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# EFFECT OF ADHESIVE STRATEGY ON RESIN CEMENT BONDING TO DENTIN

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# EFFECT OF ADHESIVE STRATEGY ON RESIN CEMENT BONDING TO DENTIN


## ABSTRACT

**Objective:** The cement bonding strategy and the polymerization mode can influence the prognosis of indirect restorations. The microtensile bond strength (μTBS) and dentin endogenous enzymatic activity of a dual-cure resin cement (PV5) used in combination with two dentin surface conditioners (accelerator-enhancer primer, TP or universal adhesive, UA) were evaluated.

**Materials and Methods:** PV5 was used to lute composite overlays after dentin treatment with TP or UA. The resin cement was self-cured, SC (1 h at 37 °C) or dual-cured, DC (20s light-cure followed by 15min self-cure at 37°C). The μTBS test, fractographic analysis, and the *in situ* zymography evaluations were performed after 24 h ($T_0$) or 1 yr ($T_{12}$) of artificial storage. Data were statistically analyzed ($\alpha$=0.05).

**Results:** TP/DC obtained the highest adhesive strengths (45±9 and 36.6±8), while UA/SC (17±8 and 11±4) the lowest, both at $T_0$ and $T_{12}$, respectively. DC resulted in superior bonding values than the SC, independent of the dentin surface treatment ($p<0.05$). The type of adhesive, curing mode and aging influenced the gelatinolytic activity ($p<0.05$).

**Conclusions:** The dual-cure resin cement used in combination with its accelerator-enhancer primer showed superior bonding performances with respect to universal adhesive. Dual-curing the resin cement was determinant to enhance bonding capability over time, independent of the adhesive strategy.

**Clinical relevance:** Clinicians must be aware to faithfully follow manufacturer's recommendation regarding the adhesive strategy suggested with the resin cement used.



**Keywords:** Curing mode, tooth primer, universal adhesive, 10-MDP, bond strength, *in situ* zymography.

## INTRODUCTION

The constant evolution of adhesive materials and the introduction of new material technologies has made it possible to perform highly conservative dental preparation approaches beyond the bounds of possibility of only few decades ago. Restorative materials possess both esthetic and mechanical properties, [1,2] and among the materials available to the clinician,[3] CAD/CAM technologies have been imposed themselves as the dentistry of the future.

The more conservative the restorative approach, the more outstanding the cement needs to be to ensure long-lasting restorations. Luting materials are the physical joint between the restoration and the tooth structure, interacting with these substrates through the establishment of chemical bonds and (micro)-mechanical retentions. [4] Resinous cements are the most commonly used materials for the cementation of both anterior and posterior indirect restorations. [4,5] The chemical composition of the cement significantly influences its clinical behavior, with 10-methacryloyloxydecyl-dihydrogen-phosphate (10-MDP)-based luting agents being universally accepted as the gold standard for enhancing strong and stable chemical bonds to both tooth and restorative materials.[6] This functional molecule has been achieved better results compared to its 10-MDP-free counterparts.[7] Specifically, this monomer establishes a strong chemical reaction through ionic binding to calcium salt at the adhesive-dentin interface and the formation of hydrolysis-resistant 10-MDP/Ca salts.[6,8] The stability is then ensured through the formation of rigid self-assembled nano-layering within the resin-dentin interdiffusion zone. Co-monomers are usually added to the material's composition to ulteriorly reinforce the physio-mechanical properties of the 10-MDP-based materials. It was noteworthy that, depending on the type of co-monomer, they could affect the rigidity of the nano-layering mechanism resulting in different bonding behaviors.[9]

The polymerization ability is directly related to the photo-initiators contained in the cement formulation,[10] as they can be chemically incompatible with functional acidic monomers [11] resulting in decreased adhesion, increased nano-infiltration phenomena and lower mechanical properties.[12,13] 10-MDP was proven to not inhibit the curing reaction, [14] ensure a durable interaction between restorations and tooth structures.[15]

