



# Microhardness of resin cements after light activation through various translucencies of monolithic zirconia

Sawanya Pechteewang, Prarom Salimee\*

Department of Prosthodontics, Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand

## ORCID

Sawanya Pechteewang

<https://orcid.org/0000-0002-8124-4175>

Prarom Salimee

<https://orcid.org/0000-0002-0511-0650>

**PURPOSE.** This study aimed to investigate the Vickers Hardness Number (VHN) of light- and dual cured resin cements cured through monolithic zirconia specimens (VITA YZ) of various translucencies: translucent (T); high translucent (HT); super translucent (ST); and extra translucent (XT) at 0, 24, and 48 h after curing.

**MATERIALS AND METHODS.** Four zirconia specimens from each translucency were prepared. Two light-cured resin cements (Variolink N LC; VL and RelyX Veneer; RL) and two dual-cured resin cements (Variolink N DC; VD and RelyX U200; RD) were used. The cement was mixed and loaded in a mold and cured for 20 s through the zirconia specimen. The upper surface of cements was tested for VHN using a microhardness tester at 0, 24, and 48 h after curing. The VHN were analyzed using two-way repeated, Brown-Forsythe ANOVA with Games Howell post-hoc analysis and independent t-tests ( $P < .05$ ). **RESULTS.** All cements showed significantly higher VHN from 0 h to 24 h ( $P < .001$ ). At 48 h, the VHN of light-cured cements were significantly lower when cured under the T groups than under XT groups ( $P = .001$  in VL,  $P = .014$  in RL). At each post curing time of each translucency, VD showed higher VHN than VL ( $P < .05$ ), and RD also showed higher VHN than RL ( $P < .05$ ). **CONCLUSION.** The translucency of zirconia has an effect on the VHN for light-cured resin cements, but has no effect on dual-cured resin cements. Dual-cured resin cement exhibited higher VHN than the light-cured resin cement from the same manufacturer. All resin cements showed significantly higher VHN from 0 h to 24 h. [J Adv Prosthodont 2021;13:246-57]

## Corresponding author

Prarom Salimee  
Department of Prosthodontics,  
Faculty of Dentistry, Chulalongkorn  
University, 34 Henri-Dunant Road,  
Pathumwan, Bangkok, 10330  
Thailand  
Tel +6622188532  
E-mail prarom@yahoo.com

Received May 12, 2021 /  
Last Revision August 13, 2021 /  
Accepted August 20, 2021

This study was supported  
by Chulalongkorn University  
graduate school thesis grant,  
Chulalongkorn University,  
Bangkok, Thailand (Grant No:  
GCUGR1225632039M-039).

## KEYWORDS

Resin cement; Zirconia; Hardness

## INTRODUCTION

Zirconia (zirconium dioxide,  $ZrO_2$ ) ceramic has been routinely used in many all-ceramic dental restorations, due to its flexural strength, combined with esthetics and good biocompatibility.<sup>1,2</sup> Zirconia is a polymorphic material

© 2021 The Korean Academy of Prosthodontics

© This is an Open Access article distributed under the terms of the Creative Commons Attribution Non-Commercial License (<http://creativecommons.org/licenses/by-nc/4.0>) which permits unrestricted non-commercial use, distribution, and reproduction in any medium, provided the original work is properly cited.

that occurs in three different crystal structures, depending on the temperature: a monoclinic phase (room temperature to 1170°C) with brittle and low mechanical properties; a tetragonal phase (1170 - 2370°C) with higher mechanical properties; and a cubic phase (2370°C).<sup>3</sup> A volume expansion (3 - 5%) occurs during the cooling process when the tetragonal phase transforms into the monoclinic phase, leading to high stress and crack formation, and a decrease in strength and toughness. This property disallows pure zirconia to be used as a dental restorative material. To maintain the beneficial mechanical properties of the tetragonal phase at room temperature, stabilization can be achieved by adding various oxides such as calcium oxide (CaO), magnesium oxide (MgO), yttrium oxide (Y<sub>2</sub>O<sub>3</sub>), and cerium oxide (CeO<sub>2</sub>). Among these stabilizers, the addition of 3 mol% diyttrium trioxide (Y<sub>2</sub>O<sub>3</sub>; also termed yttrium (III) oxide or yttria) is widely used for dental zirconia, and is known as '3% yttria-stabilized tetragonal zirconia polycrystals' (3Y-TZP).<sup>4</sup> When zirconia is subjected to heat or low stress such as grinding, sandblasting, or steam sterilizing, the tetragonal phase can transform into the monoclinic phase and influence its mechanical properties by a 3 - 5% of volume expansion which generates a compressive stress layer around the progressing internal crack tip. The stress layer prevents crack propagation, leading to high flexural strength (about 900 - 1,400 MPa) and high fracture toughness (about 5 - 10 MPa.m<sup>1/2</sup>), which is called 'transformation toughening'.<sup>5</sup>

Opacity was a disadvantage of the first generation of 3Y-TZP, and a technique of veneering zirconia with esthetic porcelain to improve appearance was introduced.<sup>1</sup> The most common problem of veneered zirconia was chipping of the veneered porcelain,<sup>6</sup> and this prompted the development of full-contour restorations of 'monolithic' zirconia. However, opacity was still a problem as the early monolithic zirconia was limited to posterior teeth.<sup>7</sup> To improve the translucency, various techniques have been developed such as a decrease in grain size,<sup>8,9</sup> elimination or reduction of the concentration of some additives such as alumina,<sup>10</sup> addition of 0.2 mol% lanthanum oxide,<sup>11</sup> and an increase in sintering temperature.<sup>12</sup> Presently, greater percentage of yttria is added to monolithic zirconia

to achieve a transparent phase. Variants of yttria content, including 4 mol% or 5 mol% yttria partially-stabilized zirconia (4Y- or 5Y-PSZ)<sup>13</sup> and 8 mol% yttria fully stabilized zirconia (8Y-FSZ),<sup>14</sup> yield a higher stabilized cubic phase. As the yttria content increases, cubic phase increases, which were reported to correlate with high translucency values.<sup>15</sup> The isotropic property of the cubic phase reduces light scattering from grain boundaries and results in more translucent zirconia than the early generations.<sup>12</sup> Nevertheless, transformation toughening occurs less in 5Y-PSZ,<sup>13,16</sup> and is absent in 8Y-FSZ,<sup>16</sup> as the stabilized cubic phase does not transform at room temperature, resulting in inferior flexural strength and toughness.<sup>13,16</sup>

