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Influence of niobium pentoxide addition on the properties of glass ionomer cements

Isadora Martini Garcia, Vicente Castelo Branco Leitune, Gabriela De Souza Balbinot, Susana Maria Werner Samuel and Fabrício Mezzomo Collares

Dental Materials Laboratory, Department of Conservative Dentistry, Federal University of Rio Grande do Sul, Porto Alegre, Brazil

ABSTRACT

Objective: To determine the influence of niobium pentoxide (Nb_2O_5) addition on the physical and chemical properties of glass ionomer cements (GICs).
Materials and methods: Five, 10 or 20 wt.% of Nb_2O_5 were incorporated into commercial GICs (Maxxion R, Vitro Molar, Vitro Fil R) and one group of each GIC remained without Nb_2O_5 (control groups). The GICs were evaluated by Knoop hardness, compressive strength, acid erosion, particle size and radiopacity. Data were analyzed by two-way ANOVA followed by Tukey's test.
Results: The addition of 10% and 20% reduced the microhardness of two GICs ($p < .05$). Compressive strength showed no difference among groups ($p > .05$). Nb_2O_5 did not influence Maxxion R and Vitro Fil R regarding the acid erosion test ($p > .05$). Vitro Molar increased its acid erosion with 10% of Nb_2O_5 ($p < .05$). Maxxion R presented 15.78 μm, while Vitro Molar and Vitro Fil R showed 5.14 μm and 6.18 μm, respectively. As the Nb_2O_5 concentration increased, the radiopacity increased for all groups. Vitro Molar and Vitro Fil R did not present significant difference to at least 1 mm aluminum ($p > .05$).
Conclusion: The addition of 5 wt.% Nb_2O_5 did not affect the tested physical and chemical properties of the GICs and improved the radiopacity of one of the cements. These materials are therefore suitable for further testing of biomimetic remineralization properties.

Introduction

Glass ionomer cements (GICs) are widely used in clinical procedures due to its adhesion to dental tissues without prior treatment [1–5] and friendly technique with reduced number of clinical steps.[6] However, GICs present some shortcomings such as high solubility in oral environment and low wear resistance.[4,7] To overcome these issues, the formulation of the glass [8] and the powder:liquid ratio [4,9] have been changed. In addition, inorganic fillers have been incorporated to GICs.[10,11]

The first attempts to increase the filler contents of GICs were the addition of silver–tin amalgam alloy powder [12] with no significant improvement in mechanical properties.[13–15] Fused amalgam was sintered to glass particles,[16] zinc sulfate,[17] zinc oxide,[18] titanium tetrafluoride,[11] ytterbium fluoride, barium sulfate,[19] strontium oxide and strontium fluoride [20] to enhance the mechanical properties and fluoride release. Calcium phosphates were also added to develop GICs with bioactivity.

Besides that, the radiopacity of GICs could be improved to overcome diagnostic of recurrent caries under restorations.[21] Nevertheless, any of these fillers could improve physical and chemical properties with bioactivity.

Niobium pentoxide (Nb_2O_5) is a metal oxide generally presenting a monoclinic form which contributes to enhance the mechanical properties when incorporated to metal alloys.[22] The addition to resin based dental materials has already showed improved the radiopacity [23,24] and microhardness [23] of adhesive systems [23] and root canal sealers.[24] In addition to biocompatibility and to improve chemical and physical properties of materials, Nb_2O_5 presents bioactivity. In previous studies, Nb_2O_5 promoted the growth of hydroxyapatite crystals on its surface when it is in contact with simulated and human saliva [25] or in simulated body fluid.[26] The bioactivity of this oxide could enhance dentin remineralization after caries disease via biomimetic remineralization process.[27] The evaluation of GICs with different Nb_2O_5

Table 1. Commercial GICs and the respective composition provided by the manufacturers.

GIC	Composition
Maxxion R	After manipulated: amino fluorosilicate glass, polycarboxylic acid, calcium fluoride and water.
Vitro Molar	Powder contains: barium aluminum silicate, dehydrated polyacrylic acid and iron oxide.
	Liquid contains: polyacrylic acid, tartaric acid and distilled water.
Vitro Fil R	Powder contains: strontium aluminum silicate, dehydrated polyacrylic acid and iron oxide.
	Liquid contains: polyacrylic acid, tartaric acid and distilled water.

concentrations is essential to develop a material with suitable properties to be future tested by biomimetic remineralization approach. However, to the best of our knowledge, there are no reports on the use of Nb_2O_5 in commercial GICs.