In recent years, an alternative curing technology, defined as "touch cure", has captured the attention of clinicians and researchers. As the term itself indicates, once the resin cement encounters the accelerator-containing primer, the actual touch cure process takes place with the interaction between the chemical components of both primer and resin cement allowing for faster polymerization of the material. [16] In this moment, a reinforcement of the adhesive strength to dentin is determined. Then, light-curing is necessary to achieve a more optimal setting of the material.[17]

Despite the encouraging results observed with this new class of cement, very few data exist on their bonding performances and ultra-morphological characteristics when used in conjunction with their accelerator-enhancer tooth primer or with a universal adhesive. Since the touch-cure polymerization is a relatively recent technology, dentists may face uncertainty on the possibility of applying alternative tooth primers to those recommended by the manufacturers for time-saving and practical purposes. It is not uncommon for clinicians to confuse the versatility of universal adhesives to be applied in every clinical situation and in combinations with all type of cements.

Accordingly, the objective of this study, was to evaluate the influence of different 10-MDP-based material/co-monomer combinations on the adhesive effectiveness of a dual-cure resin cement used with its Tooth Primer or with a universal adhesive system on the microtensile bond strength and endogenous enzymatic activity within the hybrid layer. Specifically, the null hypotheses tested were that: 1) there is no difference in terms of bond strength (MPa) and endogenous enzymatic activity between the primer/adhesive dentin treatments; that 2) light-curing the resin cement does not influence its bonding ability and the biochemical characteristics of the hybrid layer; and that 3) no differences in bond strength (MPa) and endogenous enzymatic activity exist at baseline or after 1 year of artificial storage.

## MATERIALS AND METHODS

*Specimens' collection*

The protocol was approved by the Ethical Committee of the University of Bologna (protocol N°:71/2019/OSS/AUSLBO). Twenty human molars (sample size determined from a pilot study using G*Power 3.1.9.7 for Windows: effect size f - 1.027; α error probability – 0.05; power (1-β error probability) – 0.90) extracted for periodontal or orthodontic reasons and donated from anonymous individuals following their informed consent were collected for the study. Only teeth avoid of caries, cracks, fractures, and previous dental treatments were considered. Selected teeth were maintained in saline solution at 4 °C until use, no longer than 1 month. Saline solution was refreshed every week.

*Specimens' preparation*

The crown of each tooth was cut with a low-speed diamond wheel under abundant water irrigation (Microremet, Remet, Bologna, Italy) to expose middle/deep dentin. A standardized smear layer was created on dentin surfaces with #240-grit wet silicon carbide (SiC) paper, with rounded movements for 30s.

Resin composite build-ups were layered with two 2-mm thick increments of a nanohybrid resin composite (Venus Pearl, Heraeus Kulzer GmbH, Hanau, Germany, LOT: K010200) which were compacted into a silicone mold. The final height of the restoration was 4 mm, carefully controlled with a digital caliper with a precision of ±0.1. Each composite increment was polymerized for 40 s with a LED curing lamp according to manufacturer's recommendations (Demi™ Plus, Kerr Corp., Orange, CA, USA; light output: 1.200 mW/cm², wavelength: 450-480 nm). After the placement of the final layer, the composite overlay was removed from the mold, and additionally light-cured for 40s on each side and the bottom of the restoration, previously in contact with the mold.

A resin composite cement (Panavia V5, Kuraray Noritake Dental Inc., Okayama, Japan) was used for luting the composite indirect restorations to the dentin blocks. The resin cement was used with one of the following adhesive strategy (n=10) which were applied according to manufacturer's recommendations: 1) Tooth Primer (TP; Kuraray Noritake), applied to dentin for 10s and then air-blowed (not light-cured); and 2) iBond universal

adhesive (UA; Kulzer GmbH, Hanau, Germany) rubbed on dentin surfaces for 20s, air-dried and light-cured with a LED curing lamp for 10s. Materials' information and chemical compositions are presented in Table 1.

