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Comparative study of nanomechanical properties of cements used in teeth restoration

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Abstract

The discipline of dental science includes the diagnosis of disease in the mouth and teeth, its manifestations and the procedures involved in the restoration of their integrity and function. Restoration of lost tooth structure with suitable materials plays an integral part in the successful rehabilitation of oral tissues.

Several factors influence the performance of dental restorations. These factors include the type of cement used to bond crown restoration to prepared teeth.

The nanoindentation method was used to explore the mechanical properties of different types of resin cement polymerized using different techniques. A Nano Indenter XP (from MTS Nano Instruments, USA) was used for the experimental tests.

A sample of 40 extracted human teeth were restored using two different resin cements: Variolink II (Ivoclar Vivadent, Liechtenstein) and Venus A2 (Heraeus Kulzer, Germany). Both resin cements are light-cured and one of them is self-cured so that the degree of polymerization would be higher.

The data obtained for nanohardness and the Young's modulus were analysed using ANOVA to evaluate the influence of different factors (the resin cement and polymerization technique used, the position on the tooth–restoration interface) and to determine the best performance for restoration.

The results obtained could give a useful indication of the choice of cementation technique and of the materials used for the restoration of lost tooth structure in different clinical cases.

(Some figures in this article are in colour only in the electronic version)

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1. Introduction

Driven by the growing demand for aesthetic solutions, modern conservative dentistry has turned more frequently to the study of aesthetic restorative materials such as composite resins and porcelain for the morphological and occlusal restoration of posterior teeth, thereby abandoning traditional materials such as gold and silver amalgam.

In restoring very large carious cavities, and especially when it is necessary to cover the cusp of a tooth, the choice for aesthetic restoration involves an indirect technique (inlay/onlay).

The crux of the indirect restoration method lies in the bonding of the pieces, and more precisely, in the interface between the inlay and the cavity walls. At the moment, the market offers a wide variety of resinous materials for bonding and several polymerization techniques, which, when combined, can clinically lead to different results: the objective is to obtain the maximum degree of polymerization, both for the adhesive system and for the cement in order to guarantee the long-term duration of the tooth-restoration unit.

This study has the following objectives:

- to analyse the physical properties of the adhesive interface of an inlay in composite material bonded onto extracted teeth using four different bonding procedures;
- to determine whether there is a relationship between the physical properties of the adhesive interface of an inlay in composite material bonded onto extracted teeth and the variables introduced into the different bonding procedures, i.e. the use of a light-activatable microhybrid composite or a dual cement composite and polymerization of the enamel–dentin adhesive before (pre-polymerization) or after (post-polymerization) interposing the cement and the inlay in the cavity.

2. Materials and methods

2.1. Preparation of the teeth

Forty human teeth were selected (20 molars and 20 premolars), with no caries, extracted from adult patients for periodontal reasons. After extraction, the teeth were preserved in physiological solution to prevent dehydration for a period of between 6 and 30 days.

Part of the root of each tooth was incorporated into a self-polymerizing acrylic resin support perpendicular to its longitudinal axis, below the cement–enamel junction (apically), to facilitate subsequent preparation movements.

Class II MOD (mesio-occlusal–distal) cavities, such as cavities that involve three surfaces of the dental crown, were prepared to receive composite resin inlays using a KR truncated conical cutter (6° convergence; rounded angle). All preparation was then checked on the parallelometer to make sure there were no undercut areas.

The final preparations presented the following characteristics (figure 1):

- convergence between the walls of between 6° and 10°;
- connections between rounded internal surfaces;
- 90° sealing margins;
- interproximal box with cervical–occlusal dimensions of 4 mm (minimum distance of 1 mm from the cement–enamel line), mesio-distal dimension of 2 mm and vestibular–lingual dimension of 4 mm;
- minimum depth and width of isthmus 2 mm.

Cavities with constant dimensions were sought in keeping with the existing anatomical differences between the various teeth.

