Original Article

An *in vitro* study to compare the influence of different all-ceramic systems on the polymerization of dual-cure resin cement

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Abstract

Aim: The aim of the study is to compare the effect of composition of three different all-ceramic systems on the polymerization of dual-cure resin cement, using different curing cycles and evaluated immediately within 15 min and after 24 h.

Materials and Methods: Resin cement disc samples were fabricated by polymerization through three different all-ceramic disc, namely: lithium disilicate discs – IPS e.max (Group B), leucitereinforced discs – IPS Empress (Group C), zirconia discs – Cercon (Group D), and without an intervening ceramic disc, as control (Group A). A total of 80 resin cement disc samples were fabricated for fur groups ($n=20$). Each group further consisted of two subgroups ($n = 10$), t10 and t20 according to two different exposure times of 10 and 20 s, respectively. Each of the 80 resin disc samples was evaluated for their degree of polymerization achieved, by measuring the microhardness(Vickers hardness number) of the samples immediately within 15 min and after 24 h, giving us a total of 160 readings. Oneway analysis of variance test, ttest, and paired ttest were used for multiple group comparisons followed by Tukey's *post hoc* for groupwise comparison.

Results: Direct activation of the resin cement samples of control (Group A) showed statistically significant higher mean microhardness values followed by Groups C then B and D, both immediately and after 24 h. The mean microhardness for immediate post-activation was always inferior to the 24 h post-activation test. For both 10 and 20 s curing cycle, there was a significant increase in the microhardness of the resin cement discs cured for 20 s through the different ceramics.

Conclusion: Ceramic composition affected the polymerization of dual cured resin cement. Doubling the light irradiation time or curing cycle significantly increased mean microhardness value. Greater degree of conversion leading to an increase in hardness was observed when the resin cement discs were evaluated after 24 h.

Keywords: All ceramic, curing cycle, dual-cure resin cement, microhardness (Vickers hardness number), polymerization

Access this article online	
Quick Response Code:	**Website:** www.j-ips.org
	DOI: 10.4103/jips.jips_262_18

How to cite this article: Majumder A, Giri TK, Mukherjee S. An *in vitro* study to compare the influence of different all-ceramic systems on the polymerization of dual-cure resin cement. J Indian Prosthodont Soc 2019;19:58-65.

INTRODUCTION

Over the last few decades, improved technology and invent of newer materials have changed the scope of prosthetic and restorative dentistry.[1] Among such materials are ceramics[2] and resin-based luting agents. Resin-based composite bonding and luting technology are considered as an inherent part of the state-of-the-art all-ceramic restorations.[3] Their ability to adhere to both ceramic and tooth substrate, insolubility in oral fluid, high strength, shade-matching potential, and easy handling characteristics increase fracture toughness of all-ceramic restorations. All these properties have made resin cement adhesive of choice for all-ceramic restoration.[4] Within the literature, number of studies explained, apparent strengthening of all-ceramic restorations cemented with resin cement.[5-7]

Resin-based composite polymerization is a form of addition polymerization, in which there is no by-product production and the macromolecules (polymers) are formed from smaller units (monomers) by conversion of carbon–carbon double bond into a saturated linkage of free radicals (Anusavice, 1996).[4] Success of resin cement as luting agent depends on efficient polymerization determined by amount of free radicals generated and degree of conversion (DC).[8] Factors that affect amount of free radicals generated and DC, includes properties of all-ceramic, resin cement, light used and postactivation testing time.

Crystal content, size and structure, translucency, and shade and thickness of ceramic affect amount of light passing through them. More light is attenuated by crystalline ceramics which are opaque in nature and thicker ceramic restoration. The type of resin cement (light-cured or chemical-cure or dual-cure), type and concentration of initiator, activator present, and shade of resin cement will affect polymerization.[11,16]

For adequate polymerization of both light-cure and dual-cure resin, light transmission and number of photons generated are important. Source of light (ultraviolet/ tungsten-halogen/Argon-laser/light-emitting diode (LED)/plasma-arc), distance of light guide tip from restoration, wavelength, irradiance, radiant exposure, and exposure time are important consideration.[12,13]

Strydom found that light intensity and time are the most important factors. He indicated that exposure times used by dentists for light-polymerizing cement are too short. Longer polymerization times are necessary to compensate for decrease in light intensity incident upon resin adhesive for adequate polymerization.