Resin cements have been promoted for the cementation of conventional zirconia restorations due to their superior properties over conventional cements.<sup>17-19</sup> The opacity of conventional zirconia masks the light transmission, and thus, dual-cured resin cements are preferable in clinical applications with the benefit of being both auto-cured and light-cured, resulting in a sufficient degree of conversion underneath the restoration. The improvement in translucency of monolithic zirconia led to various optical property evaluations. Although the 4 - 6% Y-TZPs were reported to be radiopaque in x-ray investigation, which mainly depend on the thickness,<sup>20</sup> blue light transmission are accessible through these translucent monolithic zirconia, depending on shade and thickness.<sup>21</sup> An investigation by Sulaiman *et al.*<sup>22</sup> on the degree of conversion of dual-cured resin cements under various monolithic zirconia specimens noted a remarkable result that Katana (Kuraray Noritake, Tokyo, Japan) and Prettau Anterior (Zirkonzahn, Gais, Italy), which has a large difference of yttria content (4.5 - 6% and < 12%), showed a comparable degree of conversion. Therefore, the amount of yttria content might be one of the factors that affect the light transmission through monolithic zirconia. Other characteristics of the zirconia variants may have contributed to these results, including the grain size or the sintering parameters.<sup>23</sup>

Since the new generation of monolithic zirconia provides high translucency similar to lithium disilicate ceramic,<sup>24</sup> more anterior applications, such as crowns, bridges, and veneers, become possible.<sup>25,26</sup>

It would be advantageous for light polymerization of resin cement below the restorations, enhancing the use of light-cured resin cements with the advantages of easier excess cement removal, extended working time, and good color stability, compared to dual-cured cements.<sup>27</sup> The degree of light transmission through the translucent monolithic zirconia should be investigated in order to assess the degree of conversion of light- compared to dual-cured resin cements, with respect to the different levels of translucency available. This would apply to determine the degree of polymerization of resin cements for more success in clinical trials. The investigation of the surface hardness of a resin-based material is an accepted method for evaluating physical properties and degree of conversion.<sup>28-31</sup> Many previous studies have used surface hardness, such as Knoop or Vickers, to test the degree of polymerization efficacy of resin cements, and a correlation has been reported;<sup>29,31,32</sup> as the degree of conversion increases, the hardness also increases.<sup>29</sup>

The purpose of this study was to investigate the Vickers hardness number (VHN) of light- and dual-cured resin cements after light transmission

through various translucencies of monolithic zirconia at three different times after curing. Three null hypotheses were tested: (1) monolithic zirconia specimens of different translucencies have no effect on the VHN of resin cement; (2) there is no difference in VHN between light- and dual-cured resin cements after light curing through monolithic zirconia; (3) there is no difference in the VHN of a resin cement at three different times after curing.

## MATERIALS AND METHODS

Four specimens from the four translucencies of monolithic zirconia (VITA YZ; VITA Zahnfabrik, Bad Säckingen, Germany): translucent (T); super translucent (ST); high translucent (HT); and extra translucent (XT) (Table 1), were prepared using CAD-CAM software (3Shape Dental System, 3Shape A/S, Copenhagen, Denmark) and sintered (VITA Zyrcomat 6000 MS; VITA Zahnfabrik) to the final size of square-shaped specimens (10 × 10 mm) with 1 ± 0.05 mm thickness, measured by a digital Vernier caliper (Series 500, Mitutoyo, Kanagawa, Japan). The specimens were pol-

**Table 1.** Translucent monolithic zirconia used in this study (Shade A3)

Material	Abbreviation	Type	Sintering temperature* (°C)	Composition* (wt%)	Translucency percentage* (%)	Lot no.
VITA YZ T	T	Translucent ZrO <sub>2</sub>	1530	ZrO <sub>2</sub> 90.4 - 94.5, Y <sub>2</sub> O <sub>3</sub> 4 - 6, HfO <sub>2</sub> 1.5 - 2.5, Al <sub>2</sub> O <sub>3</sub> 0 - 0.3, Fe <sub>2</sub> O <sub>3</sub> 0 - 0.3	32	51280
VITA YZ HT	HT	High translucent ZrO <sub>2</sub>	1450	ZrO <sub>2</sub> 90.4 - 94.5, Y <sub>2</sub> O <sub>3</sub> 4 - 6, HfO <sub>2</sub> 1.5 - 2.5, Al <sub>2</sub> O <sub>3</sub> 0 - 0.3, Er <sub>2</sub> O <sub>3</sub> 0 - 0.5, Fe <sub>2</sub> O <sub>3</sub> 0 - 0.3	42	77250
VITA YZ ST	ST	Super translucent ZrO <sub>2</sub>	1530	ZrO <sub>2</sub> 88.4 - 92.5, Y <sub>2</sub> O <sub>3</sub> 6 - 8, HfO <sub>2</sub> 1.5 - 2.5 Other oxides ≤ 1	46	74890
VITA YZ XT	XT	Extra translucent ZrO <sub>2</sub>	1450	ZrO <sub>2</sub> 86.4 - 90.5, Y <sub>2</sub> O <sub>3</sub> 8 - 10, HfO <sub>2</sub> 1.5 - 2.5 Other oxides ≤ 1	50	75410

ZrO<sub>2</sub>: zirconium oxide; Y<sub>2</sub>O<sub>3</sub>: yttrium oxide; HfO<sub>2</sub>: Hafnium dioxide; Al<sub>2</sub>O<sub>3</sub>: Aluminium oxide; ERDO<sub>3</sub>: Erbium oxide; Fe<sub>2</sub>O<sub>3</sub>: Iron (III) oxide.

\*According to the manufacturers' information.

ished by a single operator using VITA Suprinity Polishing Set, as recommended by the manufacturer's instructions, ultrasonically cleaned (VGT-1990 QTD, GT Sonic, Guangdong, China) in distilled water for 10 min and dried with absorbent paper. The translucency percentage of each specimen was calculated from the obtained opacity percentage measured by the spectrophotometer (Ultrascan PRO 74-SD-03-10, HunterLab, Reston, VA, USA) subtracted from 100.

