, the upper plate and a weight of 1kg were carefully placed over the assembly. After another 30", the weight was removed and the diameter of the spread material was measured with a calliper by the length of two diagonals over the spread zone of the paste, as shown in Figure 1. Two tests were performed for each material and an average was calculated.

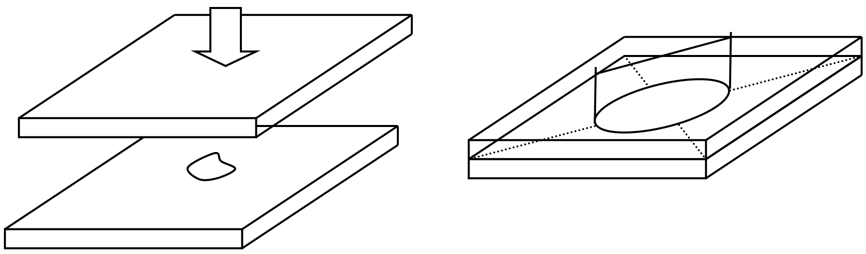

Figure 1. Schematic illustration of the spreading test.

The more viscous the material, less it spreads during the test and a smaller diameter of material is observed between the transparent plates.

Statistical Analysis

One-way ANOVA was used for group comparisons and T-test for group-wise comparisons (StatPlus, AnalystSoft Inc., USA). For all the tests, a maximum value of p=0,05 was used for statistical significance.

Results

Diametral tensile strength of the sealant samples is plot on Figure 2 with respective standard deviation.

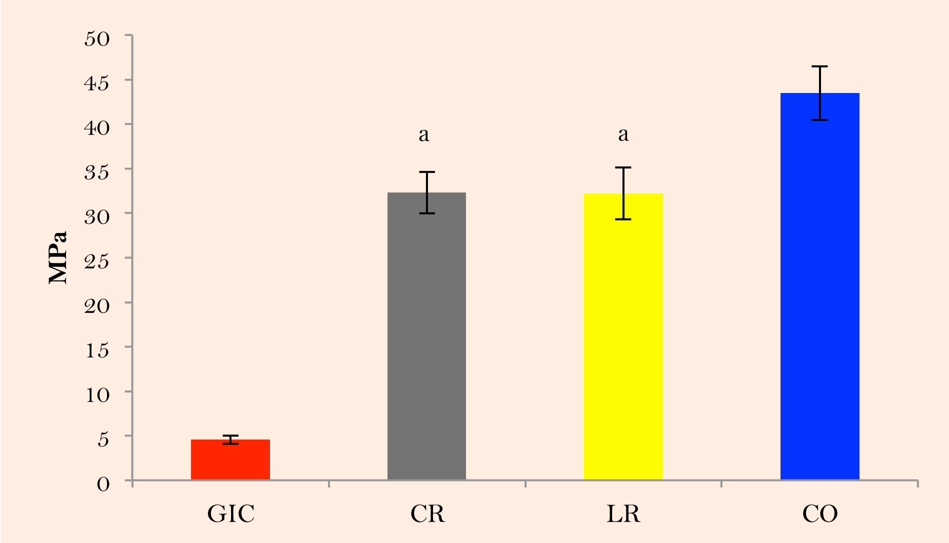

Figure 2. Diametral tensile strength (MPa) for the dental sealants. Means with the same letters are not significant different (p>0,05).

CO was more resistant than the other sealants tested. CR and LR showed similar diametral tensile strengths around 32MPa (± 2, 5). GIC presents significantly lower diametral tensile strength for all the materials tested. According with ANOVA statistical analysis, the diametral tensile strength was different among the materials ($p<0,05$). Most resistant CO and least resistant GIC are statistically different from all the others. On the other hand, CR and LR are not statistically different between each other ($p>0,05$).

Fluoride release profiles are shown on Figure 3.

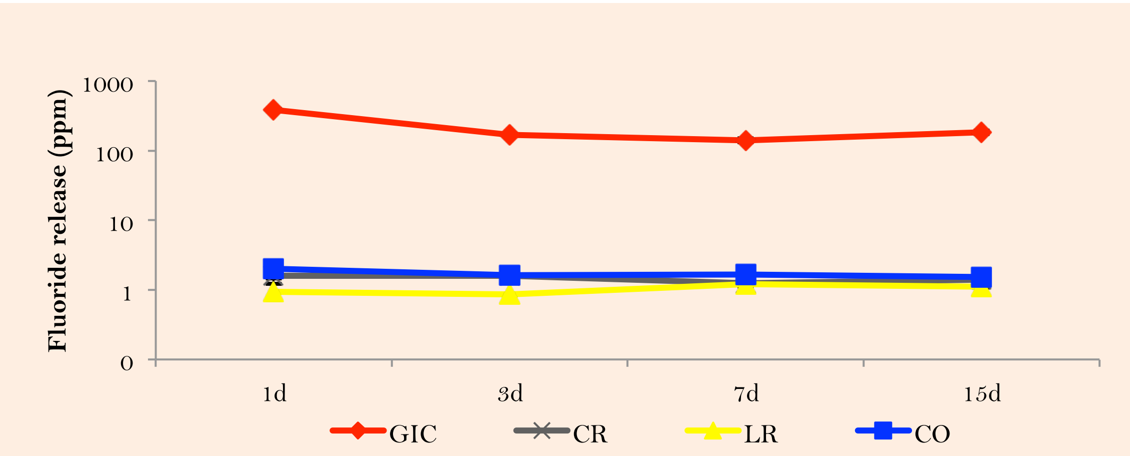

Figure 3. Fluoride release (ppm) of the tested sealants during the study period.

