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Influence of dentin conditioning with polyacrylic acid on the shear bond strength of a nano-filled resin-modified glass ionomer cement.

Influencia del acondicionamiento de la dentina con ácido poliacrílico en la resistencia al cizallamiento de un cemento de ionómero de vidrio modificado con resina con tecnología de nano relleno.

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Abstract: Purpose: This *in vitro* study aimed to evaluate the influence of dentin conditioning with polyacrylic acid on the shear bond strength of the nano-filled resin-modified glass ionomer cement Ketac N100 (3MESPE). **Material and methods:** Eighteen bovine incisors were randomly divided into two groups (n=18): group 1, without dentin surface treatment, and group 2, with dentin surface treated with 10% polyacrylic acid for 15 seconds. In both groups the primer was applied before the application of the nano-filled resin-modified glass ionomer cement (Ketac N100) and light-cured for 20 seconds. After 24 hours, the specimens were submitted to thermocycling for 350 cycles, and the teeth were immersed in distilled water at room temperature. After 24 hours, specimens were tested for shear bond strength at 1mm/minute crosshead speed. The collected data were analyzed using the non-parametric test of Mann Whitney (p<0.05). **Results:** There was a significant difference in shear bond strength values between the treatment and control groups, the group with dentin conditioning with 10% polyacrylic acid showed higher shear strength values than the group without dentin treatment. **Conclusion:** Application of 10% polyacrylic acid on dentin increases the shear bond strength values of nano-filled resin-modified glass ionomer cement.

Keywords: Dentin; adhesives; dentistry; glass ionomer cements; shear strength; temperature.

Resumen: Objetivo: Este estudio *in vitro* tuvo como objetivo evaluar la influencia del acondicionamiento de la dentina con ácido poliacrílico sobre la resistencia al cizallamiento del cemento de ionómero de vidrio modificado con resina con tecnología de nano relleno Ketac N100 (3MESPE). **Material y Métodos:** Dieciocho incisivos bovinos se dividieron aleatoriamente en dos grupos (n = 18): el grupo 1, sin tratamiento de la superficie dentinaria, y el grupo 2, con la superficie dentinaria tratada con ácido poliacrílico al 10% durante 15 segundos. En ambos grupos, el Primer se aplicó antes de la aplicación del cemento de ionómero devidrio modificado con resina con tecnología de nano relleno (Ketac N100) y se fotopolimerizó durante 20 segundos. Después de 24 horas, las muestras

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se sometieron a 350 ciclos de termociclado y los dientes se sumergieron en agua destilada a temperatura ambiente. Después de 24 horas, las muestras se evaluaron para determinar la resistencia al cizallamiento a una velocidad constante de 1 mm / minuto. Los datos recolectados fueron analizados mediante la prueba no paramétrica de Mann Whitney (p<0.05). **Resultados:** Hubo una diferencia significativa en los valores de resistencia al cizallamiento entre los grupos de tratamiento y control, el grupo con acondicionamiento de dentina con ácido poliacrílico al 10% mostró valores de resistencia al cizallamiento más altos que el grupo sin tratamiento de la dentina. **Conclusión:** La aplicación de ácido poliacrílico al 10% sobre la dentina aumenta los valores de resistencia al cizallamiento del cemento de ionómero de vidrio modificado con resina con tecnología de nano relleno.

Palabra Clave: *Dentina; adhesivos; odontología; cementos de ionómero vitreo; resistencia al corte; temperatura.*

INTRODUCTION.

The continuous search for new materials and techniques that promote effective bonding between restorative materials and hard dental tissues has been the focus of much research.[1]

From this approach, a material that chemically bonds to hard dental tissues is glass ionomer cement, but unfortunately it also has great limitations compared to resin composites in terms of aesthetics, has low wear resistance and polishing. For these reasons, in the 1990s, there was a tendency to generate hybrid materials from glass ionomer cements and resin composites, thus obtaining resin-modified glass ionomer cement resulting in many improvements.