Extent of polymerization of resin material is assessed by evaluating DC of resin matrix. It is the percentage of double bonds that have been converted to single bonds to form cross-linked polymer resin. Various methods to measure the DC are as follows: direct methods (Fourier-transform infrared spectroscopy [FTIR]/laser Ramen spectroscopy) and indirect methods (microhardness or depth of cure). Microhardness tests were used to evaluate DC. These methods correlate in their results with that of direct methods such as FTIR spectroscopy.[24,25]

This *in vitro* study was performed to compare influence of composition of different all-ceramic systems, curing time on polymerization of dual-cure resin, through the measure of Vickers microhardness of set resin sample, immediately within 15 min and after 24 h of postactivation. Only LED with light intensity of 2000W/cm square was considered in the study to evaluate the effect of exposure time on the radiant exposure on the degree of polymerization keeping other factors of light constant.

Null hypothesis

1. Composition of all-ceramic systems does not affect transmission of light through them and hence does not affects polymerization of dual-cure resin
2. Duration of light exposure time does not have any effect on the polymerization and thereby no effect on hardness achieved
3. Postactivation testing time (immediately and after 24 h) have no effect on the polymerization and the hardness of set resin.

MATERIALS AND METHODS

This *in vitro* study was done using dual-cure resin cement (Variolink N, Ivoclar Vivadent) of transparent shade and three all-ceramic systems, namely, leucite reinforced (IPS Empress), lithium disilicate (IPS e.max), and zirconia (Cercon), A3 dentin shade. Only A3 shade was considered because, apart from ceramic crystalline composition, shade and thickness of ceramics also affect the light transmission through it, so in order to evaluate the effect of composition, other factors of ceramics such as shade were kept constant.

Fabrication of all-ceramic disc
Leucite reinforced (IPS Empress), lithium disilicate (IPS e.max), and zirconia (Cercon) discs of 8 mm in diameter and thickness of 1.2 mm as measured on digital vernier caliper. IPS Empress esthetic ingot for staining technique (shade ETC-1) was pressed and stain fired with IPS Empress Universal Stains (A3) and glazed to obtain leucite-reinforced disc.

An ingot of IPS e.max, shade MO-1 was pressed and core thickness of 0.7 mm thickness was obtained. Porcelain e.max Ceram shade dentin A3 was applied and fired to obtain lithium disilicate disc. Discs were grounded to obtain total thickness of 1.2 mm and subjected to finishing and glaze firing [Figure 1].

To fabricate zirconia disc, wax pattern of 0.4 mm thickness and 8 mm diameter was obtained. Cercon brain unit was used for scanning wax pattern. Milling of base blank of presintered zirconia followed by sintering to fully dense structure was done. IPS e.max Ceram, shade dentin A3 was layered and fired, disc was then finished and glazed to obtain final disc thickness of 1.2 mm.

Fabrication of acrylic-resin mold and elastomeric-mold
Fabrication of acrylic resin mold and elastomeric molds were done. They are used for making resin samples A metal cylinder (5 mm in diameter and 1 mm thick) was secured onto a glass slab, a separating medium such as Vaseline is applied to metal cylinder for easy separation of resin mold [Figure 2]. An impression of this metal cylinder was made in PMMA resin in dough stage, thereby creating a pink colored acrylic resin mold with centered aperture of same dimensions as the metal cylinder. Similarly, elastomeric mold was made.

Preparation of dual-cure resin samples
A total of 80 dual-cure resin (Variolink N) discs, measuring 5 mm in diameter and 1 mm in thickness, for 4 groups (each group $n = 20$) is fabricated for study. Variolink N resin luting agent, transparent shade was used. Base and catalyst paste of resin cement were mixed in 1:1 ratio according to manufacturer's instructions and inserted into cylindrical elastomeric mold.

A transparent Mylar's strip was then placed over the filled orifice. This Mylar strip acts as separating layer between ceramic disc and resin. It also produced a smooth evenly finished surface layer, needed for producing accurate indentation by microhardness tester machine. It also acts as oxygen inhibiting layer.

Resin cement was activated by LED from Ivoclar Vivadent, Blue phase N [Figure 3], with tip diameter of 9 mm and irradiance of 2000 mW/cm². Light intensity of curing unit was measured with hand-held radiointensity meter from Ivoclar Vivadent.