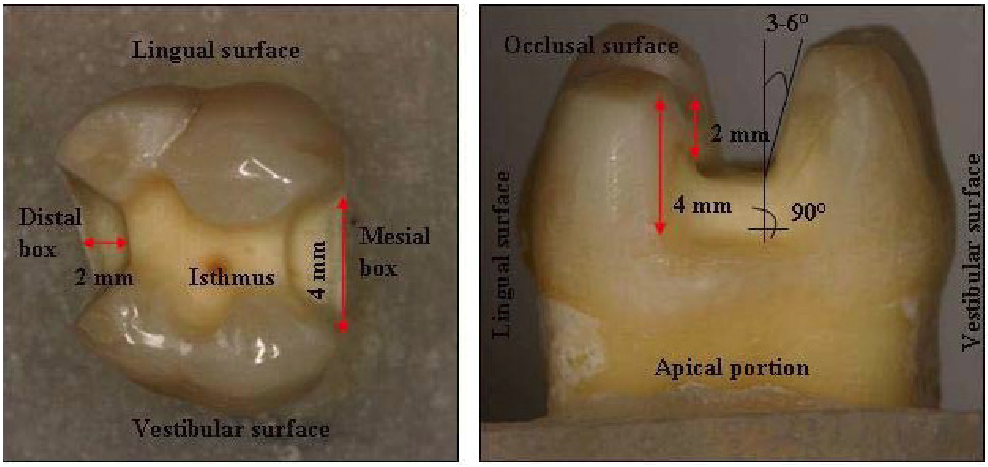

Figure 1. Preparation of Class II MOD (mesio-occlusal-distal) cavities in order to receive composite resin inlays. Each tooth shows two interproximal (mesial and distal) boxes and an isthmus (occlusal).

2.2. Preparation of the inlays

An impression was made of the prepared teeth using an addition silicone (Provil Novo, Heraeus Kulzer, Germany) following the manufacturer's instructions; the low-viscosity impression material was applied to the preparation and the high-viscosity impression was collected in a cylinder suitably sized for a single tooth, showing the entire tooth element by means of the single-phase technique ('sandwich'). Starting from the impression, a model made of plaster was created using an extra-hard IV-type (CG Fujirock EP, Belgium) of plaster mixed mechanically under vacuum for 30 s. After the impression had set (1 h), a coat of plaster hardener was spread over it and left to dry for 15 min.

A spacer varnish was applied to the cavity surfaces to create a die-space of 20 μm up to 1 mm from the edge of the prepared element.

A microhybrid composite (Venus A2, Heraeus Kulzer, Germany) was used for the inlay.

The technique involves layering the material onto the model, followed by pre-hardening using a photopolymerizing lamp (Targis Quick-Ivoclar Vivadent, Liechtenstein) and then polymerization in an oven using heat and light (Targis Power-Ivoclar Vivadent, Liechtenstein).

At the end of this process each item was mounted onto its own tooth element in order to check that there was a proper fit and that there were no imperfections.

Bonding:

The adhesive bonding technique was used for all inlays.

The internal surface of each restoration was treated as follows:

(1) sanding with 50 μm particles;
(2) etching with 37% orthophosphoric acid (Ultra etch, Ultradent Products Inc., South Jordan, UT, USA) for 1 min, rinsing with water and drying with a spray of air;
(3) application of single-component photopolymerizing adhesive (PQ1, Ultradent Products Inc., South Jordan, UT, USA) and spreading using a spray of air.

All teeth were treated as follows:

(1) total etching with 37% orthophosphoric acid for 40 s;

(2) rinsing in water for 10 s and drying in a delicate spray of air;
(3) application of single-component photopolymerizing dentin adhesive (PQ1, Ultradent Products Inc., South Jordan, UT, USA) which was left for 20 s and dispersed using a spray of air.

The 40 samples were divided into four groups of ten teeth, each containing five molars and five premolars.

Group 1: the adhesive was immediately polymerized in the cavity and on the inlay (pre-polymerization) using light from a LED lamp (Ultra-lume LED5, Ultradent Products Inc., South Jordan, UT, USA) for 20 s on each of the two surfaces to be bonded. Only next was the dual composite cement (Variolink II, Ivoclar Vivadent, Liechtenstein) obtained from 1:1 mixing of the base paste and the catalyser; a sufficient quantity of this paste was spread into the cavity and a constant force of 80 N was placed on it using a cylindrical metal weight to ensure proper fitting of the prosthesis. Having removed the surplus bonding, final polymerization was carried out for the times shown (30 s for each surface: occlusal, vestibular, lingual).

Group 2: the adhesive was only polymerized after application of the cement (final post-polymerization). In this case, photopolymerization of the adhesive and the dual composite cement occurred at the same time after inserting the inlay into the cavity (30 s for each of the three surfaces).

