Original Article

Evaluation of microhardness and water sorption/solubility of dual-cure resin cement through monolithic zirconia in different shades

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Abstract

Aim: The objective is to evaluate the effect of shades of monolithic zirconia on the microhardness and sorption/solubility of the underlying two dual-cured resin types of cement.

Materials and Methods: Eighty samples of two dual-cured resin cement discs were polymerized under 60 monolithic zirconia discs in three shades and directly activated resin discs of cement were used as the control group ($n = 10$). After 24 h storage at 37°C in an incubator, Vickers microhardness and the sorption and solubility were measured.

Statistical Analysis Used: Two-way ANOVA , one-way ANOVA, Independent t-test, Tukey's honestly significant difference, and Tamhane's T2 tests.

Results: The mean microhardness of the Variolink N resin cements were significantly higher than Panavia SA ones ($P < 0.001$). Furthermore, Variolink N cements exhibited lower sorption/solubility than Panavia SA resin cements (both $P < 0.05$). The ceramic shade had a significant influence on the microhardness of both cements ($P < 0.001$) but had no significant effect on the sorption/solubility of resin cements ($P > 0.05$).

Conclusion: Interposition of monolithic zirconia decreases the microhardness of resin cement especially Panavia SA. In Variolink N, by increasing the chroma saturation of ceramics, the microhardness decreased, however in Panavia SA, it was altered by the shades, but not in a specific pattern. For both cements, there were no statistical differences between the sorption/solubility of samples photo-cured under different shades. There was a reverse correlation between microhardness and water sorption/solubility of both cements.

Keywords: Dual-cure resin cement, microhardness, monolithic zirconia, water sorption/solubility

INTRODUCTION

Recently, monolithic zirconia was developed to overcome the most common drawback facing conventionally veneered zirconia restorations, which is chipping.[1-7] Both conventional and adhesive cements have been suggested for cementation of monolithic zirconia restorations,[5,8,9] while adhesive resin cements due to their low solubility, reduced marginal leakage, high bond strength, and better esthetic properties are preferred.[2,10-15] As monolithic zirconia has semi-translucent nature, it can decrease the light passing through; thus, adversely affecting the optimal polymerization of dual-cure resin cements,[10]

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leading to insufficient mechanical properties,[16] i.e., the microhardness of resin[11,17] or increasing water sorption (Wsp)/water solubility (Wsl).[18] Self-adhesive resin cements (SARCs) are more susceptible to Wsp/Wsl compared with conventional ones due to the incorporation of active monomers.[19] Samimi *et al*.[20] explained that the efficacy of polymerization of dual-cure cements is affected by many factors such as time of irradiation, light intensity, the conductance of light and type, thickness, and shade of ceramics. Many times, the adverse influence of one factor could be compensated by others.[20] However, it was reported that the chemical component of dual-cure resin cements is not enough to ensure optimal microhardness and does not compensate for the reduced light polymerization.[21]

Several authors have investigated the light attenuation effect of ceramics on the mechanical properties of underlying dual-cure resin cements[2,10-12,22-29] using different ceramic types,[12,26,29] thicknesses,[2,10,30] shades,[11,24,29] and light-curing protocols.[19,22,25,27] Nonetheless, any factor that reduces the transmission of light can lead to the weakening of resin cements and increasing the Wsp/Wsl and consequently, jeopardizing the longevity of indirect restorations.[19,30-33]

Previous investigations reported that ceramic thickness compared to shade has more influence on the transmission of light.[24]

However, Passos *et al*. found that DC% of Variolink II was significantly reduced by increasing the saturation of chroma (0M1, 2M2, 5M3) in low thicknesses of feldspathic disks (below 2 mm).[24] According to the results reported by Duran *et al*.,[28] as the darkness of the hybrid ceramic specimens (1M1, 1M2, 2M2, 3M2) increased, the amount of light irradiance in all thicknesses (0.8, 1.5, 2, 3) decreased, which confirms the results of Ilie and Stawarczyk.[34]

To the best of our knowledge, there is no evidence regarding the influence of different shade of monolithic zirconia on microhardness and Wsp/Wsl of the underlying resin cements.

Therefore, the aim of this *in vitro* study was to evaluate the effect of different shades of monolithic zirconia on microhardness and Wsp/Wsl of both SARCs and conventional dual-cure resin cements.