Four experimental groups ($n = 20$) were formed, which consisted of 80 resin-cement disc specimens [Figure 4]:
- Group A: Control group (without an intervening ceramic disc)

Figure 1: All-ceramic discs

Figure 2: Elastomeric and acrylic molds

Figure 3: Radiointensity meter from Ivoclar Vivadent – to measure intensity or irradiance of light in mW/cm²

- Group B: Resin cement discs cured through lithium disilicate disc
- Group C: Resin cement discs cured through the leucite-reinforced disc
- Group D: Resin cement discs cured through zirconia.

Control group specimens were obtained by direct activation, that is, without interposing any ceramic disc in-between the resin cement and light source. Wand tip of light curing unit was held in contact with Mylar's strip.

To obtain experimental group (Groups B, C, and D) specimens, one of the three ceramic discs were placed on the strip. During photoactivation, wand tip of light curing unit was held in contact with ceramic disc.

Each group further consisted of two subgroups ($n = 10$), t10 and t20, according to two different exposure times of 10 and 20 s, respectively.

Each of the 80 resin disc specimens was evaluated for microhardness (VHN) immediately (within 15 min) on day-1 and (after 24 h) day-2, giving us a total of 160 readings.

In 24-h postcure time, specimens were stored in light-proof containers at 37°C for 24 h and then evaluated for microhardness.

Parameters to be studied

DC of dual-cure resin cement is assessed indirectly by evaluating surface microhardness using Vickers microhardness tester and then comparing:

- The effect of composition of all-ceramic systems on transmission of light through them, which affects polymerization of dual-cure resin
- The effect of duration of light exposure time on hardness achieved when a fixed radiant energy reaches the specimen
- The postactivation testing time (immediately and after 24 h) on hardness of set resin.

Surface hardness measurement of dual-cure resin samples

DC of resin cement specimens was expressed in terms of Vickers hardness number (VHN), using universal indenter with Vickers hardness indenter having square-based pyramid whose opposite sides met the apex at 136° angle.

To perform Vickers test, resin cement disc was placed on an anvil that had screw threaded base. The anvil was turned and raised by screw threads until it was close to the point of the indenter [Figure 5]. Surface of resin cement disc facing light source was subjected to static load of 50 g for 15 s by means of indenter. Load was released and the anvil with specimen was lowered. Applying of load and removing, it was automatically controlled. A calibrated microscope (×40 magnification) was used to measure the square indentation to a tolerance of ± 1/1000 of a millimeter and their average calculated [Figure 6].

The area of sloping surface of indentation was calculated. Vickers hardness is obtained by dividing load by mm² area of indentation. Vickers hardness was calculated using formula, $H = P/A$, where H is VHN, P is load, and A is area.

Statistical analysis

Statistical analysis was done with Epi Info (TM) 3.5.3. EPI INFO is trademark of centers for Disease. Descriptive

Figure 4: Dual-cure resin samples

Figure 5: Samples mounted on Vickers indenter

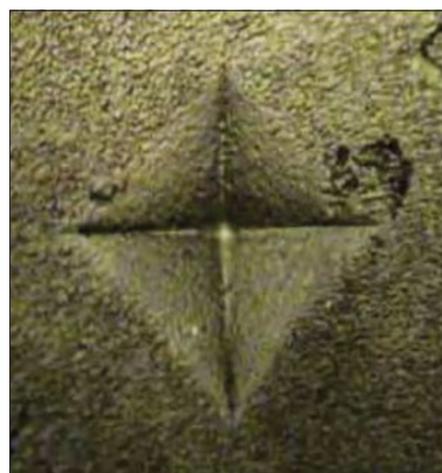**Figure 6:** Microscopic image seen at ×40 magnification of square-based pyramid-shaped indentation formed on resin sample by Vickers hardness indenter

statistical analysis to calculate means with standard deviation (SD). For comparing the effect of all-ceramic on microhardness values of resin cement analysis of variance followed by *post hoc* Tukey's test was performed with the help of critical difference at 5% and 1% level of significance to compare the mean values.

For comparing effects of curing time on microhardness achieved *t*-test was used to compare the means. Here $P < 0.05$ was taken to be statistically significant. For comparing the effect of postactivation testing time on

microhardness, paired *t*-test was used to compare the means.

RESULTS

Results of microhardness testing are shown in Tables 1, 2 and Figures 7, 8 which indicate mean and SD of VHN for each group after 10 s and 20 s of curing, respectively.

Direct activation (Group A) of resin cement showed statistically significant higher mean microhardness values as compared to experimental groups (B, C, and D), both immediately and after 24 h. The microhardness values were in the descending order of control group (Group A) followed by Empress (Group C), then e.max (Group B) and Cercon (Group D).