This tendency to fuse materials was taken to the next level with the incorporation of nano-sized particles into the polymer matrix of resin-modified glass ionomer cement, resulting in a new material called Ketac N100, the first nanofilled resin modified glass ionomer cement with a new technical development combining the benefits of resin-modified glass ionomer cement and the nano-filler technology of resin composite.[2-4]

Regarding the bonding mechanisms of resin-modified glass ionomer cements to dentin, this material has two mechanisms: the first is the chemical bond between the anions of the polyalkenoic acid chains and the calcium ions in hydroxyapatite and the second the micromechanical bond similar to that between dentin-bonding agents and dentin.[5-7] In order to optimize the bonding of the resin modified glass ionomer cements to the dentin pretreatment of this tissue with different substances such as citric, polyacrylic, phosphoric, tannic, and ethylene diamine tetra acetic (EDTA) acids has been suggested.[8-10]

The use of polyacrylic acid as a conditioning agent has been suggested prior to the application of glass ionomer cements (GICs) or resin-modified glass ionomer cements (RMGICs) in order to remove the smear layer and improve the bonding to dentin.[11-13]

The objective of the present research was to evaluate the influence of the polyacrylic acid pretreatment on the shear strength of dentin nano-filled resin-modified glass ionomer cement to the dentin. The null hypothesis tested was that conditioning of dentin with polyacrylic acid does not affect the shear strength of nano-filled resin-modified glass ionomer cement.

MATERIALS AND METHODS.

The methodology used for this *in vitro* study was based on the technical standards ISO/TS 11405 of "Dental Materials – evaluation of adherence to dental structures". In addition, the materials used in this study, their composition and manufacturers. (Table 1)

Sample preparation

The sample preparation was carried out by the researchers. Eighteen freshly extracted mandibular incisors with straight roots, similar shape and sizes from bovines with an average age of 48 months were used. The teeth were cleaned with periodontal curettes and scalpel blades to remove the remnants of the periodontal ligament, washed in tap water and examined with magnifying glasses with a 10X magnification in order to discard teeth with cracks or fractures. All the bovine teeth were submerged in distilled water for 24 hours.

The specimens were then stored in 2% glutaraldehyde for 30 days for disinfection and preservation. They were then washed in tap water and kept in distilled water until the time of the experiment. The roots were sectioned at the level of the middle third, removing the pulp content, filled with red wax and sealed with Coltosol (Coltene Whaledent, Ohio, USA).

The teeth were embedded in a silicone matrix (Speedex, Coltene Whaledent, Ohio, USA) with a rectangular shape 16 x 10 x 10 mm and filled with transparent acrylic resin leaving the dental crown exposed. At the coronal level, 2mm enamel was removed to expose dentin on both the mesial and distal sides, and worn with grain sandpaper (60, 120,180, 220, 320, 400, 600, 800, 1000, 1500); each tooth had the right half (control) and the left half (experimental) properly marked in the acrylic surface.

The 36 preparations were washed and dried with a triple syringe; pumice and water prophylaxis was performed for 10 seconds, washed and dried with absorbent paper, the work area where the restoration would be performed was delimited in the center of the prepared surface by measuring it.[14]

Restorative procedure

The samples were randomly divided into two groups: Group 1 (n=18) without conditioning of the dentin. In this group the restorations were made according to the manufacturer's instructions; first the Ketac Primer was applied using a micro brush to rub for 15 seconds, air blowed for 10 seconds and light cured for 10 seconds. In group 2 (n=18) the dentin was conditioned with polyacrylic acid at 10% during 15 seconds, washed for 30 seconds, dried with absorbent paper and then the Ketac Primer was applied as described above.

The Ketac N 100 light-curing nano-ionomer restorative was dispensed into a mixing block (mixed for 20 seconds), the material was positioned into a matrix to 2x2mm, it was light cured for 20 seconds with a halogen light curing device at an intensity of 700mw/cm^2, and then the matrix was removed and an additional light curing for 20 seconds in all faces was performed. Finally all the restorations were polished with Sof-Lex Discs (3MESPE, St. Paul MN, USA) according to the granulometry of the disc in a decreasing manner until the surface was polished in all specimens, additionally all the specimens were sealed with two layers of transparent nail varnis.[14] (Figure 1 and Figure 2)

Thermocycling Procedure

After 24 hours the specimens were subjected to thermocycling between 5°C and 55°C for 30 seconds at each temperature with a change interval of 10 seconds with a total repetition of 350 cycles to simulate 1 year of function. Finally all the specimens were submerged in distilled water for 24 hours to posteriorly subject the specimens to shear bond strength testing.[14]

Shear bond strength test

The samples were placed in to Amsler universal testing machine (Mitutoyo brand), the test conditions were environmental, through a load cell of 500 Kg, with a minimum reading unit of 1 Kg, each sample was subjected to a shear load at a speed of 1mm/minute until fracture occurred. This procedure was performed by a calibrated lab technician.[14]

Statistical analysis

For statistical analysis, Mann Whitney's nonparametric U test was employed using a level of statistical significance of 5% (p-value=0.05). The differences among groups were analyzed using the statistical software SPSS *version* 21 for Windows (SPSS Inc., Chicago, IL, USA).

