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In Vitro Study of Mechanical Properties of Teeth Restored with Bulk-fill and Universal Composites Using Different Dentin Adhesives

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ABSTRACT

Background: The most common dental materials are resin composite direct restorative materials and dentin adhesives, which are marketed with different techniques, application recommendations, and compositions, making it difficult to choose the most suitable material and method for different cases. The present study **aimed** to investigate mechanical properties of teeth restored with universal and bulk-fill composites, by using different dentin adhesives and techniques, under in vitro conditions. **Material and methods:** The study was carried out on freshly extracted premolar and molar teeth. After cavity preparation, the teeth were restored with conventional and bulk-fill resin composites, using different adhesive techniques. To assess the effect of the cavity preparation, the direct restoration, and the adhesive protocol on the tooth structure, a transillumination method and Vickers' microhardness measurements were carried out. **Results:** The universal composite showed an average hardness of 55.35 HV at the occlusal level of the restoration, while the bulk-fill composite showed an average of 79.93 HV at the same level. A statistically significant difference was found between the hardness values of the two composites ($p = 0.02$). The transillumination test revealed micro-fissures in the tooth structure in the first phase after cavity preparation and also after polymerization. **Conclusions:** The bulk-fill composite showed higher hardness values than the universal composite. The tested dentin adhesives did not significantly affect the hardness of the dentin at the level of the adhesive interface. Fissures can appear in any phase of the direct restoration, after cavity preparation and polymerization.

Keywords: resin composite, microhardness, dental tissue, mechanical properties, bulk-fill composite

INTRODUCTION

Thorough knowledge of the morphology and histology of teeth and periodontium is essential for an appropriate conservative treatment. Dental caries occur through a process of progressive deterioration of the hard tissues of the tooth. After removal of the carious lesion, the final step in the treatment of caries with conservative rehabilitation of the tooth includes direct tooth restoration.

The history of resin composites as direct esthetic restorative materials began around the 1960s, when Bowen invented the Bis-GMA matrix, mixed with silicate filler. Since then, resin composites have gone through many modifications and improvements, resulting in improved mechanical properties, esthetics, and durability.[1] Their popularity is evidenced by the fact that more than 500 million direct dental restorations are placed around the world each year.[2] However, despite its many advantages, clinicians are still not entirely satisfied resin composites. Therefore, newer types of resin-based materials were introduced in order to reduce the drawbacks encountered in clinical practice.

Universal resin composites are used in 2 mm thick layers, which present a high potential for failure in the lateral areas. A new type of resin, the bulk-fill composite was therefore developed, which can be used in a single increment of 4–5 mm, usually in the lateral areas. Adequate retention must also be ensured when using these materials. Thus, various acids and bonding agents are essential components of resin composite systems. After the complete removal of caries, the next step of the conservative treatment includes acid etching, which produces microretentions on the surface of the dental hard tissues. This is followed by the application of dental adhesives, which create an inseparable mechanical bond between the enamel/dentin and the resin composite. Due to the key role of adhesives, a large number of potential defects can occur during their use, which can impair or make it impossible to achieve an adequate restoration. The type of adhesive and the technique used to create strong bonding between the dental hard tissues and the restorative material, as well as the type of the resin composite have an important impact on future dental restorations. Three basic types of adhesive protocols can be used: total-etch (when enamel and dentin are etched with 37% orthophosphoric acid before applying the bonding agent), selective-etch (when only the enamel is etched before applying the bonding agent), and self-etch (when no etching step is needed, a self-etch type bonding agent is used). The microhardness of the tooth structure may be altered and fissures might appear during these processes.

The present study aimed to investigate the appearance of new fissures in the dental hard tissues and the microhardness level of the restoration, in teeth restored with bulk-fill and universal composites, by using different dentin bonding agents and techniques, under in vitro conditions.

MATERIALS AND METHODS

The study was carried out on freshly extracted premolar and molar teeth. The teeth were removed due to orthodontic and periodontal reasons. To study the appearance of fissures, a transillumination method was used. In the first phase, photographs were taken of the intact teeth using a DSLR camera (Nikon D3100) and a macro lens (Tamron 90 mm) with a magnification ratio of 1:1 and a standardized LED light source. A total of four photographs were taken of each tooth, corresponding to the four surfaces: mesial, distal, buccal, and lingual. The photographs revealed enamel fissures and defects suffered during extraction (Figure 1).