Group 3: bonding was carried out by pre-polymerizing the adhesive, but was carried out using the same microhybrid photopolymerizable composite with which the samples were created (Venus A2, Heraeus Kulzer, Germany).

Group 4: as in the previous group, microhybrid photopolymerizable composite was used, but polymerization was carried out at the end (post-polymerized).

2.3. Preparation of the sections

A microtome (Micromet Kermet, Germany) was used, equipped with a diamond blade with a thickness of 300 μm and cooled and lubricated with a mixture of water and mineral oil.

The machine controls the speed of the blade to reduce the heat produced by friction and has a mobile arm, which grasps the sample and is connected to a barometer to ensure a constant and preset pressure.

The sections were made at a standard speed of 700 rpm and with a pressure no greater than 90 Pa to prevent overheating and microfractures, which would otherwise alter the tooth–inlay interface. All the teeth were sectioned along a mesio-distal line going to the centre of the prosthetic restoration.

2.4. Nano indentation test

Specimens were coloured, incorporated inside a resin support (figure 2) and sectioned in order to obtain a flat surface; then they were analysed using a Nano Indenter XP (MTS Nano Instruments, USA). Each tooth was indented on the resin cement along the interface with dentin in 12 points in order to determine the nano hardness and the Young's modulus of the different bondings (figure 3). The Oliver–Pharr model was used for these calculations. According to this model, a load–unload cycle is applied and the maximum force measured on the corresponding indentation area gives the hardness of the specimen, while the slope of the unloading curve gives the Young's modulus.

Each indentation was made using a diamond Berkovich tip; a total maximum load of 100 mN was applied in five intermediate stages of load–unload cycles. The loading time for the surface was 15 s and each peak was held for 30 s. In order to set each indentation

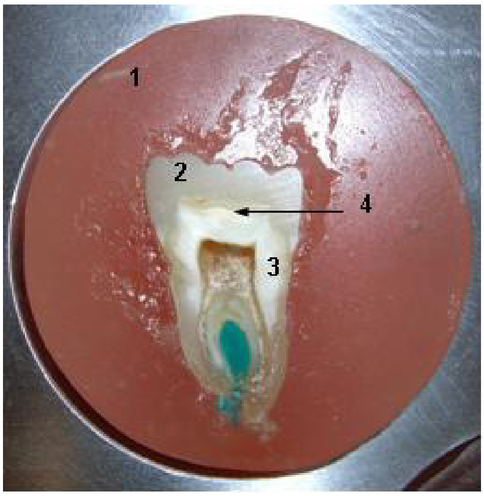

Figure 2. Example of a specimen: each tooth was incorporated inside a resin support (1) and sectioned in order to obtain a flat surface for the indentation test: MOD inlay (2); natural tooth (3); interface (4).

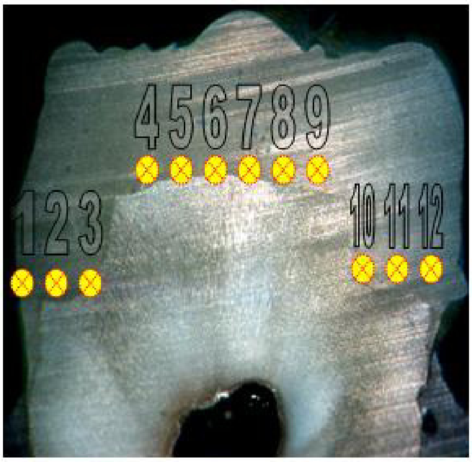

Figure 3. Points at tooth/restoration interface in which nanoindentation has been performed.

on the interface between resin cement and dentin, an optical microscope was used, with a magnification of 40×. In this way, it was possible to indent each tooth in 12 different places: six on the two boxes and six on the isthmus.

The hardness and Young's modulus data provided by the manufacturers was checked by testing three different samples of size 6 mm × 6 mm × 2 mm made of adhesive and resin cements polymerized separately using the same halogen lamp for 2 min.

The results for hardness and the Young's modulus in the resin cements inside the restored teeth were analysed using ANOVA (analysis-of-variance) to determine whether a significant relation exists between the chosen variables. In particular, the influence of three different factors was investigated: the type of resin cement used, the technique used to induce polymerization, and the position of indentation on the tooth.

Factor ‘A’: technique

- A0 stands for two polymerizations;
- A1 stands for one polymerization.

Factor ‘C’: cement

- C0 stands for Venus A2;
- C1 stands for Variolink II.