RESULTS.

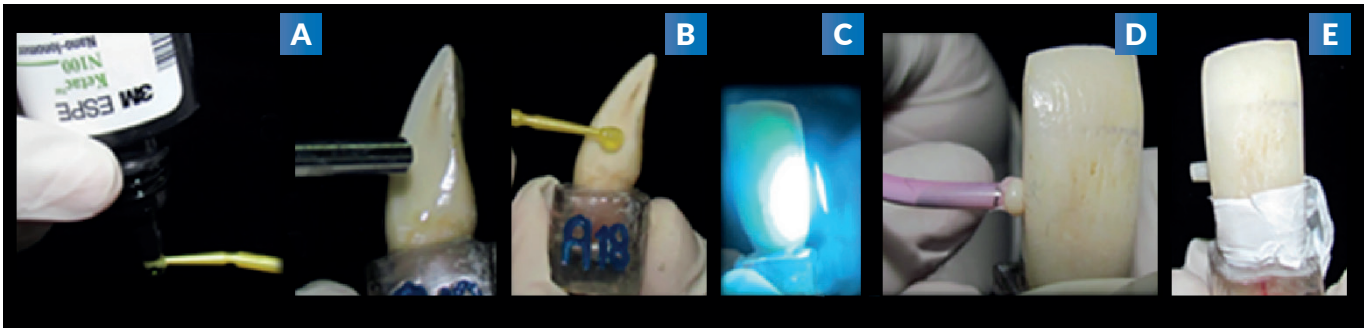
The results from the shear bond strength test were expressed in MPa. Statistically significant differences in shear bond strength were found between the control group without conditioned dentin and the experimental group with dentin conditioned with polyacrylic acid at 10% before the application of the nano-filled resin-modified glass ionomer cement Ketac N 100 (p<0.05).

The value of the Mann-Whitney U test was 33.00, asymptomatically estimated by the standard normal distribution Zc= -4.147, less than Z= -1.96, so there is sufficient statistical evidence to reject the null hypothesis. Therefore, there is a significant influence of the conditioning of the dentin with polyacrylic acid on the shear bond strength of the Ketac N100 nano-filled resin-modified glass ionomer cement, at a 95% confidence level.

The average value for conditioned dentin with polyacrylic acid before the application of Ketac N 100 nano-filled resin-modified glass ionomer cement was 4.69 MPa and the median and mode were 5.00 MPa, and the standard deviation was 1.34 MPa. The minimum value to shear bond strength for the experimental group was 2.00 Kgf/mm^2 and the maximum value for this group was 7.00 MPa.