The bonding surfaces of composite resin blocks were wet-grinded with #240-grit SiC papers for 30s and cleaned in an ultrasonic bath for 2 min. Regarding the TP groups, the restorations were first etched with K-etchant, rinsed, dried and then Clearfil Ceramic Primer Plus (Kuraray Noritake) was brushed on the whole surface and air-dried. Instead, composite overlays of UA group were conditioned with iBond Ceramic Primer (Kulzer GmbH) and air-dried.

The specimens were further divided according to the curing modality of the resin cement after placement of the composite restoration (n=5): self-cure mode (SC; 1 h at 37 °C) or dual-cure mode (DC; 20s light-cure plus 15 min of SC at 37 °C). Light-curing was performed with a LED lamp (Demi™Plus, Kerr Dental). A constant seating pressure of 1 Kg was applied during the luting procedures, until the complete setting of the resin cement, both in the SC and DC modes.

*Microtensile bond strength test (μTBS)*

After 24 h, luted specimens were cut lengthwise into resin-dentin sticks (cross-sectional area of ~0.9 $mm^2$) and stored in artificial saliva (KCl 0.9639 g/L, KSCN 0.1892 g/L, $Na_2SO_4 \cdot 10H_2O$ 0.763g/L, $NH_4Cl$ 0.178 g/L, $CaCl_2 \cdot 2H_2O$ 0.2278g/L, $NaHCO_3$ 0.6308g/L, $ZnCl_2$ 2.726 mg/L, HEPES 1.186 g/L, pH 7.4) at 37 °C for 24 h ($T_0$) or 12 months ($T_{12}$). During the 1 yr storage, the artificial saliva was refreshed every 2 weeks. Before testing, each stick was measured with a digital caliper and stressed to tension according to the non-trimming method of the microtensile bond strength test (Bisco Inc., Schaumburg, IL, USA; crosshead speed: 1 mm/min). After testing, debonded sticks were observed under a stereomicroscope at 50x magnification (Stemi 2000-C; Carl Zeiss Jena GmbH) to determine the nature of the failures, categorized as: adhesive (A), cohesive (C) or mixed (C). The dentin sides of 2 randomly selected fractured sticks were mounted on metal stubs, gold sputter-coated and observed at different magnifications under a scanning electron microscope (SEM, Nova NanoSEM 450. Thermo Fisher Scientific, Waltham, MA, USA).

*In situ zymography analysis*

One-mm thick slabs were obtained from middle/deep dentin of additional 4 human molars. In order to reduce the anatomical variability, the slabs were divided into four quarters so all the experimental groups could be tested on the same substrate. After creating a standardized smear layer, each dentin slab was treated as to form the experimental groups previously presented for the μTBS test. Successively, the bonded assemblies were sectioned vertically into 1-mm thick specimens to expose the resin-dentin interfaces. Each resin-dentin stick was then glued with cyanoacrylate to glass slides, ground down to ~50 μm, polished and prepared for *in situ* zymography.[18] Each stick was then covered with a self-quenched fluorescein-conjugated gelatine mixture (E-12055; Molecular Probes, Eugene, OR, USA), protected with a coverslip and incubated overnight in a dark humidified chamber at 37°C. The specimens were evaluated with a confocal laser scanning microscope (excitation wavelength: 488/530 nm; LeicaSP8, TCS SP2 AOBS, Leica Microsystem GmbH, Wetzlar, Germany). A series of images were taken (one image per each 1 μm into the depth of the sample) to show the hydrolysis of the

quenched fluorescein-conjugated gelatine substrate, presented as green fluorescence. The quantification of the integrated density of fluorescence signals was made with ImageJ software (National Institute of Health, Bethesda, MD, USA).

*Statistical analysis*

Failures occurred before the μTBS test (namely "premature failures") were considered as "zero bond value" and included into the statistical analysis. After assessing the normal and equal distribution of the data (Kolmogorov-Smirnov and Levene's tests, respectively), a parametric statistical analysis was run (two-way analysis of variance, ANOVA) with the "surface pre-treatment" (TP *vs* UA) and the "curing mode" (SC and DC) as the independent variables and the interactions of these factors on the μTBS. Post-hoc comparisons were also made (Tukey test).