Translucency percentage = 100 - Opacity percentage

Two light-cured resin cements (Variolink N LC by Ivoclar Vivadent AG, Schaan, Liechtenstein; VL and RelyX Veneer by 3M-ESPE, St. Paul, MN, USA; RL) and two dual-cured resin cements (Variolink N DC by Ivoclar Vivadent AG; VD and RelyX U200 by 3M-ESPE; RD) were used (Table 2). The resin cements were mixed following the manufacturers' instructions and loaded into a cavity (2 mm deep × 5 mm in diameter) in a Type 4 gypsum mold in a PVC cylinder (25 mm height × 22 mm in diameter). Then, a thin glass slide (0.15 mm thick) was placed over the mold using finger pressure to expel excess cement. Zirconia specimens in the experimental groups, or a 1-mm thick glass

slide in the control group, were placed over the thin glass slide, and the tip of a light curing unit, which delivers light between 400 and 515 nm (Elipar Trilight, 3M-ESPE, St. Paul, MN, USA), was positioned directly on the top of the of the assembly specimen (Fig. 1A). The cement was light-cured for 20 s at an intensity of 800 mW/cm<sup>2</sup>, and the light intensity was checked before each curing. Sample preparation and light activation were done in a chairside dark chamber (Dent-Mate Co., Bangkok, Thailand). Five specimens were prepared for each group, as this setup provided sufficient statistical power (using  $\alpha = .05$  and a power of 99%) from the pilot study by using statistical power analysis program (G Power 3.1, Heinrich-Heine-Universität, Düsseldorf, Germany). This resulted in 16 experimental groups and four control groups, totaling 100 specimens for each post-curing measurement time (0 h, 24 h, and 48 h) (Fig. 2).

The specimens of the 0 h groups were subjected to test immediately for the mean VHNs, while for the 24 h and 48 h groups, they were left dry in a dark chamber at 37°C until testing. The upper surface of each cement specimen was uncovered to be tested on

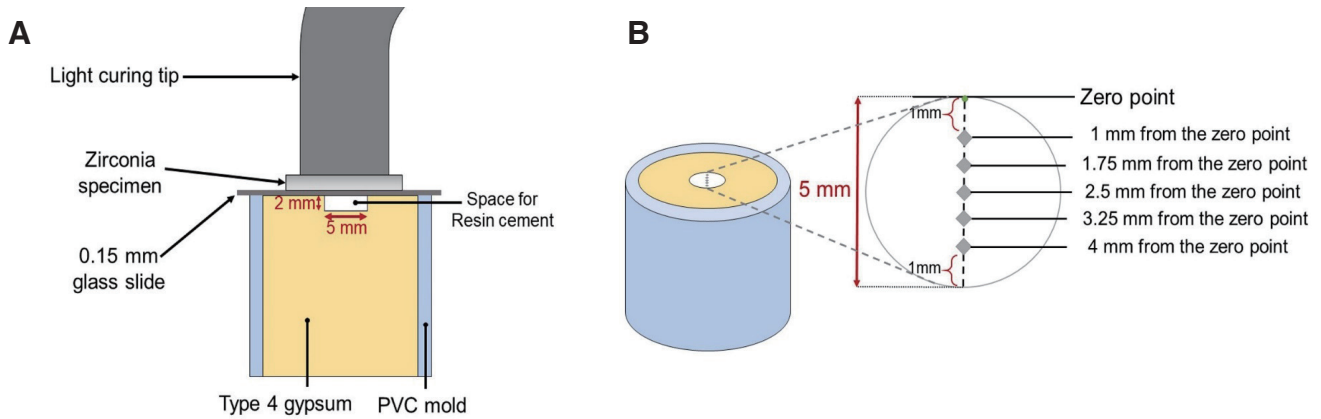
**Table 2.** Light-cured and dual-cured resin cements used in this study

Material	Abbreviation	Type	Shade	Composition* (wt%)	Lot no.
Variolink N LC	VL	Light-cured resin cement	Transparent (Base paste only)	Barium glass filler and mixed oxide (48.4%), dimethacrylates (BisGMA, UDMA, and TEGDMA) (26.3%), ytterbium trifluoride (25.0%), initiators and stabilizers (0.3%), pigments (< 0.1%)	YZ1282
Variolink N DC	VD	Dual-cured resin cement	Transparent (high viscosity)	Base: Barium glass filler and mixed oxide (48.4%), dimethacrylates (BisGMA, UDMA, and TEGDMA) (26.3%), ytterbium trifluoride (25.0%), initiators and stabilizers (0.3%), pigments (< 0.1%) Catalyst: Barium glass filler and mixed oxide (52.2%), dimethacrylates (22.3%), ytterbium trifluoride (25.0%), initiators and stabilizers (0.8%), pigments (< 0.1%)	YZ1282 Y15347
RelyX Veneer Cement	RL	Light-cured resin cement	Translucent	BisGMA and TEGDMA polymer, Zirconia/silica, and fumed silica fillers (66%)	NA13299
RelyX U200	RD	Dual-cured resin cement	Translucent	Base: methacrylate monomers containing phosphoric acid groups, TEGDMA, silanated fillers, initiator components, stabilizers, rheological additives Catalyst: methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, rheological additives, pigments	4946182

