# Microhardness of light- and dual-cured resin cements after light curing through various translucencies of translucent monolithic zirconia

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A Thesis Submitted in Partial Fulfillment of the Requirements
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# ความแข็งจุลภาคของเรซินซีเมนต์ชนิดบ่มตัวด้วยแสงและชนิดบ่มตัวสองรูปแบบหลังการบ่มตัวด้วยแสงผ่านมอนอลิธิคเซอร์โคเนียชนิดโปร่งแสงที่มีความโปร่งแสงแตกต่างกัน

น.ส.สวรรยา เพชรที่วัง

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต
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| Thesis Title | Microhardness of light- and dual-cured resin cements after light curing through various translucencies of translucent monolithic zirconia |
| By | Miss Sawanya Pechteewang |
| Field of Study | Prosthodontics |
| Thesis Advisor | Assistant Professor PRAROM SALIMEE, DDS, PhD. |

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Accepted by the FACULTY OF DENTISTRY, Chulalongkorn University in Partial Fulfillment of the Requirement for the Master of Science

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สวรรยา เพชรที่วัง : ความแข็งจุลภาคของเรซินซีเมนต์ชนิดบ่มตัวด้วยแสงและชนิดบ่มตัวสองรูปแบบหลังการบ่มตัวด้วยแสงผ่านมอนอลิธิคเซอร์โคเนียชนิดโปร่งแสงที่มีความโปร่งแสงแตกต่างกัน. ( Microhardness of light- and dual-cured resin cements after light curing through various translucencies of translucent monolithic zirconia) อ.ที่ปรึกษาหลัก : ผศ. ทญ. ดร.ปรารมภ์ ซาลิมี

*วัตถุประสงค์:* การศึกษานี้มีวัตถุประสงค์เพื่อประเมินความแข็งจุลภาคของเรซินซีเมนต์ชนิดบ่มตัวด้วยแสงและชนิดบ่มตัวสองรูปแบบ หลังการฉายแสงผ่านมอนอลิธิคเซอร์โคเนียที่มีความโปร่งแสงต่างกัน ได้แก่ ชนิดทรานส์ลูเซนต์ (T) ไฮทรานส์ลูเซนต์ (HT) ซุปเปอร์ทรานส์ลูเซนต์ (ST) และเอกซ์ตราทรานส์ลูเซนต์ (XT) *อุปกรณ์และวิธีดำเนินการวิจัย:* ทำการเตรียมชิ้นงานมอนอลิธิคเซอร์โคเนีย เป็นชิ้นสี่เหลี่ยมจตุรัสขนาด 10×10×1 มิลลิเมตร ชนิดละ 1 ชิ้น และใช้เรซินซีเมนต์ 2 ชนิด ได้แก่ Variolink® N LC และ RelyX™ U200 ซึ่งผสมและใส่ลงในช่องว่างของแบบที่มีขนาดเส้นผ่านศูนย์กลาง 5 มิลลิเมตร หนา 2 มิลลิเมตรในแบบที่สร้างขึ้นเอง ทำการกระตุ้นการบ่มตัวของเรซินซีเมนต์ด้วยการฉายแสงผ่านเซอร์โคเนีย เป็นเวลา 20 วินาที โดยใช้กลุ่มที่ฉายแสงผ่านสไลด์แก้วหนา 1 มิลลิเมตรเป็นกลุ่มควบคุม (n=5) นำชิ้นทดสอบทั้งหมด เก็บไว้ในที่แห้ง สภาวะมืด ที่อุณหภูมิ 37 องศาเซลเซียส เป็นเวลา 24 ชั่วโมงก่อนนำไปวัดค่าความแข็งจุลภาคที่ด้านบนของเรซินซีเมนต์ด้วยเครื่องทดสอบความแข็งจุลภาคแบบวิกเกอร์ (Vickers hardness tester) ด้วยแรงกดขนาด 300 กรัม นำผลการทดสอบในแต่ละกลุ่มมาหาค่าเฉลี่ยและส่วนเบี่ยงเบนมาตรฐานและทำการวิเคราะห์สถิติด้วยการวิเคราะห์ความแปรปรวนสองทาง (Two-way ANOVA) ร่วมกับทดสอบความแตกต่างระหว่างค่าเฉลี่ยรายคู่ (post hoc analysis) ที่ระดับความเชื่อมั่นร้อยละ 95 *ผลการศึกษา:* พบว่าความแข็งจุลภาคของเรซินซีเมนต์ทั้งสองชนิดภายใต้เซอร์โคเนียชนิด T มีค่าน้อยกว่าเซอร์โคเนียชนิด XT HT และ ST อย่างมีนัยสำคัญทางสถิติ เรซินซีเมนต์ชนิด RelyX™ U200 ให้ค่าความแข็งจุลภาคสูงกว่า Variolink® N LC ในทุกกลุ่มทดลองเซอร์โคเนีย *สรุปผลการศึกษา:* ความโปร่งแสงของมอนิลิธิคเซอร์โคเนียมีผลต่อความแข็งจุลภาคของเรซินซีเมนต์ทั้งชนิดบ่มตัวด้วยแสงและชนิดบ่มตัวสองรูปแบบ โดยค่าความแข็งจุลภาคของซีเมนต์ภายใต้เซอร์โคเนียชนิดที่มีความใสต่ำสุด ในเซอร์โคเนียชนิด T มีค่าต่ำกว่ากลุ่มอื่นอย่างมีนัยสำคัญ