Quantification data recorded in the *in situ* zymography were normally (Shapiro-Wilk test) but not equally distributed (Brown-Forsythe test). Accordingly, the 3-way Analysis of Variance was used to compare the density of fluorescence signal between groups. Pairwise Multiple comparisons were run to evaluate differences between factors. In all statistical analysis, the level of statistical significance was pre-set at P=0.05. The analyses were performed using SigmaPlot 14.0 (Systat Software Inc., Chicago, Illinois, USA).

## RESULTS

*Microtensile bond strength test (μTBS)*

Table 2 illustrates the mean μTBS values, standard deviations, fracture modes and statistical significances of the tested groups. According to the results obtained, bond strengths were influenced by the dentin surface treatments (with TP performing significantly better than UA; $P<0.05$) and curing mode of the resin cement (SC mode achieved inferior bonding values than the DC mode; $P<0.05$). Specifically, the combination TP/DC resulted in the highest bond strength among the groups (45±9 MPa), while UA/SC the lowest (11±4 MPa), and this condition was highlighted both at baseline ($T_0$) and after 1 yr ($T_{12}$) of artificial aging ($P<0.05$). No statistical differences were found between TP/SC and UA/DC at the two storage time intervals investigated (P=0.05). Mixed failures (occurred at the dentin interface and within the cement simultaneously) were predominant in all the experimental groups, even though adhesive failures were mostly observed in the UA groups (both SC and DC).

*In situ zymography analysis*

Figure 1 shows representative confocal microscope images of the gelatinolytic activity within the hybrid layer of the tested groups at $T_0$ and $T_{12}$.

Statistical analysis identified significant differences between materials (with TP revealing lower fluorescence at the adhesive interface compared to U), curing mode (showing an increase in gelatinolytic activity in the SC groups respect to DC) and storage time (higher endogenous enzymatic activity within the hybrid layer was identified at $T_{12}$ compared to $T_0$). The interactions between these factors were also significant ($P<0.001$).

These differences were more evident at baseline. However, at $T_{12}$ no differences were found between TP and UA ($P>0.05$). After storage, higher dentin gelatinolytic activity was observed in the SC groups defining higher fluorescence level at the resin-dent interface as well as in the underlying dentinal tubules.

## DISCUSSION

All null hypotheses set out in the research question must be rejected since: 1) differences in bond strength and endogenous enzymatic activity within the hybrid layer were observed between TP and UA; 2) dual-curing the resin cement resulted in higher bonding performances and different biochemical characteristics than the only self-cure; 3) one yr of laboratory storage affected the tested parameters in all experimental groups.

Traditionally, resin cements present two main setting modes: a purely chemical reaction (namely self-cure mode) or light activation (light-cure mode), the choice of which must be dictated by clinical situations. Dual-cure mechanism aimed at exploiting the benefits of both self- and light-curing reactions, being particularly indicated in those areas where curing lamp has difficult access. However, dual-cure cements demonstrated an unbalanced mechanism between the chemical and light reactions, highlighting how the chemically-cured component is not sufficient to achieve complete polymerization reaction without additional light-curing.[19] Additionally, chemical incompatibilities with simplified adhesive systems rich in functional acidic monomers, including the latest universal adhesives introduced on the market, have been documented.[20,21] This leads to incomplete setting of the material resulting in the hydrolysis phenomena, swelling, and reduced mechanical strength.[15] Several strategies have been approached to ameliorate the self-curing ability and bonding performances of resin cements, with the touch cure technology as one of the most recent investigated. [16]