After documentation, mesio-occluso-distal cavities were created. The cavities were standardized, 4 mm deep and 2 mm wide in the bucco-lingual direction. The depth of 4 mm was determined according to the tip of the highest cusp of the tooth so that the actual depth of the cavity varied between 1.5 and 3.5 mm. Standard dimensions were set using a caliper and a periodontal probe.

TABLE 1. The resin composites, adhesives, and techniques used in the different sample groups

Group name	Restoration material used	Adhesive used	Adhesive technique used
Group 1	Filtek™ Bulk-Fill (3M ESPE)	Adper™ Single Bond (3M ESPE)	Total-etch
Group 2	Filtek™ Bulk-Fill (3M ESPE)	Optibond™ Universal (Kerr)	Self-etch
Group 3	Filtek™ Bulk-Fill (3M ESPE)	Optibond™ Universal (Kerr)	Selective-etch
Group 4	Filtek™ Bulk-Fill (3M ESPE)	Gluma Bond Universal (Kulzer)	Self-etch
Group 5	Filtek™ Bulk-Fill (3M ESPE)	Gluma Bond Universal (Kulzer)	Selective-etch
Group 6	Charisma® Classic (Kulzer)	Optibond™ Universal (Kerr)	Self-etch

FIGURE 1. Fissures revealed on different tooth surfaces on photographs taken with the transillumination method

Photographs using the transillumination method were taken again of the prepared teeth, followed by grouping of the teeth according to different dental materials and restoration techniques. We defined a total of six groups, with four molars/premolars in each group. Following the manufacturer's instructions, we used three types of dentin adhesives combined with two different resin composites, as seen in Table 1.

The teeth with fillings were incubated at 37°C in a physiological solution, and photographs were taken using the transillumination method again after 24 hours. All the photographs were assessed qualitatively by two trained operators.

The following step was to place the teeth in a standardized mold made of condensation silicone (Zetaplus, Zhermack) and cast with dental stone (Fujirock Premium, GC). The incubated teeth were sectioned on the middle line in the mesio-distal direction using a low-speed precision cut-off machine (Micracut 151).

Microhardness was measured on the sectioned surfaces using a Vickers Hardness Tester CV-AAT 400. The measurements started each time from the occlusal surface of the resin and progressed gradually toward the dentin adhesive interface (DAI). The first four measurements were performed in the resin composite (occlusal surface, at 0.5 mm, at 1 mm, and at 1.5 mm in the composite), the fifth measurement reached the DAI each time, and the last measurement was performed in the dentin, at 0.5 mm from the DAI. During microhardness testing, we made a pyramidal indentation on the specimens, using a diamond-shaped indenter to apply a load of 50 gf (corresponding to a force of 0.490 N) for 15 s. We measured the diagonals of the indentations using a calibrated optical microscope and evaluated the hardness as the mean stress applied underneath the indenter (Figure 2).