Therefore, the purpose of this study was to determine the influence of different concentrations of Nb_2O_5 addition to commercial GICs' surface hardness, compressive strength, acid erosion and radiopacity.

Materials and methods

Three different restorative GICs were used: Maxxion R (FGM Produtos Odontológicos, Joinville, SC, Brazil), Vitro Fil R (DFL Indústria e Comércio S.A., Rio de Janeiro, RJ, Brazil) and Vitro Molar (DFL Indústria e Comércio S.A., Rio de Janeiro, RJ, Brazil). The composition of the GICs is shown in Table 1. A high purity Nb_2O_5 (CBMM, Araxá, MG, Brazil) presenting a mean particle size of 38.16 μm was used as inorganic filler to GICs. The GICs powder and liquid were weighted by analytical balance (AUW220D, Shimadzu, Kyoto, Japan) using the volume according to manufactures' instructions. The mean masses of powder and liquid were calculated through three measurements to use the same powder:liquid ratio indicated by manufactures. To obtain the experimental groups, 5 wt.%, 10 wt.% and 20 wt.% of GICs powder was replaced by Nb_2O_5. A group without addition of Nb_2O_5 was used as control for each commercial GIC. The GICs powder was hand mixed with Nb_2O_5 and manipulated with the liquid according to manufactures' instructions.

Surface microhardness

Five samples per group with 5.0 ± 0.5 mm in diameter and 1.0 mm ±0.2 thickness were prepared to evaluate GICs' surface microhardness. The samples were included in a poly (methyl methacrylate) (PMMA) mould and polished with abrasive paper (#600 and #1200 grit SiC) and felt disc saturated with an alumina suspension (Alumina 1.0 μm, Arotec, Cotia, SP, Brazil). The samples were subjected to a microhardness test in a digital microhardness tester (HMV 2, Shimadzu, Tokyo, Japan) by means of three indentations per sample with 50 g per 10 s each one. The length of the larger diagonal was measured and the Knoop hardness was calculated with the following equation:

$$\text{Knoop hardness} = \frac{14{,}228.c}{d^2}$$

where 14,228 is a constant; c is the load (g) and d is the length of the larger diagonal (μm). The Knoop hardness was the mean of the three indentations for each sample and the values were expressed in Knoop hardness number (KHN).

Compressive strength

Compressive strength test was conducted according to ISO 9917-1,[28] except for the sample sizes. Five samples per group presenting 6.0 ± 0.1 mm diameter and 12.0 ± 0.1 mm height were produced in a stainless steel mould to compressive strength assay. The samples were stored in a 100% moisture environment in a cabinet at 37 °C for 24 h. The diameter of the samples was calculated by taking the mean of two measurements using a digital caliper. The samples were placed between the plates of mechanical tester and a compressive load along the diametric axis of the sample with a cross head speed of 1 mm/min was applied. The maximum force applied, when the sample fractures were recorded, and the compressive strength (C) were calculated in MPa using the following equation:

$$C = \frac{4p}{\pi d^2}$$

where p is the maximum force applied (N) and d is the measured diameter of the sample (mm).

Acid erosion

Acid erosion test was conducted according to ISO 9917-1.[28] Five samples with 5.0 ± 0.1 mm diameter and 2.0 ± 0.1 mm thickness were produced into a PMMA mould and stored for 24 h at 37 °C in a 100% moisture environment. After 24 h, an abrasive paper (#1200 grit SiC), with continuous water irrigation, was used to flat the samples. The initial depth at

center of each sample was measured in μm with the mould in a plane reference, with a dial gauge. After the initial measurement, the samples were immersed in an eroding solution prepared also according to ISO 9917-1.[28] Before immersions, the pH was checked at 2.74 ± 0.02. The mould with the samples were immerse in 30 mL of eroding solution face upwards during 24 h at 37 °C. After 24 h immersion, the final depth at the center of each sample was measured by the same way of initial depth. The final result is the difference between the initial and the final depth at the center of each sample in μm.