Factor ‘P’: position

- P0 stands for isthmus;
- P1 stands for box.

Following ANOVA theory, eight cases were singled out, three factors with two levels. Each of them was labelled using a combination of the corresponding levels for each factor.

Subsequently, two types of adhesives were also compared for hardness and the Young’s modulus: namely PQ1 (Ultradent Products Inc., South Jordan, UT, USA) and an adhesive containing cyanoacrylate instead of methacrylate (Loctite Super Glue, Henkel Consumer Adhesives Inc., USA).

In order to make this comparison, eight specimens were made of microhybrid composite (Venus A2, Heraeus Kulzer, Germany) using a metal mould obtained through electro-erosion, to create parallelepipeds measuring 6 mm $\times$ 6 mm $\times$ 2.5 mm. The technique used for the samples involved applying the composite directly into the mould, followed by pre-hardening with a light-curing device (Targis Quick-Ivoclar Vivadent, Liechtenstein) and then optimal polymerization using heat and light (Targis Power oven-Ivoclar, Vivadent, Liechtenstein).

Two samples at a time were bonded during the bonding phase, interfacing them on one of their larger surfaces (6 mm $\times$ 6 mm) in order to obtain a total of four sandwich samples. In all cases the interface surfaces of each sample were etched by applying 37% orthophosphoric acid gel for 40 s, which was eliminated using an abundant spray of water. Next came the application of a thin coating of adhesive, which was spread uniformly using a gentle spray of air.

Following this treatment on each sample to be bonded, subsequent phases set the samples into four groups according to the adhesive and cement used: two samples were given an ethylmethacrylate-based adhesive (PQ1, Ultradent Products Inc., South Jordan, UT, USA) and the other two were assigned cyanoacrylate (Loctite Super Glue, Henkel Consumer Adhesives Inc., USA). For the other variable, two samples used the dual composite cement (Variolink II, Ivoclar Vivadent, Liechtenstein), while the remaining two used a microhybrid photopolymerizable composite (Venus A2, Heraeus Kulzer, Germany). The interface surfaces of the samples were coated with the composite cement, which was held there with a force of 80 N; surplus cement was removed and then post-polymerization was carried out via photopolymerization of the adhesive and the composite cement in one stage after interfacing the two samples, using light from a LED lamp (Ultra-lume LED5, Ultradent Products Inc., South Jordan, UT, USA) for 60 s on each of the surfaces meeting the bonded surface.

The groups were divided as follows:

GROUP 1: PQ1 Adhesive-Variolink II cement;

GROUP 2: PQ1 Adhesive-Venus A2 cement;

GROUP 3: Super Glue Adhesive-Variolink II cement;

GROUP 4: Super Glue Adhesive-Venus A2 cement.

Having been bonded in this way, the pieces were sectioned using the microtome along their sagittal axis in order to view the adhesive–cement interface; after which, each of the four sandwich samples was indented in nine points of the adhesive–composite cement interface in order to measure hardness and the Young’s modulus in the same conditions as the previous experiment.

Table 1. Comparison of the results obtained for hardness and Young's modulus of the investigated cements with the related values given by the producers.

	Hardness expected (GPa)	Average hardness obtained (GPa)	Young's modulus expected (GPa)	Average Young's modulus obtained (GPa)
Cement Venus A2	0.273	0.406 ± 0.03	8.2	10.9 ± 0.62
Cement Variolink II	0.202	0.331 ± 0.02	7.5	9.7 ± 0.74
Adhesive ethylmethacrylate	0.175		5.4	

Table 2. Average values obtained with standard deviation of hardness and Young's modulus for the eight cases labelled according to the ANOVA theory: the first number represents the technique used, the second number represents the type of cement, and the last one the position of the indentation on the tooth.

Case	Hardness (GPa)	Young's modulus (GPa)
100	0.4106 ± 0.04	11.0341 ± 0.89
010	0.3438 ± 0.05	10.6082 ± 1.16
110	0.3292 ± 0.07	9.3785 ± 1.98
001	0.3761 ± 0.09	10.0859 ± 2.04
101	0.3940 ± 0.09	10.5061 ± 2.01
011	0.3467 ± 0.05	10.0070 ± 1.27
111	0.3034 ± 0.07	8.9139 ± 1.57
000	0.4435 ± 0.12	11.9754 ± 2.73

3. Results and discussion

Results of tests performed on adhesive and resin cements polymerized separately, in order to verify hardness and Young's modulus values provided by the manufacturers, and results obtained for hardness and Young's modulus of the resin cements inside restored teeth are shown in table 1. All the obtained values are higher than expected.