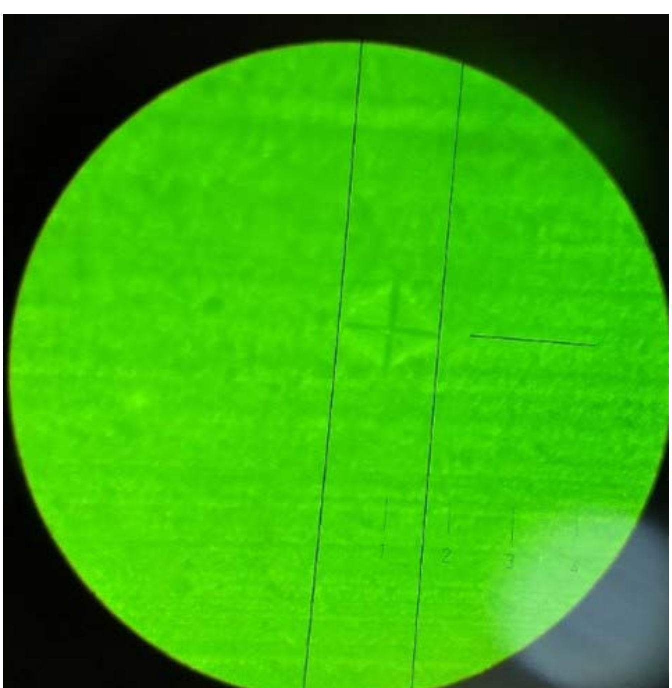


FIGURE 2. Indentation on the surface, observed under the microscope

Statistical analysis

The obtained values were introduced into Microsoft Excel tables, and non-parametric Kruskal-Wallis and Mann-Whitney tests were performed. Pearson's correlation coefficient was calculated using GraphPad Prism software, using a statistically significant threshold of $p < 0.05$.

RESULTS

A total of 144 measurements were performed at different levels of the composite restoration and dentin in the six sample groups (Figure 3).

We found no significant difference between the hardness values measured at the occlusal surface and at a depth of 1.5 mm in the bulk-fill restoration samples ($p > 0.05$). The hardness of the universal composite restoration samples from Group 6 showed significantly lower values at the composite levels compared to the bulk-fill restoration samples ($p = 0.02$) (Figure 4); no significant differences were detected at the dentin level ($p > 0.05$).