Radiopacity

Acid erosion test was conducted according to ISO 9917-1 [28] except the diameter of the samples and the aluminum step-wedge thickness. Five samples with 5.0 ± 0.5 mm in diameter and 1.0 mm ± 0.2 thickness were prepared. X-ray images were obtained with phosphorous plates in a digital system (VistaScan, Dürr Dental GmbH & Co. KG, Bietigheim-Bissingen, Germany) at 70 kV and 8 mA, with 0.2 s of exposure time and a focus-film distance of 400 mm. One sample from each commercial GIC with the same concentration of Nb_2O_5 was positioned in each film for a total of five films per concentration. An aluminum step-wedge was exposed simultaneously with the samples in all images. The aluminum step-wedge thickness ranged from 1.0 mm to 8.0 mm in increments of 1.0 mm. The images were saved in TIFF format. Digital images were handled with ImageJ software (Wayne Rasband, National Institutes of Health, EUA) and the means and standard deviations of the gray levels (pixel density) of the aluminum step-wedge and the samples were obtained in a standardized area of 1.5 mm^2.

Particle size

The means of particle sizes of the three commercial GICs were evaluated using a laser diffraction particle size analyzer (CILAS 1180 Particle-Size-Analyzer, Compagnie Industrielle des Lasers, Orleans, Loiret, France). The results of the analysis are showed in μm.

Statistical analysis

Data were analyzed by two-way ANOVA (Nb_2O_5 concentration and cement brand) for Knoop microhardness, compressive strength and acid erosion tests and by one-way ANOVA for radiopacity assay (including the radiopacity of 1 and 2 mm Al in the comparisons), followed by Tukey's *post hoc* test. All analyses were performed at a 5% level of significance.

Results

Table 2 shows the Knoop microhardness, the compressive strength and the acid erosion of the GICs. The Knoop microhardness ranged from 39.11 ± 1.08 to 63.09 ± 4.08. Compressive strength ranged from 21.67 ± 4.94 to 31.17 ± 12.45 MPa and it was not influenced by the Nb_2O_5 addition ($p > .05$) and there was no difference among the groups ($p > .05$). The addition of any concentration of Nb_2O_5 in Maxxion R and in Vitro Fil R did not influence the resistance to acid erosion of these cements ($p > .05$). However, the addition of 10% of Nb_2O_5 in Vitro Molar increased the acid degradation ($p < .05$).

The radiopacity values of the experimental groups are presented in Figure 1. The results showed an increase in radiopacity with the increase of Nb_2O_5 concentration. The group of Maxxion R with the addition of 10% of Nb_2O_5 had no statistical difference to

Table 2. Means $\pm$ standard deviation of Knoop microhardness, compressive strength (MPa) and acid erosion (μm) as function of concentration of the Nb_2O_5 added to glass ionomer cements.

	Niobium pentoxide (%)			
	0	5	10	20
Surface hardness (KHN)				
Maxxion R	59.71 ± 4.45^{ABa}	56.25 ± 15.09^{Aa}	$44.1 \pm 5,41^{Bb}$	51.02 ± 4.92^{Aab}
Vitro Molar	61.68 ± 3.43^{Aa}	63.09 ± 4.08^{Aa}	56.15 ± 4.36^{Aa}	40.96 ± 3.18^{Bb}
Vitro Fil R	51.57 ± 3.35^{Ba}	45.34 ± 2.77^{Bab}	39.11 ± 1.08^{Bb}	41.65 ± 4.64^{Bb}
Compressive strength (MPa)				
Maxxion R	30.63 ± 4.62^{Aa}	25.49 ± 5.34^{Aa}	22.49 ± 4.72^{Aa}	21.69 ± 2.27^{Aa}
Vitro Molar	31.17 ± 12.45^{Aa}	21.67 ± 4.94^{Aa}	30.69 ± 4.06^{Aa}	27.99 ± 7.55^{Aa}
Vitro Fil R	24.42 ± 5.90^{Aa}	25.88 ± 7.08^{Aa}	30.11 ± 5.61^{Aa}	24.71 ± 7.22^{Aa}
Acid erosion (μm)				
Maxxion R	22.56 ± 11.52^{Aa}	28.40 ± 1.88^{Aa}	32.05 ± 9.74^{Aa}	35.97 ± 3.00^{Aa}
Vitro Molar	9.09 ± 5.7^{Bb}	22.81 ± 8.86^{Aab}	23.67 ± 9.47^{Aa}	23.11 ± 10.18^{Ba}
Vitro Fil R	18.64 ± 9.01^{ABa}	25.25 ± 2.48^{Aa}	24.33 ± 2.35^{Aa}	21.59 ± 1.70^{Ba}