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ปีการศึกษา 2562 ลายมือชื่อ อ.ที่ปรึกษาหลัก .............................

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KEYWORD: Microhardness, Translucent monolithic zirconia, Resin cement

Sawanya Pechteewang : Microhardness of light- and dual-cured resin cements after light curing through various translucencies of translucent monolithic zirconia. Advisor: Asst. Prof. PRAROM SALIMEE, DDS, PhD.

*Purpose:* This study aims to investigate the microhardness of light- and dual-cured resin cements under various translucencies of monolithic zirconia specimen; translucent (T), super translucent (ST), high translucent (HT), and extra translucent (XT). *Materials and Methods:* Four different translucencies of zirconia specimen were prepared in the square shape, size 10×10×1 mm. The light-cured resin cement (Variolink® N LC) and the dual-cured (RelyX™ U200) resin cement were mixed and loaded in a 2-mm height and 5-mm diameter of space in a customized mold and cured through the zirconia specimens and 1-mm thick glass slide in a control group, using the light curing unit for 20 seconds (n=5). All samples were stored dry in a dark room at 37°C for 24 h. The microhardness test was performed on the top of the cement layer with a Vickers hardness tester with a 300-g load. The results of mean and standard deviation of Vickers hardness number (VHN) were recorded and statistically analyzed with two-way ANOVA and post hoc analysis ($\alpha$=0.05). *Results:* The mean VHN values of both cements were significantly lower when cured under T group compared to the XT, HT, and ST groups. RelyX™ U200 resin cement showed higher VHN value than Variolink® N LC in all groups. *Conclusion:* The VHN of both light- and dual-cured resin cement was influenced by different translucencies of zirconia. For both light- and dual-cured resin cements under the lowest translucency of T zirconia showed significantly lower than the other groups.

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Sawanya Pechteewang

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## Introduction

Zirconia (Zirconium dioxide, $ZrO_2$) ceramic has become more routine material in many all-ceramic dental restorations, due to their increased flexural strength, combined with esthetic and good biocompatibility (1, 2). Zirconia is a polymorphic material that occurs in three different crystal structures, depending on the temperature, monoclinic phase (room temperature to 1170$^{\circ}$C) with brittle and low mechanical property, tetragonal phase (1170$^{\circ}$C – 2370$^{\circ}$C) with higher mechanical property and cubic phase (2370$^{\circ}$C) (3). The volume expansion (3-5%) will occur in the cooling process when the tetragonal phase transforms into the monoclinic phase, leading to high stress and producing crack formation, and a decrease in strength and toughness. According to this condition, pure zirconia could not be used for dental restorative material. In order to maintain the beneficial mechanical properties of the tetragonal phase at room temperature, the stabilization can be done by adding of some oxides such as calcium oxide (CaO), magnesium oxide (MgO), yttrium oxide ($Y_2O_3$), and cerium oxide ($CeO_2$). Among these stabilizers , the solution of 3 mol% yttria (3Y) are widely used for the dental zirconia which is called yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP) (4). When the zirconia was impacted with heat or stress in clinical situations such as grinding, sandblasting, or autoclave, the tetragonal phase might transform into the monoclinic phase and influence the zirconia's mechanical property. This transformation results in a volume expansion of 3-5% and generates a compressive layer. The layer prevents crack propagation, leading to high fracture strength (about 900-1,400 MPa) and toughness (about 5-10 MPa.m$^{1/2}$) of zirconia. This phenomenon is called "transformation toughening" (5). Besides, when the defects formed larger than the compressive layer, this advantage will be lost, creating an increase in tensile stresses, susceptibility to surface degradation, and enhancement in the surface roughness.