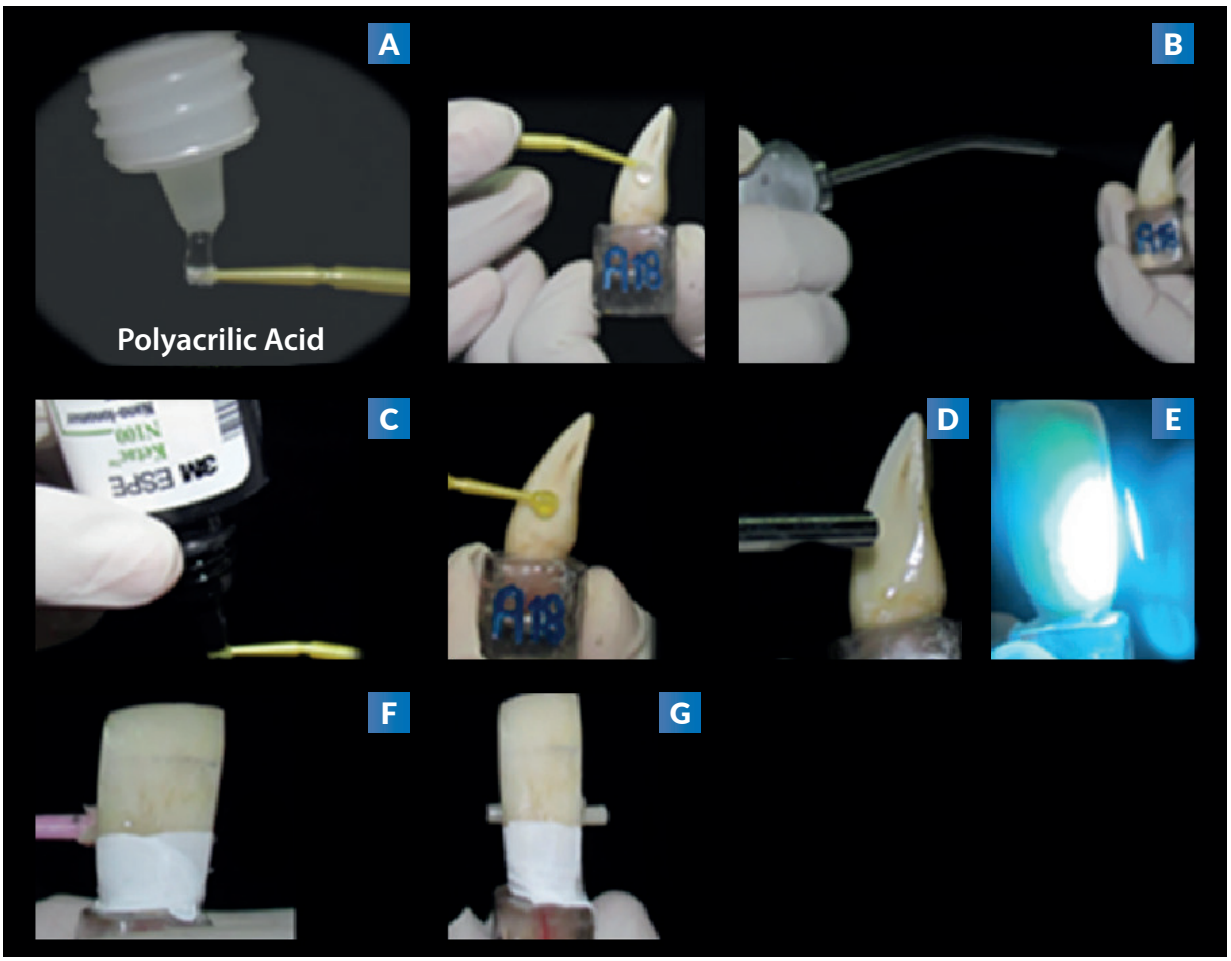
In the control group the average value was 2.05 MPa, the median value was 2.00 MPa and the mode was 1.00 MPa while the standard deviation was 1.51 MPa. The minimum value to shear bond strength in this group was 1.00 MPa and the maximum value was 7.00 MPa. (Table 2)

Figure 1. Restoratives procedures in the control group.



A: Application of the Ketac Nano primer. **B:** Air blowing the Ketac Nano primer. **C:** Light Curing the Ketac Nano primer. **D:** Application of the nano-filled resin-modified glass ionomer cement Ketac N 100 on the dentin surface. **E:** Specimen with the restoration completed on the right side.

Figure 2. Restoratives procedures in the experimental group.



A: Dentin conditioning with polyacrylic acid. **B:** Washed the Polyacrilic acid an dried of the dentin surface. **C:** Application of the Ketac Nano Primer. **D:** Air blowing the Ketac Nano primer. **E:** Light Curing the Ketac Nano Primer. **F:** Application of the nano-filled resin-modified glass ionomer cement Ketac N 100 on the dentin surface. **G:** Specimen with the restoration completed on the left side.

Table 1. Materials used in this study with the composition of materials according to information obtained from the manufacturers.