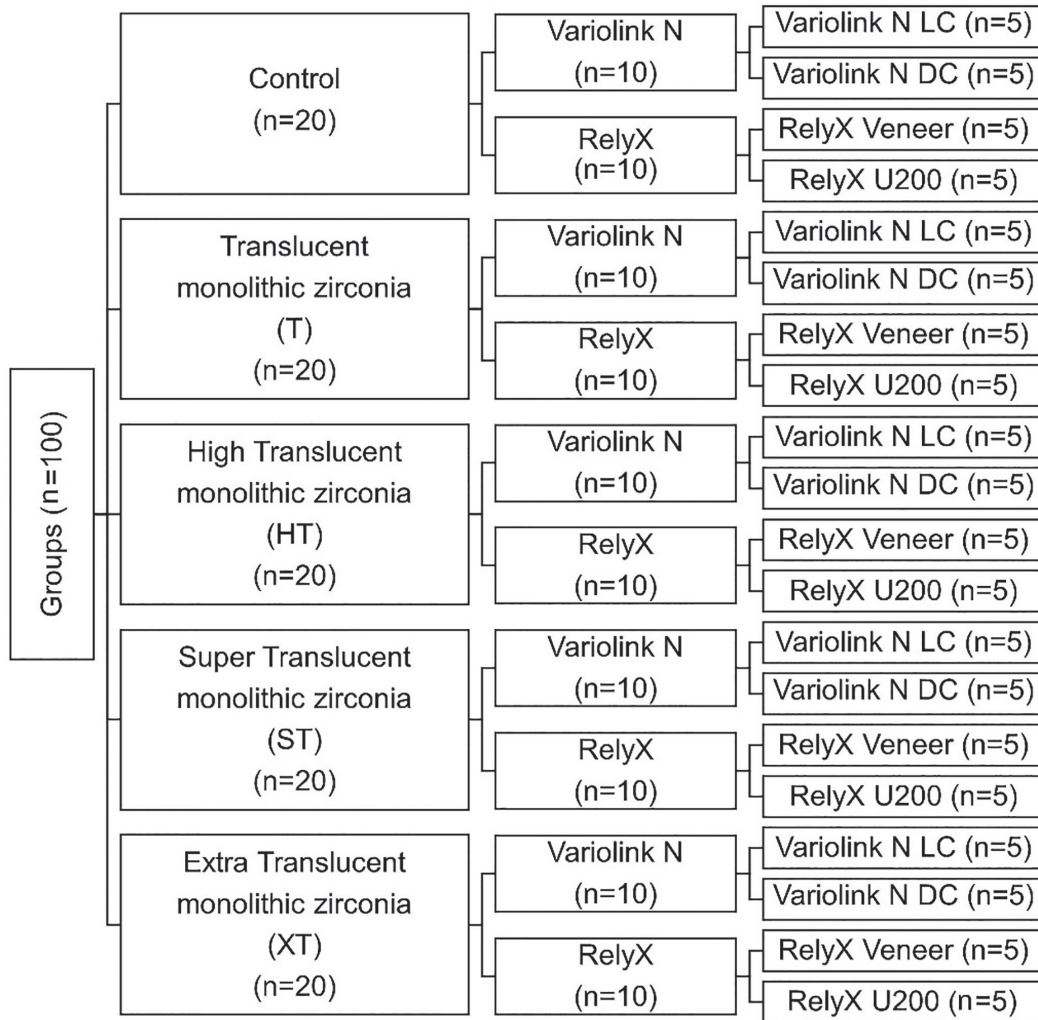
DC: dual-cured; LC: light-cured.

BisGMA: Bisphenol-A-diglycidylether dimethacrylate; UDMA: Urethane Dimethacrylate; TEGDMA: triethylene glycol dimethacrylate.

\*According to the manufacturers' technical data, safety data sheets and instruction for use.



**Fig. 1.** (A) Diagram shows the light-curing assembly of the specimen, (B) Diagram shows the hardness indentation points on the upper surface of resin cement specimen.



**Fig. 2.** Control and experimental groups in this study.

Vickers hardness tester (FM-810, Future-Tech Corp., Kawasaki, Japan) with a load of 300 g for 15 s in a linear pattern as shown in Fig. 1B. The interface of the resin cement and the gypsum surface was marked as the zero point. Five measurements of each sample were obtained and the mean VHN was calculated. The VHN percentage was calculated from the mean VHNs of each experimental group and compared with the VHN of its control group at the final measurement of 48 h.

The data were analyzed by IBM SPSS Statistics for Windows version 22 (IBM SPSS Statistics, IBM Corp, Chicago, IL, USA). Two-way repeated-measured ANOVA was performed to determine the effect of different translucencies of zirconia and resin cements and their interactions on the mean VHNs, with different measurement time as a repeated factor. Brown-Forsythe ANOVA with Games Howell post hoc analysis and independent t-tests were used to analyze the difference among groups. All *P*-values < .05 were considered statistically significant.

## RESULTS

The results of two-way repeated-measured ANOVA are shown in Table 3. The VHN was significantly affected by all independent variables of translucency, resin cement, and measurement time, all two-way interactions except the measurement time and translucency, and the three-way interaction of all parameters.

The means and standard deviations of VHN for VL

and VD are shown in Table 4, and those for RL and RD are shown in Table 5.

According to the translucencies, the higher VHNs of all resin cements were associated with the higher translucencies of zirconia in the order XT > ST > HT > T. At 48 h, the mean VHNs of light cured cements (VL and RL) under zirconia XT were significantly higher than those under zirconia T (*P* < .05), while the significant difference was not observed in both dual cured resin cements (VD and RD).

According to the measurement time, all resin cements showed significantly higher mean VHNs of 24 h group than the 0 h group (*P* < .05), but most of the values were not significantly different when compared with 48 h group (*P* > .05). At each measurement time, the mean VHNs of both dual cured resin cements were significantly higher than those of light cured resin cements from the same manufacturer at each level of the same translucency (*P* < .05)

According to the VHN percentage (Table 6), the lowest translucency of zirconia T had the lowest mean VHN percentage among all translucencies for every cement and measurement time. At 48 h, the light-cured cement VL and RL showed higher VHN percentage (91.4 and 95.7) than dual-cured VD and RD (88.5 and 89.6). In contrast, the highest translucency of zirconia XT showed the highest VHN percentage among all translucencies for every cement and measurement time. The light-cured cements showed higher VHN percentage (VL = 99.1% and RL = 99.6%) than the dual-cured cements from the same manufacturer (VD = 94.9% and RD = 98.0%) at 48 h.