Our null hypotheses were as follows: The shade of monolithic zirconia does not affect the microhardness of dual-cure resin cements, and nor the Wsp/Wsl values of dual-cure resin cements.

MATERIALS AND METHODS

The study was approved by institutional review board. A total of 60 monolithic zirconia discs (precolored, high translucent, Zircostar. Kerox Dental Ltd., Magyarország, Hungary) in the dimension of 10 mm and thickness of 1 mm were milled by computer-aided design/computer-aided manufacturing system in three shades A1, A2 and A3 ($n = 10$). After that, the thickness of each specimen was evaluated with a digital caliper, and if needed ground by silicon carbide grinding paper to the specific thickness for the unification of the samples. The discs having a discrepancy of more than 0.1 mm were excluded from the experiments. Subsequently, specimens were sintered according to the manufacturer instructions for 8 h in 1450°C and then overglazed (Ivoclar Vivadent) on one surface. The specimens were then cleaned ultrasonically in distilled water for 15 min before testing and air-dried individually for 30 s.

Preparation of dual-cure resin samples

A total of 80 resin samples were prepared by placing the dual-cure resin cements (Variolink N, Panavia SA) in cylindrical silicon molds (8 mm in diameter and 1 mm in thickness) [Table 1].

For sample size calculations, a power analysis was performed based on the study of Kim *et al*.[19] The base and catalyst paste of the cements were mixed in 1:1 ratio according to the manufacturer's instructions, inserted into the molds, and then a transparent Mylar's strip and glass slab were placed over the filled orifice. The Mylar strip provided separation between cement and ceramics and also produced an even surface of cements, needed for evaluating Vickers microhardness. Following the removal of the slab, the specimens were light-cured by LED (Blue LEX GT1200), in attenuated mode, with an irradiance of 1200 mW/cm² for 20 s, according to the following chart:

- Group 1: Control group; direct activation (without the presence of ceramic discs)
- Group 2: Resin cement disc cured through A_1 monolithic zirconia
- Group 3: Resin cement disc cured through A_2 monolithic zirconia
- Group 4: Resin cement disc cured through A_3 monolithic zirconia.

Each group was further divided into two subgroups ($n = 10$) according to the two dual-cure resin cements (Variolink N and Panavia SA). In the control group, the light guide tip was held on the Mylar strips, while in the experimental groups, the resin was cured by placing the tip of the

light-curing unit directly onto the glazed surface of monolithic zirconia discs for 20 s. Then, 15 min after the initiation of light-curing, the specimens were removed from the silicone molds.[35] The periphery of specimens was finished by silicon carbide paper to remove the flashes. The specimens were stored in light-proof boxes at 37°C for 24 h postcuring time.

Surface microhardness measurement

A microhardness tester (SCTMC, MHV-1000Z) was used for Vickers microhardness measurement. The tester was set for a 50 gr (0.49N) load applying on the bottom surface of each specimen for 15 s. Three indentations were made for each specimen, and then, the mean values were reported for each one.

Water sorption/solubility measurement

For the Wsp/Wsl test, according to the instructions of ISO 4049,[35] the specimens were kept in a desiccator (Labx Company), as shown in Figure 1, with fresh dry silica gel at (37°C ± 1°C) for 22 h. Then, the specimens were transferred to the other desiccator (23°C ± 1°C) and stored for 2 h.

The specimens were then repeatedly weighed after 24 h intervals by an electronic balance (GR-3000, A&D CL Toshiba) to an accuracy of ± 0.1 mg until reaching a constant mass (m_1), as shown in Figure 2.

According to the ISO specifications, the diameter (*d*) and the thickness (*h*) of specimens were measured in *mm* using a digital micrometer. The volume (*V*) was calculated in *mm* as follows: $V = \pi \times (d/2)^2 \times h$, where $\pi = 3.14$.

After that, the specimens were immersed in a light-proof glass containing distilled water at 37°C for 7 days.

To prevent changes in the PH, the water was replaced daily with fresh distilled water. After this period, the specimens were removed, gently washed with distilled water, air-dried, and then weight to record m_2.

Again, the specimens were moved into the desiccators until the constant mass was reached (m_3). Wsp and Wsl in $\mu g/mm^3$ were calculated using the following equations: $Wsl = (m_1-m_3)/V$; $Wsp = (m_2-m_3)/V$.