Conventional GIC exhibited greater fluoride releasing pattern compared to CR, LR and CO: more than 500 ppmF^- during the first day and between 100 ppmF^- and 400ppm F^- from the third until the fifteenth day. The other three materials were not statistically different from each other ($p<0.05$) and did not release more than 2ppm of fluoride during the time intervals studied.

Variation in CIE L*a*b* measurements (ΔE) are given in Table 2 together with statistical analysis.

Table 2. Mean ΔE of the sealants tested.

Material	Mean ΔE
GIC	8,05 (±0,4)[a]
CR	11,88 (±1,65)
LR	7,51 (±0,2)[a]
CO	4,9 (±0,2)

Means with the same letters are not significantly different (p>0.02).

One-way ANOVA showed significant difference among the materials tested (p value = 0,05). CO showed the lowest colour variation (mean ΔE=4,9) among the materials tested and CR showed the highest value (mean ΔE=11,88). Despite statistically different mean ΔE between CR and LR materials, of GIC showed a colour variation (mean ΔE=8,05) not statistically different from CR or LR (mean ΔE=7,51).

The average spread of each material tested is plotted in diameter in Figure 4.

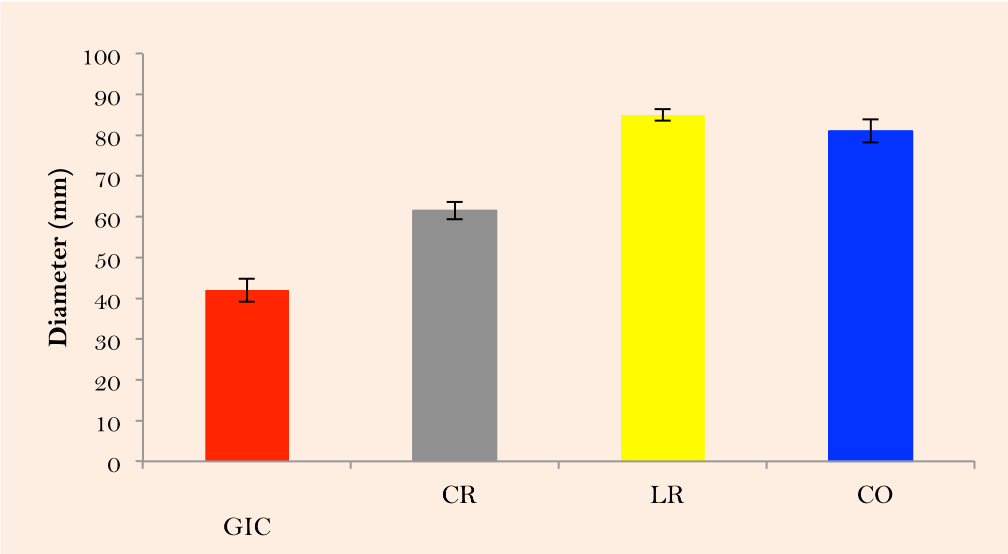

Figure 4. Average diameter of the spread material (mm).

From Figure 4, sealants with self-cured are more viscous than the light cured ones. Among the materials tested, LR was the least viscous and GIC was the most viscous.

Discussion

The relative effectiveness of different types of sealants has yet to be established [6]. Nevertheless, pit and fissure sealants are considered as a very effective mean to prevent dental caries. Clinicians may find many different characteristics between commercially available products: unfilled, filled with various degrees and sizes of particles, tinted in non aesthetic colours or aesthetic shades, with fluoride release and other more or less relevant variables.

The tested null hypothesis was rejected since differences were found between materials regarding all tested properties. In this study, the mechanical properties of glass ionomer cement (GIC), compomer (CO), chemical (CR) and light cured resin based (LR) fissure sealants were measured by diametral tensile strength test. The test consists of applying a compressive force to a cylindrical sample positioned diametrically, resulting in a well distributed perpendicular tension force internally in the body of the sample. It evaluates indirectly the tensile strength of materials and is particularly indicated for materials that are predominantly under elastic deformation [10], like dental resins or glass ionomer cements.

Among the materials studied, CO shows the greater mechanical properties. On the other hand, conventional GICs shows the lowest) strength. Other authors [11, 12] also observed significantly higher strength of resin based dental sealants in comparison to GIC dental sealants.