**Table 3.** Two-way repeated measures ANOVA statistical analysis

Source	Type III sum of squares	df	Mean squares	F	<i>P</i>
Time	35268.95	1.86	18973.94	9440.31*	< .001
Cement	7261.33	3.00	2420.44	1208.36*	< .001
Translucency	895.83	4.00	223.96	111.81*	< .001
Cement*translucency	104.43	12.00	8.70	4.34*	< .001
Time*cement	1780.44	5.58	319.28	158.85*	< .001
Time*translucency	9.35	7.44	1.26	0.63	.744
Time*cement*translucency	90.03	22.31	4.04	2.01*	.008

df: Degrees of freedom, F: F value

**Table 4.** Mean (SD) VHN of VL and VD

Resin cement	Zirconia	Translucency percentage	Time		
			0 h Mean (SD)	24 h Mean (SD)	48 h Mean (SD)
VL	T	11.3	15.59 (0.81) <sup>a, A</sup>	44.03 (0.59) <sup>a, B</sup>	47.63 (0.92) <sup>a, C</sup>
	HT	18.4	16.95 (0.95) <sup>ab, A</sup>	46.05 (0.88) <sup>ab, B</sup>	50.77 (0.33) <sup>b, C</sup>
	ST	19.7	16.95 (2.21) <sup>ab, A</sup>	46.34 (2.92) <sup>abc, B</sup>	51.47 (1.46) <sup>b, C</sup>
	XT	22.9	17.00 (1.14) <sup>ab, A</sup>	47.60 (0.64) <sup>bc, B</sup>	51.62 (0.38) <sup>bc, C</sup>
	Control	100	19.03 (0.79) <sup>b, A</sup>	48.32 (1.87) <sup>abc, B</sup>	52.10 (1.12) <sup>bc, C</sup>
VD	T	11.3	32.50 (1.90) <sup>c, A</sup>	52.02 (2.16) <sup>cd, B</sup>	52.45 (2.29) <sup>abd, B</sup>
	HT	18.4	34.87 (1.61) <sup>cd, A</sup>	54.41 (2.06) <sup>d, B</sup>	55.45 (0.91) <sup>de, B</sup>
	ST	19.7	35.71 (1.68) <sup>cd, A</sup>	55.05 (0.85) <sup>d, B</sup>	56.02 (0.42) <sup>de, B</sup>
	XT	22.9	37.78 (0.49) <sup>d, A</sup>	55.84 (1.72) <sup>d, B</sup>	56.26 (2.01) <sup>cde, B</sup>
	Control	100	40.27 (0.59) <sup>e, A</sup>	58.68 (3.85) <sup>d, B</sup>	59.26 (2.15) <sup>e, B</sup>

Note: The same lower-case letters indicate no significant column differences (Brown-Forsythe ANOVA,  $P > .05$ ). The same upper-case letters indicate no significant row differences (repeated-measured ANOVA in row,  $P > .05$ ). SD: Standard deviation. T: Translucent; HT: High Translucent; ST: Super Translucent; XT: Extra Translucent.

**Table 5.** Mean (SD) VHN of RL and RD

Resin cement	Zirconia	Translucency percentage	Time		
			0 h Mean (SD)	24 h Mean (SD)	48 h Mean (SD)
RL	T	11.3	30.19 (1.08) <sup>a, A</sup>	51.14 (0.62) <sup>a, B</sup>	51.76 (0.84) <sup>a, B</sup>
	HT	18.4	33.05 (0.79) <sup>b, A</sup>	52.31 (0.77) <sup>ab, B</sup>	52.33 (1.31) <sup>ab, B</sup>
	ST	19.7	33.83 (1.96) <sup>abc, A</sup>	52.46 (0.82) <sup>ab, B</sup>	52.61 (1.03) <sup>ab, B</sup>
	XT	22.9	34.17 (1.00) <sup>bd, A</sup>	53.86 (1.50) <sup>abc, B</sup>	53.89 (0.74) <sup>b, B</sup>
	Control	100	34.48 (1.13) <sup>be, A</sup>	54.06 (0.22) <sup>bc, B</sup>	54.11 (0.62) <sup>b, B</sup>
RD	T	11.3	34.07 (1.25) <sup>bf, A</sup>	53.38 (1.12) <sup>ab, B</sup>	56.33 (0.33) <sup>c, C</sup>
	HT	18.4	36.71 (0.98) <sup>cdef, A</sup>	55.95 (1.88) <sup>bcd, B</sup>	56.68 (0.98) <sup>c, B</sup>
	ST	19.7	37.07 (0.67) <sup>ce, A</sup>	56.39 (1.10) <sup>cd, B</sup>	57.51 (0.57) <sup>c, B</sup>
	XT	22.9	37.84 (0.39) <sup>c, A</sup>	58.01 (1.04) <sup>d, B</sup>	61.63 (2.71) <sup>cd, B</sup>
	Control	100	38.22 (0.76) <sup>c, A</sup>	61.86 (0.77) <sup>e, B</sup>	62.89 (0.33) <sup>d, B</sup>

Note: The same lower-case letters indicate no significant column differences (Brown-Forsythe ANOVA,  $P > .05$ ). The same upper-case letters indicate no significant row differences (repeated-measured ANOVA in row,  $P > .05$ ). SD: Standard deviation. T: Translucent; HT: High Translucent; ST: Super Translucent; XT: Extra Translucent.

**Table 6.** VHN percentages of experimental groups compared to control groups, at 48-h

Zirconia	Translucency percentage	Mean VHN (%)											
		VL			VD			RL			RD		
		0 h	24 h	48 h	0 h	24 h	48 h	0 h	24 h	48 h	0 h	24 h	48 h
T	11.3	29.9	84.5	91.4	54.8	87.8	88.5	55.8	94.5	95.7	54.2	84.9	89.6
HT	18.4	32.5	88.4	97.5	58.8	91.8	93.6	61.1	96.7	96.7	58.4	89	90.1
ST	19.7	32.5	88.9	98.8	60.3	92.9	94.5	62.5	97	97.2	58.9	89.7	91.5
XT	22.9	32.6	91.4	99.1	63.8	94.2	94.9	63.2	99.5	99.6	60.2	92.2	98
Control	100	36.5	92.7	100	68	99	100	63.7	99.9	100	60.8	98.4	100

T: Translucent; HT: High Translucent; ST: Super Translucent; XT: Extra Translucent.