There was significant increase in polymerization [Tables 3 and 4] of all groups including control group for 20 s curing than 10 s curing when tested immediately and after 24 h.

Microhardness for immediate postactivation test was inferior to 24-h postactivation test in both direct activation and through different ceramics [Tables 5 and 6].

DISCUSSION

In the present study, the choice of ceramic systems and their fabrication technique was influenced by recent trends. The glass-ceramic discs, leucite-reinforced and lithium disilicate, were heat pressed and zirconia-based ceramics was fabricated using computer-aided design/computer-aided manufacturing technique.

The DC of resin matrix has a direct influence on mechanical properties of resinous materials.[18] It determines the surface hardness and wear resistance of resin materials.[10,20]

Various direct and indirect methods are applied to evaluate the DC of resin cement. Although FTIR[18,20] or laser

Raman spectroscopy[21] is the most sensitive test among direct methods, they are, however, very expensive and time-consuming.[22] The various indirect methods are depth of cure[14] scratch test and microhardness testing.[15,23] These indirect methods are not only economic but also easy to perform and exhibits differences between different exposure

Table 1: Mean (mean±s.d.), ANOVA and CD values of microhardness values (VHN) of resin sample for all groups using 10 seconds of light exposure on DAY-1 and DAY-2

Study Group	Immediately on Day 1	After 24 h on Day 2
	Mean±s.d	Mean±s.d
Control	28.17±0.68	53.22±4.24
Lidi Silicate	23.22±0.89	28.03±1.72
Leucite	24.47±0.63	31.49±0.81
Zirconia	16.94±1.44	26.71±1.31
$F_{3,39}$	233.27	263.42
P	<0.0001*	<0.0001*
At 5% level of significance (CD_5)	1.60	4.01
At 1% level of significance (CD_1)	2.15	5.37

Table 2: Mean (mean±s.d.), ANOVA and CD values of microhardness values (VHN) of resin samples for all groups for 20 seconds oflight exposure on DAY- 1 and DAY-2

Study Group	Immediately on Day 1	After 24 h on Day 2
	Mean±s.d	Mean±s.d
Control.	41.01±3.44	57.90±9.84
Lidi Silicate	30.18±0.85	39.90±0.92
Leucite	33.20±0.39	46.98±1.78
Zirconia	20.48±0.67	35.68±0.64
$F_{3,39}$	217.93	37.26
P	<0.0001*	<0.0001*
At 5% level of significance (CD_5)	3.03	8.34
At 1% level of significance (CD_1)	4.04	11.19

Table 3 : *t*- testdone to compare the effect of different exposure times or curing cycle on microhardness (VHN) of dual-cured resin cement on Day 1 after curing

Study Group	Curing Time (mean±s.d)		t_{18}	P
	10 seconds	20 seconds		
Control GROUP - A	28.17±0.68	41.01±3.44	11.57	<0.0001*
IPS e.max GROUP - B	23.22±0.89	30.18±0.85	17.88	<0.0001*
IPS EMPRESS GROUP - C	24.47±0.63	33.20±0.39	37.25	<0.0001*
CERCON GROUP - D	16.94±1.44	20.48±0.67	7.04	<0.0001*
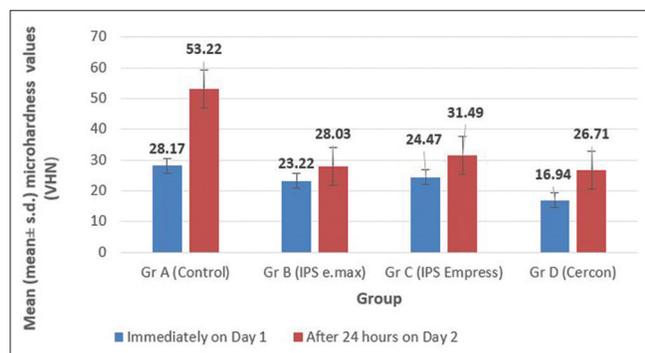
Figure 7: Mean (mean ± standard deviation) of resin sample for all groups using 10 s of light exposure on day-1 and day-2
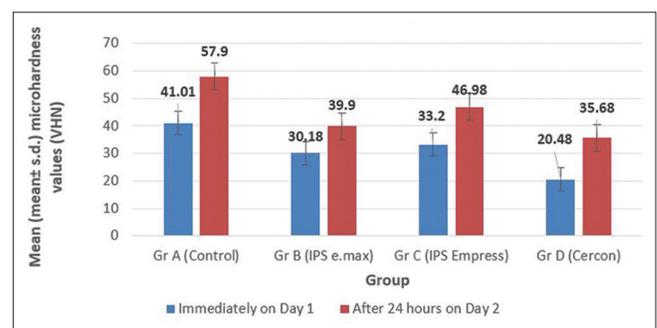
Figure 8: Mean (mean ± standard deviation) of resin sample for all groups using 20 s of light exposure on day-1 and day-2