Same capital letter indicates no statistical difference in same column ($p > .05$).
Same small letter indicates no statistical difference in same row ($p > .05$).
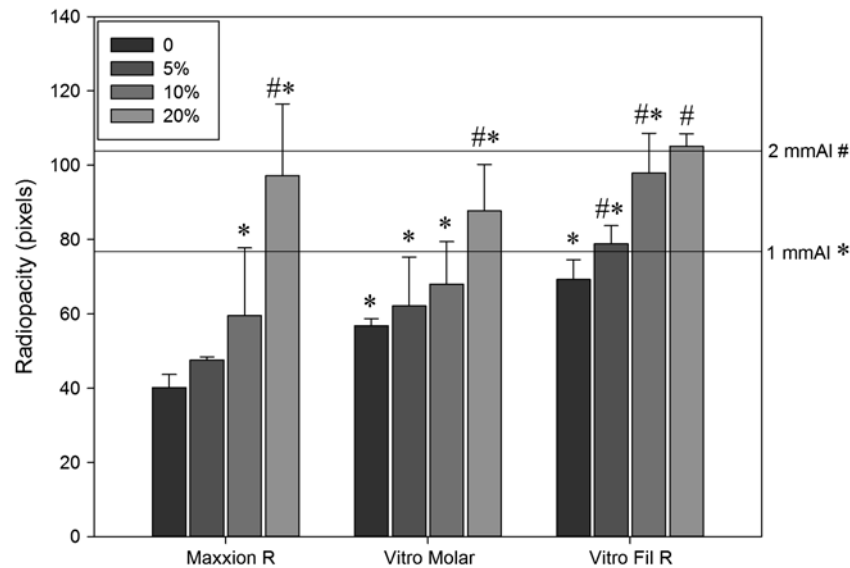
Figure 1. Radiopacity (pixels) as function of concentration of the Nb_2O_5 added to glass ionomer cements. Equal symbols refer to no difference ($p > .05$) against references (1 mm Al or 2 mm Al).

1 mm of aluminum ($p > .05$) and with the addition of 20% of the Nb_2O_5, this cement showed no statistical difference to 2 mm of aluminum ($p > .05$). The group of Vitro Molar without the addition of Nb_2O_5 showed no statistical difference to 1 mm of aluminum ($p > .05$) and with the addition of 20%, this cement had no statistical difference to 2 mm of aluminum ($p > .05$). The group of Vitro Fil R without the addition of Nb_2O_5 also showed no statistical difference to 1 mm of aluminum ($p > .05$), while with the addition of 5% of Nb_2O_5, this cement presented no statistical difference to 2 mm of aluminum ($p > .05$).

The analyses indicated different particles sizes among GICs brands. Maxxion R showed 15.78 μm, the higher mean size, while Vitro Molar and Vitro Fil R presented 5.14 μm and 6.18 μm, respectively.

Discussion

Conventional GICs can be used to restore and seal caries lesion without the need of electric, rotatory and photocuring devices.[29,30] In the present study, it was observed that the addition of 10 wt.% of Nb_2O_5 reduced microhardness values ($p < .05$) of two commercial GICs, while the other cement had it decreased at 20 wt.% of Nb_2O_5 addition ($p < .05$). Higher concentrations of Nb_2O_5 weakened the bulk of the cement and may lead to this result because this oxide is probably not chemically attacked by poly acrylic acid and it is not able to react in acid–basic reaction to take part in GICs setting.

In the current research, there was no statistical difference for compressive strength test ($p > .05$) among the groups with different Nb_2O_5 concentrations or different commercial GICs. GICs compressive strength is related to cross-linking of trivalent ions in the matrix and is a mechanical property of brittle materials related to masticatory forces *in vivo*. The tested filler was not able to interact specially due to its insolubility in the medium. At higher oxide concentrations, the disruption of the glass network [10] or the low wetness of the glass particles by the acid [31] can decrease GICs' physical properties. However, Nb_2O_5 did not altered the compressive strength of the experimental cements in any concentration added.