Statistical analysis

The data were statistically analyzed using IBM SPSS V.22 (SPSS Inc., IBM Corp., Illinois, USA). In addition to the standard descriptive statistical calculation (mean ± standard deviation), two-way analyses of variance (ANOVA) was conducted to show possible interaction between ceramic shade and type of resin cement. One-way ANOVA and Independent *t*-tests were used to compare different variables between groups. Tukey's honestly significant difference and

Figure 1: The specimens being kept in a desiccator

Figure 2: Weighting the specimens by an electronic balance

Table 1: Resin cements used in this study

Brand name	Composition (lot number)	Manufacturer	Type	Filler loading/particle size
Variolink N	Bis-GMA, UDMA, and TEGDMA (W00243)	Ivoclar Vivadent, Schaan, Liechtenstein	Dual-cure conventional	Base: 73.4%wt. (46.7%vol.) Catalyst: 71.2%wt. (43.6%vol.) 0.04-3.0 μm
Panavia SA Cement Plus	10-MDP –Bis-GMA - TEGDMA - HEMA (3B0127)	Kuraray Noritake Dental Inc. Japan	Dual-cure Self-adhesive	68%wt. (45%vol.) 0.02-20 μm

Bis-GMA: Bisphenol A diglycidylmethacrylate, UDMA: Urethane dimethacrylate, TEGDMA: Triethylene glycol dimethacrylate, MDP: Methacryloyloxydecyl dihydrogen phosphate, HEMA: Hydrophobic aromatic dimethacrylate - 2Hydroxymethacrylate

Tamhane's T2 tests were performed to determine significant differences in subgroup comparisons. The significance level was considered 0.05.

RESULTS

There was a statistically significant interaction effect between ceramic shade and type of resin cement for microhardness ($P < 0.001$), sorption ($P = 0.004$), and solubility ($P = 0.004$) variables. With the Shapiro–Wilk test, the variables were checked for normality that has been established. Therefore, One-way ANOVA and Independent t-tests were used. The mean microhardness of resin cements according to monolithic zirconia shades are shown in Table 2. The mean microhardness of the Variolink N resin cement was significantly higher than Panavia SA for every zirconia shade (all $P < 0.001$). In both resin cements, microhardness values were significantly lower in all experimental groups with different zirconia shades than the control group (all $P < 0.001$). Ceramic shade had a significant influence on the microhardness of Variolink N cement. For this cement, resin samples were polymerized through A_1 ($P = 0.001$), and A_2 ($P = 0.013$) ceramic shades had significantly superior microhardness values compared to the samples polymerized through A_3 ceramic shade. However, there was no statistically significant difference between the microhardness of resin cements polymerized through A_1 and A_2 ceramic shades ($P = 0.712$). Similarly, In Panavia SA cement, the difference between microhardness of resin samples polymerized through A_1 and A_2 ($P = 0.971$) also, through A_2 and A_3 ($P = 0.323$) ceramic shades was not significant. For this cement, resin samples polymerized through A_3 ceramic shade had significantly superior microhardness values compared to the samples polymerized through A_1 ceramic shade ($P = 0.038$).

The Wsp and Wsl values of the resin cements that cured under different ceramic shades are presented in Tables 3 and 4. Overall, Variolink N resin cement exhibited lower sorption (all $P < 0.05$) and solubility ($P < 0.001$) than Panavia SA resin cement with no statistical differences between the curing under different types of ceramic shades ($P = 0.654$, $P = 0.982$). For Panavia SA cement, also, there were no statistical differences between Wsp and Wsl of samples photo-cured under different ceramic shades (all $P > 0.05$). The control group for Panavia SA samples had significantly lower Wsp, and Wsl values compare to other groups with different zirconia shades (all $P < 0.05$).

DISCUSSION

Long-term clinical performance of resin-bonded

Table 2: The mean and standard deviation for microhardness of resin cements under different monolithic zirconia shades

Monolithic zirconia shade	Cement		P*
	Panavia SA	Variolink N	
Without ceramic	24.34±0.79^A	60.59±3.40^A	<0.001
A1	5.78±0.42^B	46.81±3.73^B	<0.001
A2	6.11±0.81B^C	45.06±2.54^B	<0.001
A3	7.08±0.82^C	40.25±3.22^C	<0.001
P$^+$	0.001>	0.001>	

*Independent t-test, $^+$One-way ANOVA F test. In each column, mean values with at least a common letter were not statistically different