Table 2 shows average values with standard deviation of hardness and Young's modulus for the eight cases analysed with ANOVA theory.

The use of ANOVA on the Young's modulus data that was collected (confidence level 98%) shows that the best performance is given by the microhybrid composite resin cement being illuminated twice (figure 4).

With regard to the hardness data (confidence level 95%), ANOVA demonstrates that the technique used to polymerize the cement does not influence the mechanical properties of the tooth–restoration interface (figure 5).

The results are in agreement with the intrinsic properties of the materials used: the microhybrid composite employed for the inlay (Venus A2), if also used as cement, provides better performance because of its higher percentage of inorganic filler and lower organic resin (Bis-GMA) content. Its complete photopolymerization ensures that the desired physical properties are achieved. In a clinical setting, the question is whether light-curing energy will be sufficient to cross materials that are not perfectly translucent or to span increasing distances from the light source. As for hardness, the highest values were found at the isthmus areas located closest to the surface (inlay thickness 2 mm), while the lowest hardness values were found in the deepest parts of the interproximal box (inlay thickness 4 mm).

Though a dual-cured resin cement such as Variolink II (with its low inorganic filler content and higher percentage of TEGDMA organic resin) has lower viscosity and lower mechanical properties, the rationale for its clinical use lies in the fact that it is easier to handle and can

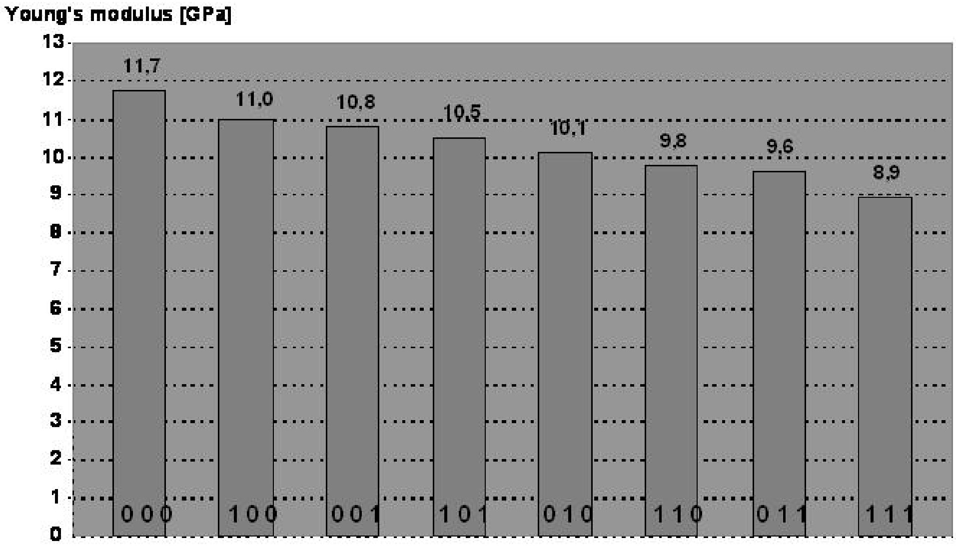

Figure 4. ANOVA results for Young's modulus. The best performance is given by the microhybrid photopolymerizable composite BisGMA illuminated twice.