Vitro Molar presented a better result in acid erosion test. Although it is not possible to evaluate in detail the differences among the compositions of the GICs with the information provided by manufactures, Vitro Molar is a high-viscosity GIC, consequently, it presents smaller particles and more amount of them. In this way, the power/liquid proportion is higher providing better physical behavior.[32] In addition, it was showed that Vitro Molar, when compared to other brands, as Maxxion, presented lower roughness.[33] This result can also be related to the better response of Vitro Molar compared to this GIC, since superficial characteristics influence the perform of the materials in front of acid erosion test. Two commercial cements showed no statistical difference ($p > .05$) between them at the same concentrations of Nb_2O_5. It may be explain due to their similar particle mean size and, consequently, the same volume ratio of Nb_2O_5 incorporated into their glass powder. However, at higher concentrations of Nb_2O_5 (10 wt.% and 20 wt.%), one of the commercial cements showed

significant difference ($p < .05$) to its control group, corroborating with the Knoop microhardness test for the use of Nb_2O_5 5 wt.% concentration. Besides the oxide was not able to react in gelation process, the usual agglomeration of fillers can act as stress-concentrating defect sites, which decrease material properties.[34] Surface hardness and resistance to acid erosion are specially related to this event due to agglomerates debond and surface wear. In the present study, for one GIC, higher amounts than 5 wt.% may have increased the chances of agglomerates formation.

Researches are focused in the improvement of bioactivity of restorative materials besides other properties.[35] GICs have shown to be effective in the remineralization process of dental tissues in ART and it is possible to treat patients with this material based on the philosophy of minimally intervention dentistry. Thus, there is a decrease of the risk of accidental mechanical pulp exposure particularly in young patients.[36] To improve the bioactivity of GICs, calcium phosphates are used [37,38] such as nanocrystals of calcium fluorapatite and hydroxyapatite [39] to act as nuclei to remineralization process after the ionic saturation of the liquid medium.[40] However, it was shown that hydroxyapatite is consumed during GIC setting [39] and may not be available for bioactivity and the release of calcium and phosphate ions lasts a few months.[37] Low concentration of Nb_2O_5 can be an alternative filler for this purpose, since it was observed in this study that higher amounts of Nb_2O_5 decreased the properties of GICs as it could not react during setting. Furthermore, it is well known that the local ion-rich alkaline environ favors apatite formation and matrix phosphorylation.[40] Nb_2O_5 can enhance the environ pH, as it was showed when this oxide was added to bioglass,[41] change the acidic environ of ionomer based materials and turn it more favorable to tooth remineralization.

Materials more easily distinguished from adjacent structures are those with radiopacity slightly higher than enamel.[42] By increasing the radiopacity of the restorative material, it is possible to improve the accuracy of recurrent caries and marfinal defects diagnosis.[21] Nb_2O_5 can enhance radiopacity values of dental materials [23,24] due to its higher atomic number (41) compared to the components around it, as fluorine (9), aluminum (13) and silicon (14). In this study, none of the three GICs tested without Nb_2O_5 reached to 2 mm of Al, corresponding less to 1 mm of enamel ($p < .05$) and all three GICs increased their radiopacity to 2 mm of Al ($p > .05$) at 20 wt.% of Nb_2O_5 incorporation. Nevertheless, Vitro Fil presented the best radiopacity result because it showed no statistical difference ($p > .05$) to 2 mm of Al at low Nb_2O_5 incorporation. Thus, at 5 wt.% of Nb_2O_5 addition, it is possible to increase Vitro Fil radiopacity similar to enamel's value without affecting the other evaluated properties.

The development of GICs with different Nb_2O_5 concentrations was necessary to make it possible a better understanding about the influence of this oxide on the commercial cements. By means of the tests proposed in this study, it was possible to evaluate some of the chemical (acid erosion) and physical (surface hardness, compressive strength and radiopacity) behavior of the developed materials, which can contribute to predict the *in vivo* performance and to reject some of the tested concentrations of Nb_2O_5 addition. It was necessary to create a material with suitable properties to be future tested by biomimetic remineralization approach. The addition of 5 wt.% Nb_2O_5 did not affect the tested physical and chemical properties of the GICs and improved the radiopacity of one of the cements. These materials are therefore suitable for further testing of biomimetic remineralization properties.

Disclosure statement

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