## DISCUSSION

The surface hardness of a resin-based luting cement has been shown to be correlated with its degree of conversion, and therefore, the microhardness test is a reliable indirect method to evaluate the degree of conversion of the resin cement.<sup>28,29,31,33</sup> As the degree of conversion increases, a higher hardness of the material will be evident.<sup>29</sup> A lower degree of conversion of resin cement could lead to poor properties such as marginal leakage, lower color stability, increased water sorption, change in dimensional stability, and other factors that can lead to restoration failure.<sup>27,32,34</sup>

According to the effect of different translucencies of zirconia on the VHN of the resin cements, zirconia XT was associated with a higher VHN than zirconia T, except for the dual-cured cement VD and RD groups; therefore, the first null hypothesis was partially rejected. The results showed that the VHN of the resin cement was significantly higher when cured through the zirconia XT, which has the highest translucency. The VHN tended to be less in the lower translucency groups of ST, HT and T, respectively). High amounts of yttria in Y-TZP were reported to correlate with high translucency values,<sup>15</sup> which were associated with its microstructural character and chemical composition.<sup>23</sup> The increasing yttria content from 3 - 9 wt% was related to the increased grain size and cubic phase, thus leading to an increase in translucency of zirconia.<sup>35</sup> The same materials used in this present study (VITA YZ HT, ST, and XT) were investigated by Sen and Isler<sup>23</sup> for the crystalline phases after sintering, by using X-ray Diffraction (XRD). They revealed that the highest percentage of the cubic phase was found in VITA YZ XT (32.9 wt%), followed by ST (27.2 wt%) and HT (13.7 wt%), and zirconia XT showed the largest mean grain size, followed by zirconia ST and HT. This showed the same trend of translucent level obtained in this study and zirconia XT showed the highest translucency and significantly allowed a greater VHN value, which related to the degree of conversion of resin cements underneath. The degree of conversion of the resin cements under monolithic zirconia directly depended on the light irradiance and radiant exposure. Furthermore, it decreased according to increasing in thickness and differed among

brands of monolithic zirconia.<sup>21,22</sup>

The translucency percentage of the zirconia specimens in this study was measured to confirm the levels of translucency according to the manufacturer's information. The value obtained in our study differed from the manufacturer's data, which might be due to the different equipment and methods used. The translucency percentage of zirconia T was the lowest (11.3%) and was lower than zirconia HT (18.4%), which contain the same amount of yttria, while its value was about half of zirconia XT (22.9%). According to the manufacturer's information, a higher translucency of zirconia HT than zirconia T might be due to a small amount of erbium oxide ( $\text{Er}_2\text{O}_3$ ) added and a lower sintering temperature applied, which may affect the crystalline structure. Note that the translucency percentage of zirconia HT and ST (19.7%) were closer to that of zirconia XT. However, the final measurement at 48 h showed that the VHNs of the cements under zirconia HT and ST were significantly lower than those of zirconia XT and were not statistically different from zirconia T. This may suggest that these ranges of increased translucency of zirconia HT and ST were not sufficient to perform a statistical difference of VHN compared to zirconia T.

Both dual-cured resin cements showed higher VHN than the light-cured cements from the same manufacturer (VD > VL; RD > RL). Therefore, the second null hypothesis was rejected. For Variolink N, the VHNs in light-curing mode (VL) were significantly lower than the dual-curing mode (VD). Hofmann *et al.*<sup>30</sup> also reported that VHNs of dual-cured resin cements by using light-curing mode with base paste alone showed lower VHNs than by using dual-curing mode with the mixture of base and catalyst paste through leucite-reinforced glass-ceramic. This result may be explained by the less content of inorganic filler in the base paste of Variolink N but more dimethacrylates compared to its catalyst paste. Furthermore, fewer amounts of initiators and stabilizers may contribute its slow rate of polymerization. Thus, when using the mixed pastes in dual-cured mode, a higher amount of fillers and less resin matrix contents from catalyst paste might explain for higher VHNs than using in light-cured mode. For the RelyX groups, RD contains a greater proportion of filler particles (72 wt% filler) than RL (66 wt%



filler).<sup>34,36</sup> A higher filler loading results in a higher post-curing surface hardness, as rigid inorganic particles provide a strong interfacial bond between the filler particles and resin matrix, which directly contributes to higher hardness.<sup>37,38</sup> Apart from filler content, the filler size also plays a role in the surface hardness of cement.<sup>30</sup>

According to the measurement times, almost all groups showed a significantly higher VHN at 24 h than 0 h, while the values were not significantly higher from 24 h to 48 h, except for VL and RD under zirconia T. Consequently, the third null hypothesis was partially rejected. These results were in agreement with Yan *et al.*,<sup>33</sup> who investigated both degree of conversion and VHN of two light-cured and two dual-cured resin cements, from 1 min to 7 days after light activation. They showed that the degree of conversion and the VHN values significantly increased during the first one hour and the values were not significantly different from 24 h to 7 days. The VHN reflects the curing state of the resin material and perform a continuation of the polymerization reaction,<sup>39</sup> which were explained by the presence of free monomers with potential in mobility to allow low rate interaction.<sup>40</sup> For light-cured resin materials, light exposure causes a rapid increase in viscosity, especially with high-intensity curing lights.<sup>41</sup> Even though the first exposure to the light curing unit produces immediate curing and hardening, polymerization still proceeds for a period of up to 24 h.<sup>33,41</sup> The so-called 'dark polymerization' of unreacted monomers can continue the after the cessation of light activation.<sup>42</sup> During the early period of polymerization, the cement with a slow rate of conversion required a longer time to be completely polymerized.<sup>33</sup> Note that the VHNs of VL at 0 h was relatively low (15.59-19.03) compared to those of VD, RL and RD which range from 30.19 to 40.27. This may imply that using Variolink N in light curing mode showed the slowest initial polymerization, as mentioned above, which were much slower than using in dual curing mode of VD. This might due to the higher percentage of initiators and stabilizers in catalyst paste that can accelerate the initial polymerization immediately after light activation. However, the dark polymerization of VL could adequately compensate its low initial light polymerization and reach its ulti-

mate value with a slower processing time.

According to the data of VHN percentage, at 48 h, the range of the value of light-cured resin cements (91.4 - 99.6%) was higher than dual-cured resin cements (88.5 - 98.0%), depending on the zirconia translucency. This may suggest that the light-cured resin cements exhibit higher degree of conversion than the dual-cured resin cements under any translucency of zirconia. However, dual-cured resin cements might be advantageous that the level of translucency of zirconia did not affect the VHN values. Typically, dual-cured resin cement seems to have different relative contributions of light- and chemical-cured mechanisms depending on brands.<sup>33,43,44</sup> This might be the differences in material specifications that influence the different initial rates of conversion after light activation and affect the final degree of conversion.<sup>33,45</sup>

The zirconia specimens from the same manufacturer were used in this study to eliminate the influence from the differences in brands as reported in the study of Sulaiman *et al.*<sup>22</sup> Therefore, the influence of the translucency level can be emphasized. The shade of a cement has been reported to affect polymerization; darker shades showed lower microhardness.<sup>31,46</sup> So-called 'transparent' shades of resin cement exhibited more light absorption than opaque shades, being associated with increased curing depth and microhardness.<sup>47</sup> In the present study, the 'transparent' shades of Variolink N (VL and VD) and the 'translucent' shade of RelyX (RL and RD) were used to minimize the impact of shade on the VHN and focused on the effect of the translucency of the zirconia and the post-curing interval.