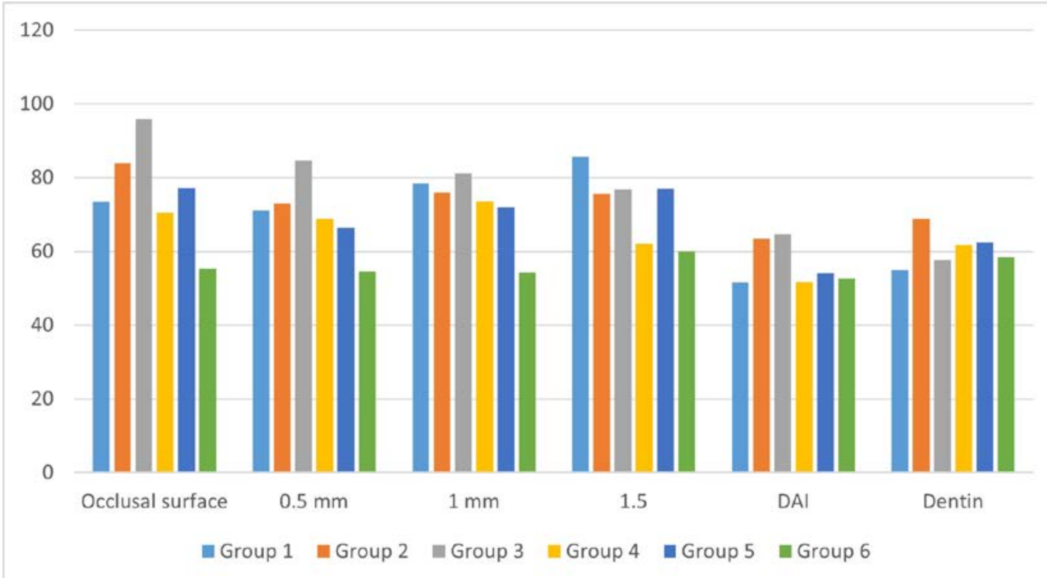


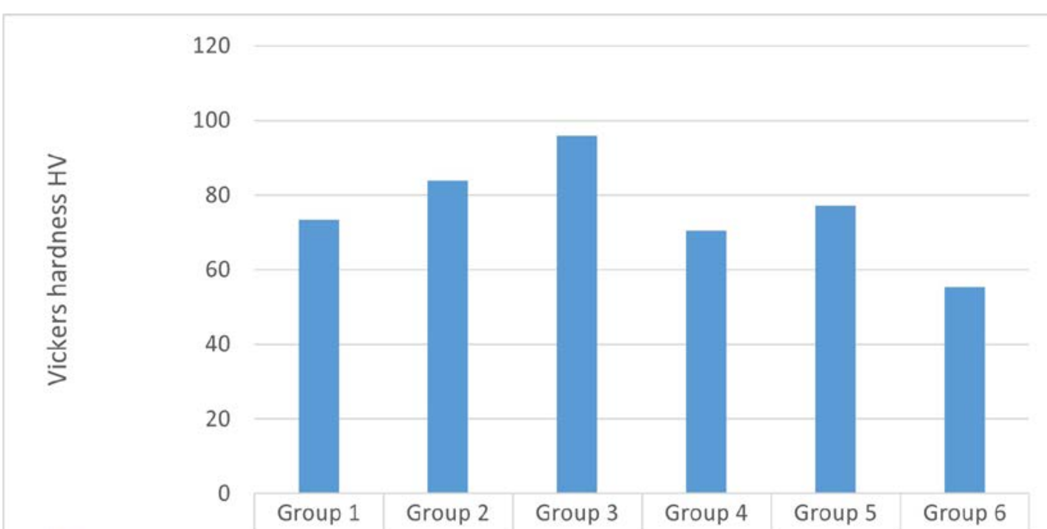
FIGURE 3. Vickers microhardness measurements at different depths

The microhardness values of the dentin levels of different groups restored with bulk-fill composite were compared according to the adhesive protocol used. Selective-etch and self-etch techniques did not show significant differences ($p = 0.475$) in regards to microhardness. The lowest dentin tissue hardness (54.98 HV) was measured in Group 1, where a total-etch technique was used, but this was not significantly lower than in the other groups ($p = 0.149$) (Figure 5). The highest microhardness values (68.88 HV) were obtained in Group 3, where a self-etch protocol was applied by using Optibond™ adhesive material.

At the DAI level, no significant differences were found between groups with different adhesive protocols ($p > 0.05$). The lowest microhardness values (51.63 HV) for the DAI level were found in Group 1.

A positive correlation between the microhardness of the occlusal surface composite level and that of the DAI was detected ($R = 0.56$) in the bulk-fill restored groups. In all cases, the microhardness of the composite layers was higher compared to that of the dentin layers, regardless of the adhesive protocol (Figure 5).

The photographs taken using the transillumination method revealed that the majority of new fissures appeared after cavity preparation. The qualitative analysis of



	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6
Occlusal surface	73.45	83.92	95.92	70.54	77.14	55.35

FIGURE 4. Column graph of mean microhardness values measured at the occlusal surface

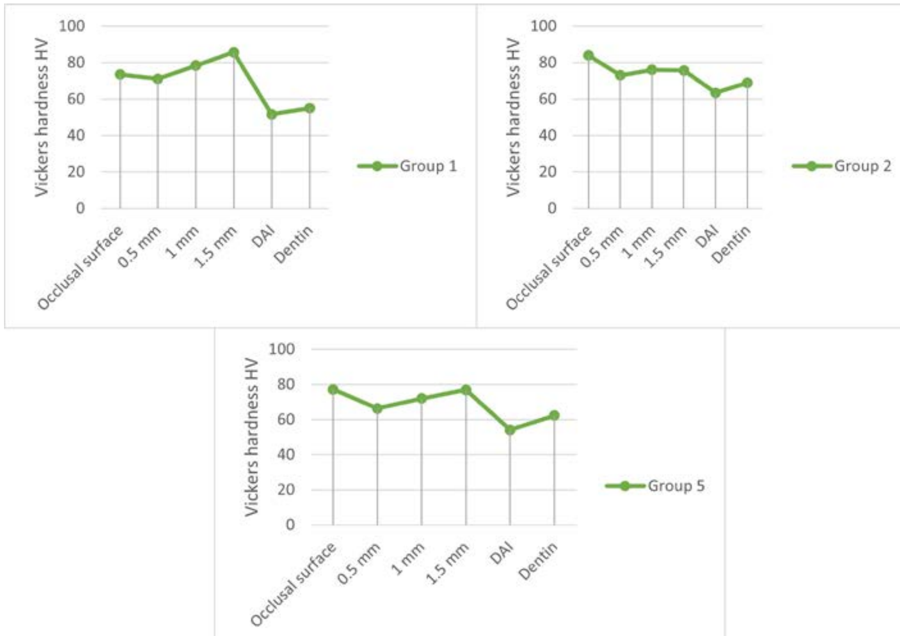


FIGURE 5. Microhardness values at the different measurement levels for three groups with different etching protocols

the photographs made after restoration showed that most fissures appeared in Group 1, where a total-etch adhesive protocol and bulk-fill resin composite were used.