Table 4: *t*-test done to compare the effect of different exposure times or curing cycle on micro hardness (VHN) of dual-cured resin cement on Day 2

Study Group	Curing Time (mean±s.d)		t_{18}	*P*
	10 sec	20 sec		
Control GROUP - A	53.22±4.24	57.90±9.84	1.38	0.18
IPS e.max GROUP - B	28.03±1.72	39.90±0.92	19.24	<0.0001*
IPS Empress group -C	31.49±0.81	46.98±1.78	25.04	<0.0001*
CERCON GROUP -D	26.71±1.31	35.68±0.64	19.45	<0.0001*

Table 5: Paired *t*- test done to compare effect of post activation testing time on micro hardness of dual-cured resin cement for 10 sec curing time

Group	For 10 sec of Curing Time (mean±s.d)		t_{18}	*P*
	Immediately on Day 1	24 h after curing		
Control GROUP - A	28.17±0.68	53.22±4.24	18.44	<0.0001*
IPS e.max GROUP - B	23.22±0.89	28.03±1.72	7.85	<0.0001*
IPS empress group -C	24.47±0.63	31.49±0.81	21.63	<0.0001*
CERCON GRROUP - D	16.94±1.44	26.71±1.31	15.87	<0.0001*

Table 6: Paired *t*-test to compare the effect of post activation testing time on micro hardness (VHN) of dual-cured resin cement for 20 sec curing time

Study Group	For 20 sec of Curing Time (mean±s.d)		t_{18}	*P*
	Immediately on Day 1	24 h after curing		
Control GROUP - A	41.01±3.44	57.90±9.84	5.12	<0.0001*
IPS e.max GROUP - B	30.18±0.85	39.90±0.92	24.53	<0.0001*
IPS empress group -C	33.20±0.39	46.98±1.78	23.91	<0.0001*
CERCON GROUP - D	20.48±0.67	35.68±0.64	51.87	<0.0001*

situations.[9] In a study conducted by Rueggeberg *et al.*,[24,25] it was observed that surface hardness measurements showed results similar to FTIR spectroscopy. Therefore, in the present study, indentation testing (VHN) was used to check the microhardness of the dual-cured resin cement.

There is wide variation in composition and crystal content of ceramics from different manufacturers, which may impact the quantity of photons that pass through them for activation of resin cement[14] Hence, in this study, frequently used ceramic systems of different compositions and crystallinity were tested, and comparison was made between direct and indirect activation of resin cement.

Albeit the resin cement is directly cured, it shows 55%–75% of DC. However, when cured indirectly through ceramic prosthesis, composition, opacity, and thickness and shade of ceramic will attenuate the intensity of light[26,27] and reduce the number of photons reaching resin cement. The corollary is a low DC% leading to inferior physicomechanical properties, and the prognosis of indirect restorations could suffer.

The result of the study showed that VHN for leucite reinforced is greater than lithium disilicate followed by zirconia. As the crystalline content increases, translucency decreases, and the polycrystalline ceramics such as zirconia appear opaque and are expected to attenuate more light.

To provide satisfactory polymerization where curing light is attenuated by ceramic restoration, manufacturers may increase concentration of tertiary amine. This, however, will have undesirable effect of making materials less color stable. Further work is necessary to develop appropriate balance between rate and efficiency of cure and color stability.

Strydom[17] has indicated that irradiation times used by dentists for light-polymerizing cement are too short. Longer polymerization times are necessary to offset decreases in light intensity incident upon resin adhesive due to both overlying ceramic material and light source factors to achieve an adequate DC. Therefore, in this study, it has been tried to increase the efficiency of cure by increasing the light exposure time from manufacturer's recommended 10 s to 20 s to elevate the quantity of photons that reach the cement and to improve the DC.