A relevant information obtained in our study, was that the resin cement tested (PV5) attained the highest bond strength in association with its tooth primer (TP) depending on the curing mode of the resin cement (Table 2). Indeed, the highest adhesion result was achieved between PV5 and TP when the cement was used in the DC mode. However, it was puzzling to observe no differences between TP/SC and UA/DC. Speculatively, this would indicate that when the resin cement was used in the self-cure mode with its tooth primer, it demonstrates similar adhesive behavior as when the same cement was used in the dual-cure mode with a different (universal) adhesive system, indicating that to optimize the touch-cure technology to the maximum, the dual-curing mode of the cement should be used.[14,22] In the same way, the biochemical results indicated that the resin cement polymerization modality rather than the bonding strategy was able to roughly activate the dentinal endogenous enzymes within the hybrid layer (Figure 1). Similar results have recently been reported, where maximization of adhesive capacity and mechanical properties was achieved when TP was used with dual-cured Panavia V5.[14] The spectrophotometric aspect of this reaction, which has not been the subject of this study, will have to be analyzed in future studies to better identify the problem underlying the reaction between the cement used in self-cure mode and the primer.

Both the polymerization enhancer primer and the universal adhesive used in this study contain 10-MDP in their formulation (Table 1). With the intent to further improve the chemical bonding at the dentin interface, additional monomers were combined to 10-MDP based adhesives.[9,,23] However, recent studies reported that, depending on the co-monomer group, the rigidity of the nano-layering mechanism could be compromised, limiting the adhesion ability to dentin.[9] Both dentin surface conditioners tested in this study contain 10-MDP, but they

possess different formulations with TP containing HEMA and UA 4-META as co-monomers (Table 1). Previous studies speculated that the interaction between 10-MDP and HEMA (i.e. such of TP) to form 10-MDP-HEMA complex may weaken the protective function of 10-MDP towards collagen and, therefore, result in a deficit of the adhesive strength.[9,24] These assumptions were corroborated by our results, as bond strength in the TP groups significantly drop after 1 year of laboratory storage (Table 2). Likewise, higher endogenous enzymatic activity in the form of increased fluorescence settled within the hybrid layer was observed in the TP groups after artificial aging and no different gelatinolytic activity could be detected between the bonding materials (Figure 1).

Taking the chemical aspect into consideration, the combination of 10-MDP with carboxylic acid (i.e. 4-META) resulted in low bonding performances due to increased hydrolysis phenomena.[15,25] The unbalanced proportion between hydrophilic and hydrophobic components seemed more pronounced for UA, being more susceptible to greater water sorption from the underlying hydrated dentin.[26,27] The hydrophilicity of 4-META monomer from one side and the slow self-polymerization reaction of the resin cement has been considered the factors jeopardizing the adhesion to the tooth substrate.[15] These results are also corroborated by the higher adhesive failure percentage observed in the UA groups (both in the SC and DC groups) (Table 2). Another aspect that should be taken into consideration when analyzing the adhesive behavior of UA, particularly when used with resin cement in the SC mode, is that the adhesive was light-cured prior to the application of the cement. Light-curing the universal adhesive before cement application resulted in limited chemical reactions between the cement and the dental substrate with consequent premature debonding of the restoration.[14,15] It remains to be verified whether leaving the adhesive system uncured would influence the cement/adhesive/dentin interaction.

Even though the idea of evaluating the effects of different surface conditioners, specifically in presence or not of the proper polymerization activator, on the adhesive parameters is not new to the literature,[14,16,28] to the best of our knowledge, this is the first time that these effects were evaluated after long-term artificial aging. Likewise, there are no evaluations of enzymatic activity in the hybrid layer under the same experimental conditions.

Even though the manufacturer's instructions of the resin cement tested in this study indicate to use it in combination with the polymerization enhancer primer, our results showed that, when using a universal adhesive instead of the primer, the mechanism of adhesion to dentin seems to be more related to the polymerization mode of the cement. While it remains an obligation for the clinician to meticulously respect the indications of use suggested by each manufacturer for their products, it is not so unusual for products of different brands to be used interchangeably. Consequently, dentists must be aware that this could have an impact on the long-term durability of the restorations.