The greater mechanical strength of compomers can be interpreted by the fact that compomers have their glass particles silanated to bond covalently to the polymer matrix and differently from conventional glass ionomer cements, compomers hardens by acid-base reaction and by photo-initiated matrix polymerisation. Mechanical properties are thus improved in relation to non filled resin based materials, which are not taking advantage of the presence of particles, and in relation to conventional glass ionomer cements.

Previous study analysed the sensitivity of commercially available glass ionomer cement, compomer and filled resin based composite to water and observed that compomers are more resistant than glass ionomer cements at the first stages of tested conditions [13]. Other research [14] tested the flexural strength of traditional resin-based pit and fissure sealants and flowable resin composites, reporting that all the materials tested show non-significantly different values. However, this study compared the mechanical properties of light cured resin based materials.

Regarding fluoride release of dental sealants, it was observed that fluoride release capacity of glass ionomer sealants is higher compared to resin based sealants [15]. In this study, GIC exhibited a significantly higher fluoride-release pattern compared to CR, LR and CO sealants during the study period.

Results of the present study refer to the release of fluoride into deionized water. Other studies show similar pattern of fluoride release at the surface of glass ionomer and resin based materials in other immersion media. Another study [16] observed that GICs release more fluoride than resin based materials. Authors reported that different pH environments significantly affect the rate of fluoride release. However, fluoride release in GICs showed a direct correlation with material degradation observed in microscope images.

Depending on the staining solutions used, i.e., coffee, red wine, juices, or cola soft drink, different ΔE [17,18] are observed for the dental materials. In this study, the pigmenting solution was prepared with products that could be easily found in dietary habits of children.

The results show that chemically cured resin CR experienced the most intense colour change in comparison to the other materials tested. This is also in accordance with other studies [19,20].

Chemically cured composites discolour to dark yellow or brown while the light cured composites discoloured only slightly. Chemically cured composites have lower degree of conversion than the light cured ones, suggesting a more intense susceptibility to colour change. Intrinsic characteristics include physical-chemical reactions within the material, involving colour change of parts of the material itself. Extrinsic factors include the accumulation of staining substances and plaque over the material related to dietary, smoking and hygiene habits.

Regarding the two light curing materials, the compomer showed significantly lower colour change in comparison to the unfilled light cured resin. Factors such as material composition, filler size and presence of hydrophilic methacrylates are often related to the susceptibility of a material to colour change. Light cured unfilled resins are shown in the literature [21,22] to have greater colour stability than conventional composites and that could be explained by the presence of the silane surrounding the composite particles. Silanes have high water absorption levels. Other authors [23] also found that conventional glass ionomer cements are more susceptible to colour change than compomers.

Variations on viscosity may influence the penetration of dental sealants into deeper regions of pits and fissures and material layer thickness. In a recent review about the clinical application of dental sealants [24], the authors affirm that penetration is an important but yet poorly recognized factor in sealant application and retention and that penetration is inversely proportional to the viscosity. More viscous materials would have a limited flow capacity, while lower viscosities would allow the material to spread more easily. Previous study investigated the formation of tags in enamel from materials with increasing viscosities and found a positive correlation between resin fluidity and tag length [25].

In this study, a simplified test for assessing viscosity of materials was used. Conventional acid-base glass ionomer cement and chemical cured resin showed higher viscosity than light cured materials. That is easily understood once the setting reaction of these materials is continuously developing with the test. According to manufacturer data, glass ionomer cement and chemical cured resin based material have a setting set time of around 3'. Test protocol designs force application at 30' from the beginning of manipulation, giving time for the matrix to establish new network links. However other authors [26] observed that glass ionomer cements present low stress relaxation time and are better capable of reducing the contraction stresses by viscous flow than chemical cured resin based materials.

This study provides technical information about each sealant type and characteristics to clinicians for evidence-based decision-making. Dental sealants need to have enough mechanical properties to withstand physiological mastication but preventive pit and fissure sealing does not seem to require materials with high mechanical properties [13]. As a conclusion to this study, all the materials seem adequate to the clinical indication and can be used according to each patient situation. A distinction can be made between materials depending on the clinical field conditions and the ability to control the presence of saliva and the technique required for the efficient application of the material [27]. If dental anatomy presents very narrow pits and fissures, a sealant with low viscosity such as the light cured resin-based sealant used in this study should be recommended. In the case of high risk of caries, a fluoride therapy with low doses and high frequency is preconized and conventional glass ionomer cement designed for sealant application should be used.

Conclusion

Dental materials specially designed for pit and fissure sealant application have particular physicochemical characteristics that have to be taken into account by clinicians to provide adequate preventive sealing.

The presence of oral clefts impacts the quality of life of children and their families, thus requiring the implementation and maintenance of multidisciplinary interventional strategies for the full reestablishment of children in relation to aesthetics, ensuring psychological support for such individuals.

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