DISCUSSION

Resin composite materials with high values of hardness are recommended for use in the enamel level of teeth. The microhardness of the bulk-fill resin composite material showed higher values compared to the universal composite material. Other studies using bulk-fill resin composites have found no significant differences in resistance compared to universal composites.[3] In case of low-viscosity bulk-fill composites, several studies found lower hardness values compared to high-viscosity bulk-fill composites and universal composites.[2,4] These results might be explained by the fact that low-viscosity materials have a lower filler particle content than high-viscosity resin composites.

Several studies have investigated different bulk-fill type resin composites, since their simple application method shortens chair time. Different bulk-fill materials presented different microhardness values. Comba *et al.* reported that low-viscosity bulk-fill resins showed adequate resistance even at a depth of 4 mm, while high-viscosity bulk-fill composites showed a gradually decreasing hardness after a depth of 2 mm.[5] However, in the present study, measurements were only taken at a depth of 1.5 mm in the composite level, and no significant difference in microhardness was found between different layers.

Another study, also investigating bulk-fill resins, compared the mechanical properties of the materials using different layering techniques. One technique involved inserting the material into the cavity in a single increment (4 mm depth), while the other one involved inserting the material in two 2-mm thick layers. The authors observed decreased microhardness values for in case of the single-layer insertion technique, but this was statistically significant for only one material.[6]

Rooz and Jordehi revealed similar results to ours. They also measured microhardness, using 2 and 4 mm thicknesses of bulk-fill type materials, but they took into consideration the different color shades as a possible influencing factor. They only measured the superficial and the bottom layer of the samples. In their results, the samples with 4 mm thickness showed lower hardness values than the samples with 2 mm thickness.[7]

In studies comparing conventional composites with bulk-fill type composites, it was found that although microhardness was decreased towards the deeper layers of the composite, better resistance and hardness can be achieved with bulk-fill composites compared to conventional composites, especially at cavity depths of 4 mm.[8–11] Lempel *et al.* studied the curing depth of different bulk-fill composites and conventional composites. Another study tested the Bulk Fill Posterior (3M ESPE Filtek) composite and found no significant differences between the top layer and the deeper layers regarding microhardness.[9]

Most adhesive systems can be used with either total-etch or self-etch techniques. Manufacturers have focused on developing the simplest possible adhesive technology to meet the needs of clinicians for a simple, fast, user-friendly, and technique-insensitive bonding system. Results from previous research have suggested a strong relationship between the bonding agent and the used technique, with some materials performing better with total-etch techniques and others being more sensitive to self-etch techniques.[12] Based on a study published in 2020, a significant difference in bond strength was found as a function of the adhesive and technique used, and the authors recommend the use of acid etching before the application of universal adhesives.[13] However, acid etching might influence the hardness of the dental tissue. Hence, different etching materials and procedures are tested,[14] and different solutions to recover the original microhardness of dentin are studied.[15] In the present study, no significant differences were found between the microhardness of the dentin at the adhesive interface level in the case of different adhesive protocols, but the lowest microhardness values were found when the total-etch technique was used. The acid etching of the dentin resulted in lower microhardness values at the adhesive interface and dentinal levels, but these values were not significantly lower than in other groups. We obtained the highest microhardness values using Optibond™ adhesive with the selective-etch protocol, where the surface of the dentin was not additionally etched. Transillumination was carried out according to the method of Rosatto *et al.*,[2] and similarly to their results, we detected new fissures after cavity preparation and restorations.

CONCLUSIONS

The used dentin adhesives and adhesive protocols did not significantly affect the hardness values of the dentin tissue at the adhesive interface level, although the lowest microhardness values were found in the case of the total-etch technique. The bulk-fill type composite used in the present study showed higher microhardness values than the universal composite. New microfissures appear in the dental hard tissues after both cavity preparation and polymerization of the resin composite restoration.

CONFLICT OF INTEREST

Nothing to declare.

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