## CONCLUSIONS

Within the limits of this study, the following conclusions can be drawn:

1) Differences exists between the adhesive strategies tested. Accordingly, when the proper primer/adhesive dentin treatments as indicated by manufacturer's recommendations are not faithfully respected, the bond strength and gelatinolytic activity within the hybrid layer of the resin cement can be compromised;

2) Independent of the adhesive strategy, dual-curing the resin cement resulted in enhanced bonding capabilities;

3) Artificial aging demonstrated that the curing mode, more than the materials tested, was determinant to maintain the stability of the bond.

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**LEGENDS**

**Table 1**. Tested materials with their respective manufacturers, batch numbers, and chemical compositions.

**Table 2**. Mean microtensile bond strength values (MPa) ± standard deviations and failure mode distributions (%) among the experimental groups. SC: self-cure mode; DC: dual-cure mode; TP: Tooth Primer; UA: iBond universal adhesive. One resin cement was used for luting procedure (Panavia V5). A: Adhesive failures occurred at the dentin interface; M: mixed adhesive and cohesive failure.

**Figure 1**. *In situ* zymography results. **A**) Resin dentin interfaces incubated with quenched with fluorescent gelatin. Mages acquired in the green channel (a,c,e,g,i,k,m,o). Fluorescence is lower in the DC groups and at $T_0$, as compared to the SC and aged groups ($T_{12}$). Composite images obtained by merging the differential interference contrast image and those obtained in the green channel (b,d,f,h,j,l,n,p). **B**) Bar graph showing the data quantification of the endogenous enzymatic activity within the adhesive interfaces of the tested groups. TP: Tooth Primer; U: iBond universal adhesive; SC: self-cure mode; DC: Dual-cure mode; $T_0$: evaluation after 24 h; $T_{12}$: evaluation after 1 yr of artificial aging; D: dentin; HL: Hybrid layer; R: resin cement.

**Table 1**

| Materials | | Manufacturer (LOT) | Composition |
|---|---|---|---|
| Dual-cure resin cement | Panavia V5 | Kuraray Noritake Dental (LOT: CJ0093) | Paste A: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass filler, silanated, fluoroalminosilicate glass filler, colloidal silica V5 Paste<br>Paste B: Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, silanated alminium oxide filler, accelerators, dl-camphorquinone, pigments. |
| Self-etch Primer | Panavia V5 Tooth Primer | Kuraray Noritake Dental (LOT: 8A0029) | 10-MDP, HEMA, hydrophobic aliphatic dimethacrylate, water, accelerators (non-amine reducing agent) |
| Universal adhesive | iBond universal | Kulzer GmbH (LOT: K010028) | 10-MDP, 4-META, methacrylates, acetone, water |