Table 3: The mean and standard deviation for water sorption of resin cements under different monolithic zirconia shades

Monolithic zirconia shade	Cement		P*
	Panavia SA	Variolink N	
Without ceramic	35.23±8.46^A	18.26±6.83^A	0.001
A1	56.13±11.51^B	17.19±4.53^A	<0.001
A2	54.79±12.63^B	18.01±9.52^A	<0.001
A3	55.04±9.20^B	21.42±6.58^A	0.002
P$^+$	0.001	0.654	

*Independent t-test, $^+$One-way ANOVA F test. In each column, mean values with at least a common letter were not statistically different

Table 4: The mean and standard deviation for water solubility of resin cements under different monolithic zirconia shades

Monolithic zirconia shade	Cement		P*
	Panavia SA	Variolink N	
Without ceramic	9.43±2.88^A	1.35±0.86^A	<0.001
A1	21.59±8.27^B	1.90±3.54^A	<0.001
A2	20.23±6.25^B	1.59±3.28^A	<0.001
A3	18.71±6.57^B	1.65±2.26^A	<0.001
P$^+$	0.003	0.982	

*Independent t-test, $^+$One-way ANOVA F test. In each column, mean values with at least a common letter were not statistically different

restorations mainly depends on the adequate polymerization of resin cements. Microhardness is a simple and reliable method for determining the amount of resin polymerization.[2,3]

The present study evaluated the effect of monolithic zirconia ceramic in different shades on the microhardness and sorption/solubility of underlying dual-cured resin cements. The results indicated that the first hypothesis was rejected since the shades of monolithic zirconia affected the microhardness. The second hypothesis was accepted; no difference in the sorption/solubility was observed between the cements photo-cured under different ceramic shades.

In this study, monolithic zirconia had negatively affected the microhardness of resin cements, especially Panavia SA resin cement. The direct mode of activation showed higher microhardness values than activation through monolithic zirconia. The mean microhardness [Table 2] in the presence of monolithic zirconia disc lay in the range of 5.8–7 for Panavia SA and 40–46.8 for Variolink N, while this value

was 24.3 and 60.6 in the control group for Panavia SA and Variolink N, respectively. This observation could be related to light attenuation caused by the placement of intervening high translucent monolithic zirconia.[1] Ilie and Stawarczy[34] assessed the amount of light transmitted through monolithic zirconia with different, thicknesses, and polymerization conditions. They found that there were few differences between light irradiance of conventional and translucent zirconia, but glass-ceramics were more translucent than zirconia. In respect to the amount of transmitted light, similar results were achieved by Sulaiman *et al.*[10]

Several previous studies[10,24,26,30,36,37] showed that higher microhardness values are found when using a direct mode of activation. However, some studies have found insignificant differences between direct and indirect light-curing through a 1 mm lucite disc[25] or 1.5 mm zirconia disc.[20]

Gültekİn *et al.*[22] evaluated the depth of cure, and Vickers microhardness of dual-cure resin (Panavia F2.0) cured with two different light-curing units (LED, QHT) under the different thickness of zirconia (0.5, 1, 1.5 mm) layered with feldspathic porcelain. They concluded that light curing, especially with QHT, was not enough for optimal curing of resin under thicker zirconia restoration.

According to our findings, the mean microhardness of the Variolink N was significantly higher than Panavia SA under all ceramic shades. It could be related to various compositions of cements including type, amount, and size variations of filler and monomer, Different catalyst systems, and consequently considerable different mechanical properties in various curing protocols. The percentage of filler content is different between Variolink N and Panavia SA, which are respectively 72.3%wt and 68%wt. Alovisi *et al.*[21] also described two dual-curing resin cements (Rely-X Ultimate and Panavia SA) irradiated through translucent monolithic zirconia with different time protocols. Rely-X Ultimate, which contains a greater percentage of filler, revealed more microhardness than Panavia SA.