Material	Description	Composition	Manufacturer	Batch number
Ketac N 100 (light - curing nano- ionomer restorative)	Nano – filled glass ionomer cement.	Paste A: Fluor aluminosilicate glass, HEMA, copolymer of acrylic and itaconic acid, nanoparticles of silica and zirconia, methacrylates and di-methacrylates. Paste B: HEMA, water, copolymer of polyalkenoic acid, nanoparticles of silica and camphorquinone.	3MESPE, St. Paul MN, USA	N223632
Ketac N 100 (nano-ionomer primer)	Dentin conditioner of the nano – filled glass ionomer cement.	Water (40 -50%), HEMA (35- 45%), co-polymerof acrylic and itaconic acid (10 -15%), photo – initiators.	3MESPE, St. Paul MN, USA	N213314
Prothoplast (Liquid)	Liquid compound of the Zinc Polycarboxylate Cement.	Polyacrylic Acid at 10 %.	Subiton Laboratories, Buenos Aires, Argentina.	ADX1139

Table 2. Shear bond strength values expressed in MPa of control and experimental group (dentin surface treated with 10% polyacrylic acid).

	N	Average	Median	Mode	Standard Deviation
Control Group	18	2.05	2.00	1.00	1.51
Experimental Group	18	4.69	5.00	5.00	1.34

DISCUSSION.

The aim of this study was to evaluate the influence of the dentin conditioning with polyacrylic acid at 10% on the shear bond strength of the nano-filled resin-modified glass ionomer cement Ketac N 100 (3MESPE).

When the polyacrylic acid at 10% is used for conditioning the dentin before the application of the nano-filled resin-modified glass ionomer cement Ketac N 100 (3MESPE) this material presents a higher shear bond strength value compared with no conditioning of the dentin. This suggests that conditioning of the dentin with polyacrylic acid prior the placement of the nano-filled resin-modified glass ionomer cement Ketac N 100 (3MESPE) in a cavity is a procedure that should be carried out routinely to improve the bond of this material to dentin.

This result is in agreement with El-Askary *et al.*,[15] who found that the pretreatment step with polyacrylic acid effectively improves bonding of the nano-filled RMGI to dentin. In that study they evaluated an experimental group similar to the present research, in which the dentin surface was conditioned with 25% polyacrylic, posteriorly the Ketac nano primer was applied and finally the nano-filled RMGI Ketac N 100 was applied to the dentin.

Hamama *et al.*,[16] who reported RMGIC conditioning with polyacrylic acid prior to using the nano-filled RMGI Ketac N 100 improves the bonding to dentin and does not adversely affect short-term bond, because only the Ketac N100 primer, which seems not to be a 'true' RMGIC-based material, does not have an adequate conditioning activity on the dentin, affecting the chemical bonding

and the micromechanical retention of the material to the dentin. From this perspective, the negative influence of the smear layer that is generated when preparing the dental cavity and that obliterates the dentinal tubules, and promotes a lack of intimate contact between the material and the dentin, should be highlighted.

According to Mauro *et al*.,[17] the smear layer prevents the intimate contact of the resin-modified glass ionomer cement with the dentin and consequently the chemical and / or physical interactions (micromechanical retention). This was confirmed in this research, as the lowest shear bond strength values were observed when dentin did not receive any treatment prior to the application of Ketac N100 (control group).

Korkmaz *et al*.,[18] evaluated the shear bond strength of a nano-composite, a flowable nano-composite and a nano-filled RMGI to dentin *in vitro*. They showed the use of Ketac nano primer only first to nano-filled RMGI resulted in the lowest shear bond strength values to the dentin. Additionally according to Coutinho *et al*.,[19] the nano-filled RMGI bonded as effectively to dentin as the conventional glass ionomer cement but bonded less effectively than the resin-modified glass ionomer cement. The bonding mechanism should be recognized as the micro-mechanical interlocking provided by the surface roughness, most likely combined with chemical interaction through its acrylic / itaconic acid copolymers.

We suggest to the pretreatment with polyacrylic acid has the ability to remove the smear layer and leave the smear plug, producing a partial demineralization of the dentin, leaving hydroxyapatite around the collagen fibers, allowing the chemical interaction of the carboxylic groups of the resin- modified glass ionomer cement with dentin hydroxyapatite. For mechanical retention the formation of a hybrid layer between the dentin and the resin-modified glass ionomer cement is necessary.

CONCLUSION.

The null hypothesis was rejected as there were significant differences among the studied groups.

Within the limitations of this in vitro study, we conclude that the application of polyacrylic acid at 10 % on dentin increases the shear bond strength values of nano-filled resin-modified glass ionomer cement Ketac N 100. The conditioning of the dentin with polyacrylic acid at 10 % for 15 seconds before the application of the Ketac nano primer and the placement to nano-filled resin-modified glass ionomer cement Ketac N 100 showed the highest shear bond strength to the dentin.

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