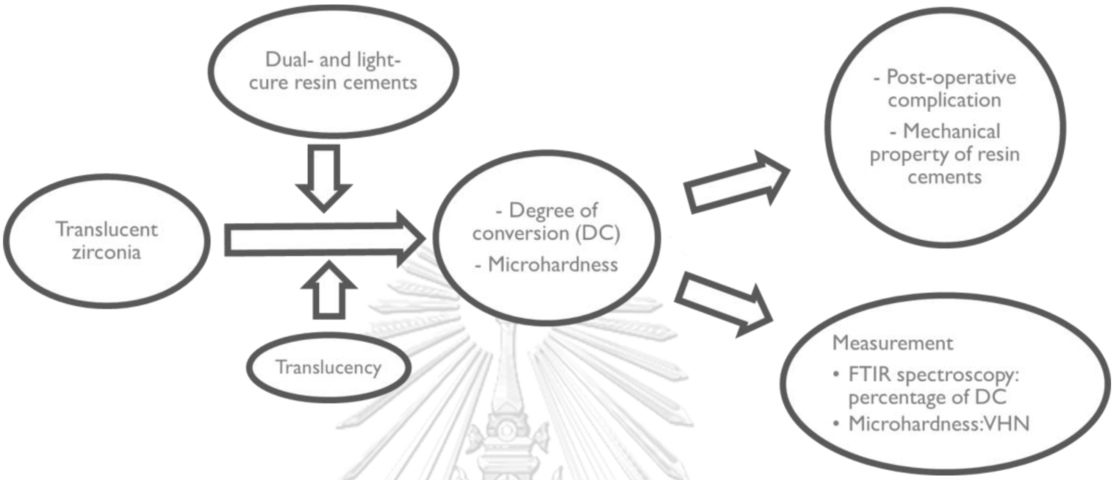
The opacity was the drawback of the first generation of 3Y-TZP containing zirconia. Veneering porcelain technique was introduced to improve the appearance of dental zirconia (1). The most common problem in this veneered zirconia is porcelain chipping (6). This resulted in the development of full-contour restorations of zirconia or *monolithic zirconia* which aimed to overcome the complication of chipping. However, opacity is still the problem which led the early monolithic zirconia was only limited in the posterior teeth (7). To improve the translucency appearance, various techniques can be used such as the decrease in grain size (8, 9), elimination or reduction the concentration of some additives such as alumina (10), addition the content of 0.2% mol lanthanum oxide (11), and increase of the sintering temperature. However, the sintering temperature above 1550°C will compromise the flexural strength and stability of zirconia. Moreover, the porosity occurring during the sintering procedure is the other important factor to handle (12).

Resin cements have been established for the cementation of zirconia restorations (13-15). Since the opacity of conventional zirconia restoration can disturb the light transmission, dual-cured resin cements may be indicated in the clinical applications. Dual-cured resin cements merge the advantageous properties of being cured by both chemical and light. The presence of the self-cured and photo-initiators in the cement allows a sufficient degree of conversion (DC) underneath the restoration. Since translucent monolithic zirconia provide high translucency parameter like lithium disilicate ceramic (16), more variations for esthetic anterior restoration might be clinical applied. The higher translucency of the zirconia should allow more light transmission through the restoration. Therefore, the light-cured resin cements might also be applied for cementation of this translucent zirconia as the advantages such as more color stability, easily excess cement removal, and extended-working time, etc (17). However, the ability of light transmission through this zirconia should be investigated to confirm for the degree of conversion of the light-cured resin cement.

Many previous studies have used the surface hardness of resin cements to test for the degree of conversion of resin cements. Knoop or Vickers hardness tests is supposed to correlate with DC (18) as both methods demand lower cost and can be easily and quickly prepared than the direct measurement with Fourier Transform Infrared Spectrometer (FTIR) technique. Therefore, investigating the hardness of the material is an accepted method for evaluating physical properties and DC (19-23).

The purpose of this study is to investigate the microhardness of light- and dual-cured resin cements after light transmission through various translucencies of translucent monolithic zirconia.

## Conceptual framework



## Research Question

Would the different translucencies of translucent monolithic zirconia have the effect on microhardness of two different light- and dual-cured resin cements?

P: Translucent monolithic zirconia.

I: Different translucencies of translucent monolithic zirconia and different resin cements.

C: Direct light curing through transparent microscope slide.

O: Microhardness of light- and dual-cured resin cements.

## Research Objectives

To evaluate the effects of translucency of monolithic zirconia on the microhardness of two different light- and dual-cured resin cements.

## Research Hypotheses

$H_0$: Monolithic zirconia specimens at different translucencies have no effect on the microhardness of light- and dual-cured resin cements.

$H_a$: Monolithic zirconia specimens at different translucencies have an effect on the microhardness of light- and dual-cured resin cements.