Based on our results, the shade of monolithic zirconia had a significant effect on the microhardness, but not on the Wsp and Wsl of resin cements. In Variolink N, by increasing the chroma of monolithic zirconia, the microhardness decreased; as in A3, the lowest microhardness value (40.2) was seen. Microhardness of Panavia SA was altered by shades, but not in the same pattern as Variolink N. In Panavia SA, the difference between microhardness of

resin samples polymerized through A_1 and A_2, and those polymerized through A_2 and A_3 zirconia shades was not significant. The resins that were cured through A3 zirconia shade showed higher microhardness than A1. It may be attributed to different composition of Panavia SA which was differently affected by the curing protocol (i.e., light intensity and time of curing) through different shades of monolithic zirconia. In this study, the ceramic shade showed different effects on the microhardness of the two tested cements, which is in line with the study by Passos *et al.*,[29] in which the authors had assessed the effect of feldspathic ceramic shades (0M1, 2M2, 5M3) on the degree of conversion (DC%) of Variolink II resin cement (A3 and transparent shade). DC% of both cements was decreased only when ceramic shade was 5M3. In other shades, there were no significant differences between groups, except when the transparent cement was cured for a longer time, DC% of 2M2 reached more than 0M1. In a study by Moreno *et al.*,[38] they implied that shade saturation of the ceramics affects the transition of light more than hue. Their findings are in line with the values of Variolink N resin cement in our study.

Kilinc *et al.*[24] evaluated the effect of thickness and shade of IPS Empress (ETC_{1-4}) on three different dual and light cure cements. Based on their results, only one shade of ceramic (ETC3) at 3–4 mm thickness, negatively influenced one resin cement (Calibra). This might be discussed that at that certain thickness, the hue of ETC3 goes to yellow-brown, which affects the polymerization. However, in the remaining two other resin cement groups, not only ceramic shade did not have any effect but also in some thicknesses as the amount of darkness increased, the microhardness of underlying resin numerically increased too, but was not statistically significant.

According to the results of the present study, there are significant differences in the Wsp/Wsl values between the Variolink N and Panavia SA resin cements. In all groups, Panavia SA showed higher amounts of sorption/solubility.

As shown in Table 3, the Wsp of resin cements tested ranged from 17.2 to 56.1 $\mu g/mm^3$. Only Panavia SA, when cured through monolithic zirconia discs, showed higher sorption values (54.7–56.1) than ISO 4049 standard, in which the maximum Wsp value is considered to be 40 $\mu g/mm^3$. On the other hand, direct photo-curing of Panavia SA showed lower Wsp[39] than ISO 4049 standard. Variolink N in all groups showed lower Wsp ranging between 17.2 and 21.4 [Table 3]. Thus, indirect photo-activation through monolithic zirconia adversely affects Wsp/solubility of Panavia SA compared to Variolink N. This difference might be related to different

cement composition.[14] Materials including more HEMA will have higher Wsp.[14,40] HEMA flows more easily in water than Bis-GMA, which is due to its lower molecular weight and hydrophilic chemical structure.[40-43] As Panavia SA cement has HEMA in its composition, it can be considered as the main reason for increased Wsp compared to Variolink N in which Bis-GMA exists.

These findings were in agreement with those of Aguiar *et al.*[31] They assessed the effect of light exposure on Wsp and solubility of SARCs. They observed the highest Wsp in auto polymerized groups, and only G-Cem was not affected by the curing mode. Moreover, in a study by Kim *et al.*[19] dual-curing modes showed higher DC% in all self-adhesive dual-cure resin tested, whereas curing mode did not affect sorption/solubility of some cements.

Tavangar *et al.*[13] investigated the Wsp/solubility and compressive strength of three resin cements and one conventional Glass ionomer. They found that having high Wsp and solubility does not necessarily decrease the compressive strength of resin luting cements. However, we observed a reverse correlation between microhardness and Wsp/solubility in our tested cements.

Using flat monolithic zirconia specimens was a limitation in our study. While in clinical situations, occlusal cusps keep the light tip away from the cements; thus adversely affecting the microhardness.[10] Furthermore, it is recommended to assess the clinical performance of different shades and brands of monolithic zirconia restorations with different curing conditions. As the pH value of saliva affects the sorption/solubility of dental cements,[40] for better clinically simulation of the oral environment, further investigations are warranted to include various storage media.

CONCLUSIONS

Within the limitation of this study, the following conclusions can be drawn:
- Vickers microhardness of resin cements was significantly reduced with the superposition of monolithic zirconia, especially in Panavia SA resin cement
- The shade of monolithic zirconia had a significant influence on the microhardness of resin cements
- Wsp/solubility of both cements was not affected by the shade of monolithic zirconia
- There was a reverse correlation between microhardness and Wsp/solubility of tested resin cements.

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Conflicts of interest

There are no conflicts of interest.

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