Results of the current study showed lower hardness values for immediate 10 s of curing [Table 1] through ceramic discs as compared to 20 s curing [Table 2].

This deficient polymerization of resin cement after 10 s curing time, negatively affect physical and mechanical properties. It has been already proven that even well-polymerized resin cement can release residual monomers, so a poorly polymerized resin cement would elute more substances from them which can lead to irritation of pulp and soft tissues, stimulate proliferation of bacteria, and cause allergic reactions. Thus, curing protocol has critical effect on the hardness and a major factor influencing the clinical performance of resin-based cement.[28] Therefore, it was concluded that the manufacturer's recommended 10 s curing protocol may not be enough to achieve satisfactory hardness and DC of resin cement.

In clinical situation, it is also important to know the immediate hardness obtained after initial cure of resin cement. This is critical for initial management of restoration, such as finishing and occlusal adjustments. Therefore, this study has evaluated initial and final hardness by measuring VHN immediately and after 24 h.

In the present study, immediate testing time [Table 5] showed lower hardness values than 24 h testing time [Table 6] for both 10 and 20 s curing cycles. These results are in

accordance with a study conducted by Valentino *et al*. in 2010.[29] Hence, one can be suspicious of prosthesis being unstable immediately after cementation and could be dislocated during chewing. Hence, during cementation procedure, it is recommended to follow curing protocol that includes additional time to allow for adequate polymerization. Moreover, patients should be advised to avoid biting on hard foodstuff for at least next 24 h.[29]

Hardness obtained by resin cement when used under ceramic discs was less than that of the controls which were directly exposed to light for both 10 and 20 s of curing. These findings confirm that indirect activation through ceramic discs decreases amount of light reaching luting material, which needs to be compensated for, by increasing curing cycle timings.

For 24-h postactivation testing of both 10 and 20 s curing cycle, there was significant increase in microhardness of resin cement discs cured for 20 s through different ceramics except for direct light-activation group. The control group did not show statistically significant difference in 24 h testing for both 10 and 20 s curing cycle, which justifies previous studies done by Meng *et al*. that when resin cement are polymerized in a dual mode, the faster reaction promoted by light activation hinders chemical component of polymerization.[30]

Meng *et al*.[30] showed that even low-intensity irradiation of dual-cured resin cement still had large number of free radicals, mostly from trapped chemical catalysts in hardening resin matrix, which did not increase the overall DC% of materials. Considering the findings of Meng and above discussion, it is fair to speculate that chemical component of resin cement contributed sparsely to overall polymerization after dual activation through different ceramic discs. Hence, significant chemically induced continuation of polymerization after light initiation is difficult to achieve. Therefore, duration of inhibition and level of initial conversion caused by light exposure are highly influential factors upon final cure of dual-cured resin.[25]

The behavior of cement used in this study also seems to depend more on light activation. Therefore, in an effort to try to maximize the DC as much as possible, increased light-curing cycle times may be recommended.

The thickness of ceramics used in the current study is 1.2 mm, designed to be as close as possible to that used clinically. It has been reported that when thickness of restorative materials was increased, the DC and final hardness of most dual-cured resin cement were reduced.[19,26]

Limitations of the study

The *in vitro* nature of the study does not replicate intraoral conditions. Saliva may cause water sorption of resin cement. Higher intraoral temperatures may have an influence on kinetics of chemical reaction. It is also subjected to cyclic loading due to masticatory function during first 24 h, which also affects microhardness of resin. Hence, further *in vivo* investigations are needed.

In this *in vitro* study, only single brand of dual-cure resin cement was used. It should also be noted that different brands of dual-cured resin cement have different ratios of light, chemical catalysts; this may result in differences of polymerization efficiency of different commercial brands resin cement.

CONCLUSION

Within the limitations of study, it may be concluded as follows:
- Direct activation of dual-cure resin achieves higher hardness than when cured through ceramic systems, irrespective of curing cycle used
- Ceramic composition affects polymerization of dual-cured resin cement due to attenuation of radiant exposure reaching cement. In this study, microhardness of resin cement discs cured through leucite-reinforced ceramic disc was significantly greater than lithium disilicate disc followed by zirconia disc
- Doubling the light exposure time significantly increases microhardness of the resin. Hence, dual-cured resin cement should always be photo-activated for longer periods than recommended
- This *in vitro* study also showed that there is increase in hardness of the resin cement when measured after 24 h due to residual chemical polymerization.

Conflicts of interest
There are no conflicts of interest.

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