The limitation of this study by using one brand and one specific shade of translucent monolithic zirconia to investigate the influence of different translucencies may restrict the obtained result. Further studies of increasing the light curing time or intensity for low-translucency zirconia should be conducted to clarify the relationship of curing time and polymerization of the underlying cement. Moreover, longer storage time might be evaluated since some studies had reported the significantly increased in the degree of conversion and VHN value after 7 days.<sup>48,49</sup>

Clinicians should consider that an increase in the translucency of monolithic zirconia restorations could

increase light penetration, which may or may not affect the polymerization efficacy of the resin cement used, and possibly long-term durability of the restoration. The dual-cured resin cements might exhibit higher hardness and do not depend on the translucency, while the light-cured resin cements might result in a higher degree of conversion and the hardness depends on the translucency, and some cements may show slow initial rate of polymerization.

## CONCLUSION

Within the limitations of this study, it can be concluded that different translucencies of zirconia have an effect on the VHN for light-cured resin cements (RL and VL), but do not have an effect on dual-cured resin cements (RD and VD). Dual-cured resin cement exhibit a higher VHN than the light-cured resin cement from the same manufacturer. All resin cements show significantly higher VHN from 0 h to 24 h, while the VHN do not significantly higher from 24 h to 48 h, except in VL and RD under zirconia T.

## ACKNOWLEDGEMENTS

The authors are grateful to Prof. Martin Tyas, Melbourne Dental School, for the manuscript revision, Asst. Prof. Soranun Chantarangsu and Dr. Nareudee Limpuangthip, Chulalongkorn University, for their assistance in statistical analysis.

## REFERENCES

1. Akagawa Y, Hosokawa R, Sato Y, Kamayama K. Comparison between freestanding and tooth-connected partially stabilized zirconia implants after two years' function in monkeys: a clinical and histologic study. *J Prosthet Dent* 1998;80:551-8.
2. Ichikawa Y, Akagawa Y, Nikai H, Tsuru H. Tissue compatibility and stability of a new zirconia ceramic in vivo. *J Prosthet Dent* 1992;68:322-6.
3. Miyazaki T, Nakamura T, Matsumura H, Ban S, Kobayashi T. Current status of zirconia restoration. *J Prosthodont Res* 2013;57:236-61.
4. Denry I, Kelly JR. State of the art of zirconia for dental applications. *Dent Mater* 2008;24:299-307.
5. Vagkopoulou T, Koutayas SO, Koidis P, Strub JR. Zirconia in dentistry: part 1. Discovering the nature of an upcoming bioceramic. *Eur J Esthet Dent* 2009;4:130-51.
6. Koenig V, Vanheusden AJ, Le Goff SO, Mainjot AK. Clinical risk factors related to failures with zirconia-based restorations: an up to 9-year retrospective study. *J Dent* 2013;41:1164-74.
7. McLaren EA, Lawson N, Choi J, Kang J, Trujillo C. New high-translucent cubic-phase-containing zirconia: clinical and laboratory considerations and the effect of air abrasion on strength. *Compend Contin Educ Dent* 2017;38:e13-6.
8. Xiong Y, Fu Z, Pouchly V, Maca K, Shen Z. Preparation of transparent 3y-tzp nanoceramics with no low-temperature degradation. *J Am Ceram Soc* 2014;97:1402-6.
9. Zhang F, Vanmeensel K, Batuk M, Hadermann J, Inokoshi M, Van Meerbeek B, Naert I, Vleugels J. Highly-translucent, strong and aging-resistant 3Y-TZP ceramics for dental restoration by grain boundary segregation. *Acta Biomater* 2015;16:215-22.
10. Zhang H, Li Z, Kim B-N, Morita K, Yoshida H, Hiraga K, Sakka Y. Effect of alumina dopant on transparency of tetragonal zirconia. *J Nanomater* 2012;2012:1-5.
11. Zhang F, Inokoshi M, Batuk M, Hadermann J, Naert I, Van Meerbeek B, Vleugels J. Strength, toughness and aging stability of highly-translucent Y-TZP ceramics for dental restorations. *Dent Mater* 2016;32:e327-37.
12. Zhang Y. Making yttria-stabilized tetragonal zirconia translucent. *Dent Mater* 2014;30:1195-203.
13. Zhang Y, Lawn BR. Novel zirconia materials in dentistry. *J Dent Res* 2018;97:140-7.
14. Ghosh A, Suri AK, Pandey M, Thomas S, Rama Mohan TR, Rao BT. Nanocrystalline zirconia-yttria system—a raman study. *Mater Lett* 2006;60:1170-3.
15. Sulaiman TA, Abdulmajeed AA, Donovan TE, Ritter AV, Vallittu PK, Närhi TO, Lassila LV. Optical properties and light irradiance of monolithic zirconia at variable thicknesses. *Dent Mater* 2015;31:1180-7.
16. Shahmiri R, Standard OC, Hart JN, Sorrell CC. Optical properties of zirconia ceramics for esthetic dental restorations: A systematic review. *J Prosthet Dent* 2018; 119:36-46.
17. Blatz MB, Phark JH, Ozer F, Mante FK, Saleh N, Bergler M, Sadan A. In vitro comparative bond strength of