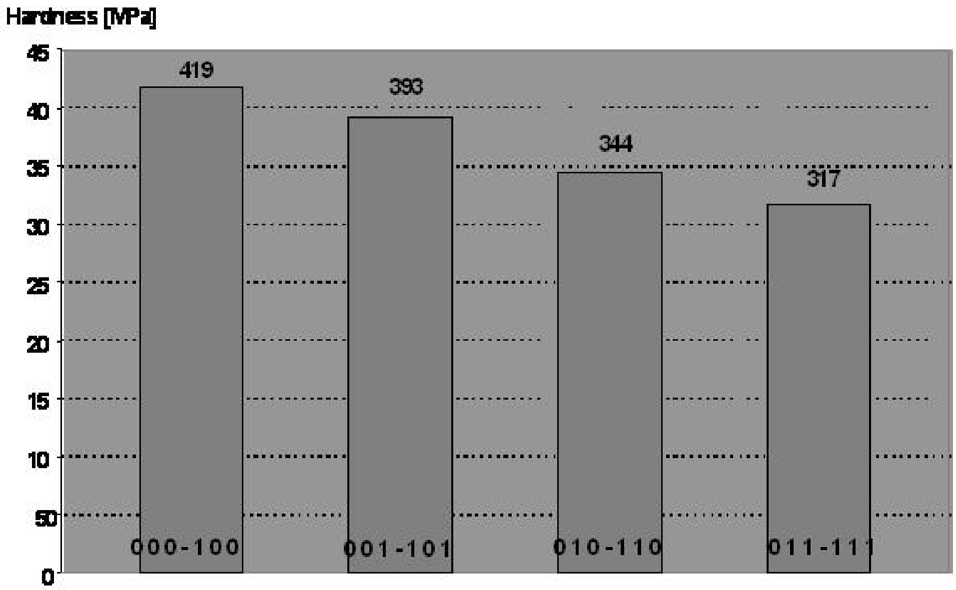

Figure 5. ANOVA results for hardness. The best performance is given by the microhybrid photopolymerizable composite BisGMA. In this case the ANOVA demonstrates that the technique used to polymerize the cement does not influence the mechanical properties of the tooth–restoration interface.

reach full polymerization even in the deepest areas where lamp light may not penetrate with sufficient energy, as is demonstrated by the statistically insignificant differences between the values obtained in the isthmus and the interproximal box.

Table 3. Comparison of the obtained results for hardness and Young's modulus of the investigated cements using two different adhesives. A sample of eight specimens was bonded using PQ1 as adhesive and another one was bonded using cyanoacrylate as adhesive.

Cement	Hardness (ethylmethacrylate) (GPa)	Hardness (cyanoacrylate) (GPa)	Young's modulus (ethylmethacrylate) (GPa)	Young's modulus (cyanoacrylate) (GPa)
Venus A2	0.428 ± 0.15	0.212 ± 0.03	12.5 ± 3.37	8.9 ± 1.02
Variolink II	0.397 ± 0.01	0.197 ± 0.03	13.1 ± 2.70	8.2 ± 1.36

Using a microhybrid composite as cement ensures better mechanical performance, though it also entails clinical complications, as the inlay is more difficult to insert into the cavity because of the composite's high viscosity. If not handled carefully (and, if necessary, heated), the composite can thus hinder complete inlay insertion or lead to the formation of cracks or fractures in the inlay if the latter is pushed with excessive force.

The difference between the actual values obtained for hardness and the Young's modulus of resin cements at the interface with dentin and the expected values is probably due to the increased density of methacrylate chains during polymerization. The need to create a bond between the resin cement and the adhesive leads to a similar chemical composition of the two materials: both of them in fact contain methacrylate, and the interaction between the chains and the consequent creation of cross-links determines adhesion.

At the start, both adhesive and resin cements are liquid solutions of similar monomers, so what probably occurs is that, during polymerization, the actual density of the chains at the interface between the adhesive and cement increases owing to the chain mobility inside a liquid and the attraction related to their chemical similarity. This can lead to a higher hardness and Young's modulus.

In order to demonstrate this phenomenon, two types of adhesives were also compared, as explained in the previous section. The data obtained are shown in table 3. The results for hardness and the Young's modulus in resin cements, for the samples containing the cyanoacrylate adhesive, are similar to those associated with the pure material samples and in line with the data provided by the manufacturers. On the other hand, the results for the samples containing the ethylmethacrylate adhesive gave higher mechanical properties.

4. Conclusion

Several factors influence the performance of dental restorations. These factors include the type of cement used to bond a crown restoration to prepared teeth and the polymerization technique adopted.

The nanoindentation method was used to explore the mechanical properties of different types of resin cement polymerized using different techniques.

A sample of 40 extracted human molars was restored using two different resin cements: Variolink II (Ivoclar Vivadent, Liechtenstein) and Venus A2 (Heraeus Kulzer, Germany).

- The use of ANOVA on the Young's modulus data that was collected shows that the best performance is given by the microhybrid composite, which has been suggested only recently as a cement besides restoration material, illuminated twice.
- With regard to the hardness data, ANOVA demonstrates that the technique used to polymerize the cement does not influence the mechanical properties of the tooth–restoration interface.

- The presence of the same basic monomer inside both the adhesive and the resin cement increases the mechanical properties of the tooth–restoration interface.

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