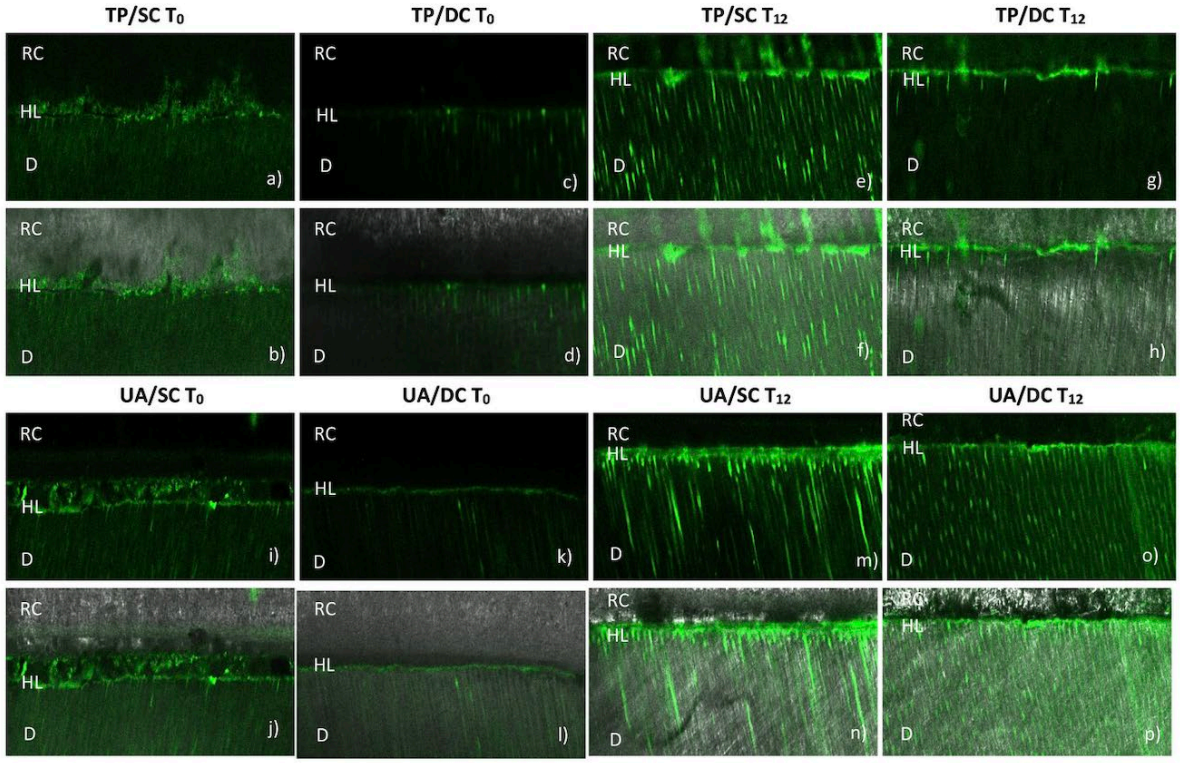
Bis-GMA: bisphenol A di (2-hydroxy propoxy) dimethacrylate; TEGDMA; triethylene glycol methacrylate; 10-MDP; 10-methacryloyloxydecyl dihydrogen phosphate; HEMA; 2-hydroxyethyl methacrylate; 4-META: 4-Methacryloxyethyl trimellitate anhydride.

**Table 2.**

| **Tested Materials** | **TP** | | **UA** | |
|---|---|---|---|---|
| **Polymerization Mode** | **SC** | **DC** | **SC** | **DC** |
| **$T_0$** | $34.5\pm8^{B1}$ M: 98%; A: 2% | $45\pm9^{A1}$ M: 100% | $17\pm8^{C1}$ M: 78%; A:22% | $37.6\pm15^{B1}$ M: 92%; A: 8% |
| **$T_{12}$** | $25\pm5^{B2}$ M: 97%; A:3% | $36.6\pm8^{A2}$ M: 100% | $11\pm4^{C2}$ M: 78%; A: 22% | $25.6\pm9^{B2}$ M: 95%; A% 5% |

Different superscript letters and numbers (in rows and columns, respectively) indicate statistically significant differences among groups ($p<0.05$).

A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TP/DC T0
TP/SC T12
TP/DC T12
UA/SC T0
UA/DC T0
UA/SC T12
UA/DC T12
RC
HL
D
a)
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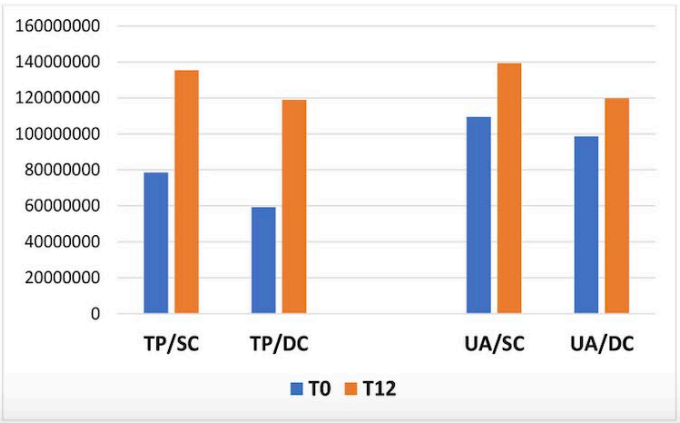
j)
k)
l)
m)
n)
o)
p)

B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TP/SC
TP/DC
UA/SC
UA/DC
T0
T12