- contemporary self-adhesive resin cements to zirconium oxide ceramic with and without air-particle abrasion. *Clin Oral Investig* 2010;14:187-92.
18. Peutzfeldt A, Asmussen E. Adhesive systems: effect on bond strength of incorrect use. *J Adhes Dent* 2002;4:233-42.
  19. Re D, Augusti D, Sailer I, Spreafico D, Cerutti A. The effect of surface treatment on the adhesion of resin cements to Y-TZP. *Eur J Esthet Dent* 2008;3:186-96.
  20. Pekkan G, Saridag S, Pekkan K, Helvacioğlu DY. Comparative radiopacity of conventional and full-contour Y-TZP ceramics. *Dent Mater J* 2016;35:257-63.
  21. Ilie N, Stawarczyk B. Quantification of the amount of blue light passing through monolithic zirconia with respect to thickness and polymerization conditions. *J Prosthet Dent* 2015;113:114-21.
  22. Sulaiman TA, Abdulmajeed AA, Donovan TE, Ritter AV, Lassila LV, Vallittu PK, Närhi TO. Degree of conversion of dual-polymerizing cements light polymerized through monolithic zirconia of different thicknesses and types. *J Prosthet Dent* 2015;114:103-8.
  23. Sen N, Isler S. Microstructural, physical, and optical characterization of high-translucency zirconia ceramics. *J Prosthet Dent* 2020;123:761-8.
  24. Church TD, Jessup JP, Guillory VL, Vandewalle KS. Translucency and strength of high-translucency monolithic zirconium oxide materials. *Gen Dent* 2017;65:48-52.
  25. Manziuc MM, Gasparik C, Negucioiu M, Constantiniuc M, Alexandru B, Vlas I, Ducea D. Optical properties of translucent zirconia: a review of the literature. *Eurobiotech J* 2019;3:45-51.
  26. Spitznagel FA, Boldt J, Gierthmuehlen PC. CAD/CAM ceramic restorative materials for natural teeth. *J Dent Res* 2018;97:1082-91.
  27. Novais VR, Raposo LH, Miranda RR, Lopes CC, Simamoto PCJ, Soares CJ. Degree of conversion and bond strength of resin-cements to feldspathic ceramic using different curing modes. *J Appl Oral Sci* 2017;25:61-8.
  28. Alovisi M, Scotti N, Comba A, Manzon E, Farina E, Pasqualini D, Michelotto Tempesta R, Breschi L, Cadeno M. Influence of polymerization time on properties of dual-curing cements in combination with high translucency monolithic zirconia. *J Prosthodont Res* 2018;62:468-72.
  29. Ferracane JL. Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins. *Dent Mater* 1985;1:11-4.
  30. Hofmann N, Papsthart G, Hugo B, Klaiber B. Comparison of photo-activation versus chemical or dual-curing of resin-based luting cements regarding flexural strength, modulus and surface hardness. *J Oral Rehabil* 2001;28:1022-8.
  31. Reges RV, Moraes RR, Correr AB, Sinhoreti MA, Correr-Sobrinho L, Piva E, Nouer PR. In-depth polymerization of dual-cured resin cement assessed by hardness. *J Biomater Appl* 2008;23:85-96.
  32. De Souza G, Braga RR, Cesar PF, Lopes GC. Correlation between clinical performance and degree of conversion of resin cements: a literature review. *J Appl Oral Sci* 2015;23:358-68.
  33. Yan YL, Kim YK, Kim KH, Kwon TY. Changes in degree of conversion and microhardness of dental resin cements. *Oper Dent* 2010;35:203-10.
  34. Lopes Cde C, Rodrigues RB, Silva AL, Simamoto Júnior PC, Soares CJ, Novais VR. Degree of conversion and mechanical properties of resin cements cured through different all-ceramic systems. *Braz Dent J* 2015;26:484-9.
  35. Camposilvan E, Leone R, Gremillard L, Sorrentino R, Zarone F, Ferrari M, Chevalier J. Aging resistance, mechanical properties and translucency of different yttria-stabilized zirconia ceramics for monolithic dental crown applications. *Dent Mater* 2018;34:879-90.
  36. Barbon FJ, Moraes RR, Calza JV, Perroni AP, Spazzin AO, Boscato N. Inorganic filler content of resin-based luting agents and the color of ceramic veneers. *Braz Oral Res* 2018;32:e49.
  37. Kim KH, Ong JL, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. *J Prosthet Dent* 2002;87:642-9.
  38. Rangrez TA, Mobin R. Polymer composites for dental fillings. In: Asiri AM, Inamuddin, Mohammad A, editors. *Applications of nanocomposite materials in dentistry*. Cambridge, UK; Woodhead Publishing; 2019. p. 205-24.
  39. Ciccone-Nogueira JC, Borsatto MC, de Souza-Zaron WC, Ramos RP, Palma-Dibb RG. Microhardness of composite resins at different depths varying the

- post-irradiation time. *J Appl Oral Sci* 2007;15:305-9.
40. Tarumi H, Imazato S, Ehara A, Kato S, Ebi N, Ebisu S. Post-irradiation polymerization of composites containing bis-GMA and TEGDMA. *Dent Mater* 1999;15:238-42.
  41. Craig RG, Powers JM, Sakaguchi RL. *Craig's restorative dental materials*. 12th ed. St. Louis; Mosby Elsevier; 2006. p. 161-98.
  42. Marghalani HY. Post-irradiation vickers microhardness development of novel resin composites. *J Mater Res* 2010;13:81-7.
  43. Braga RR, Cesar PF, Gonzaga CC. Mechanical properties of resin cements with different activation modes. *J Oral Rehabil* 2002;29:257-62.
  44. Kumbuloglu O, Lassila LV, User A, Vallittu PK. A study of the physical and chemical properties of four resin composite luting cements. *Int J Prosthodont* 2004;17:357-63.
  45. Lu H, Mehmood A, Chow A, Powers JM. Influence of polymerization mode on flexural properties of esthetic resin luting agents. *J Prosthet Dent* 2005;94:549-54.
  46. Guiraldo RD, Consani S, Consani RL, Berger SB, Mendes WB, Sinhoreti MA. Light energy transmission through composite influenced by material shades. *Bull Tokyo Dent Coll* 2009;50:183-90.
  47. Moreno MBP, Costa AR, Rueggeberg FA, Correr AB, Sinhoreti MAC, Ambrosano GMB, Consani S, Correr Sobrinho L. Effect of ceramic interposition and post-activation times on knoop hardness of different shades of resin cement. *Braz Dent J* 2018;29:76-81.
  48. Bandéca MC, El-Mowafy O, Saade EG, Rastelli ANS, Bagnato VS, Porto-Neto ST. Changes on degree of conversion of dual-cure luting light-cured with blue LED. *Laser Phys* 2009;19:1050-5.
  49. Fonseca RG, Cruz CA, Adabo GL. The influence of chemical activation on hardness of dual-curing resin cements. *Braz Oral Res* 2004;18:228-32.