$H_1$: There is no difference of microhardness of light- and dual-cured resin cements after light transmission through the translucent monolithic zirconia.

$H_b$: There is a difference of microhardness of light- and dual-cured resin cements after light transmission through the translucent monolithic zirconia.

**Proposed Benefits**

To provide recommendations for awareness to clinicians for optimal polymerization of light- and dual-cured resin cements in patient who receives translucent monolithic zirconia restoration.

# Literature Review

## Zirconia ceramic

Zirconia (ZrO2) is a crystalline dioxide of zirconium with a proper mechanical property for being dental restorations. Due to their increased flexural strength, good biocompatibility (1, 2), and characteristic esthetic properties (24), the zirconia ceramics become more routine restoration material in many dental works.

There are three crystalline phases of zirconia: monoclinic (M), tetragonal (T), and cubic (C) phases. Monoclinic phase is stable at room temperature when in form of pure zirconia. Monoclinic phase transforms to tetragonal phase when temperature rising above 1170°C, then transforms to cubic phase at temperature above 2370°C. In dentistry, 3 mol% yttria (3Y) are used to be the stabilizer of the tetragonal phase at room temperature. This stabilizer induces tetragonal phase transforms to monoclinic phase with 3-5% volume expansion of the grains, which prevent the crack within 3Y-TZP containing zirconia. This phenomenon is called “transformation toughening” , leading high fracture strength (about 900-1,400 MPa) and toughness (about 5-10 $MPa.m^{1/2}$) of zirconia.

However, the opacity was the drawback of the first generation of 3Y-TZP containing zirconia. Veneering porcelain technique was introduced to improve the appearance of dental zirconia (1). However, the most common problems in this veneered zirconia is porcelain chipping, suspected that causing by the mismatch of coefficient of thermal expansion (CTE) between zirconia substructure and veneering porcelain(1). Moreover, there are many contributing factors has been reported leading this chipping complications; porcelain shrinkage in sintering process, porosities, insufficiency substructure design, and weak bonding between veneering porcelain and zirconia substructure (1). There are many recommended methods to decrease the veneering chipping problems such as using CAD-CAM technique for veneering porcelain to core zirconia substructure, adaptation of firing processes, and developing of full-contour restorations of zirconia material which is called monolithic zirconia or a full contoured structure of Y-TZP.

## Development generations of dental zirconia ceramic

Alumina, one of the factors that causes *the first generation*'s opacity, is added during the sintering process yielding reducing porosity within zirconia. Moreover, the addition of alumina into 3Y-TZP containing zirconia promotes stabilization of tetragonal zirconia by separation into grain boundaries. Because of the differences of indices of refraction between alumina and zirconia, light transmission is compromised. *The second generation* of 3Y-TZP containing zirconia is more translucent than the first generation, resulting from the lower proportion of alumina (0.25 wt% to 0.05 wt%).

Presently, increased yttria has been added in dental zirconia fabrication leading higher amount of cubic phase. *The third generation* of zirconium ceramic is 5Y-PSZ, which contains 5 mol% yttria, yielding the 50% cubic phase in dental zirconia was stabilized. The advantage of 5Y-PSZ is more translucent than the early generations since the isotropic property in various crystallographic directions of cubic phase induces light transmission at grain boundaries. Nevertheless, transformation toughening is not occurred in 5Y-PSZ, in the consequence of stabilized cubic phase does not reconstruct at room temperature, resulted in reduction of flexural strength and toughness. *The newest generation* is 8 mol% yttria containing zirconia or 8Y-CZ, which can fully stabilize the cubic phase. To be concluded, as increased of yttria content, the more translucent of zirconia is occurred. On the other hand, the more presence of cubic phase influences compromised in zirconia mechanical properties due to the absence of transformation toughening (25).

| Yttia containing zirconia | Percentage of yttria | Material brand name | Manufacturer | Marketing name | Advantage | Disadvantage |
|---|---|---|---|---|---|---|
| **3Y-TZP** | 3 mol% | *1st generation:* | | Translucent zirconia | Transformation toughening enhances zirconia to resist micro-cracking | Opaque appearance |
| | | Lava Frame | 3M ESPE | | | |
| | | Prettau Zirconia | Zirconzhan | | | |
| | | KaVo Everest | KaVo Dental | | | |
| | | ZH | Vita Zahnfabrik | | | |
| | | Vita YZ T | Wieland Dental | | | |
| | | Zenostar MO | | | | |
| | | | | High-translucent zironia | | |
| | | *2nd generation:* | 3M ESPE | | | |
| | | Lava Plus | Dentsply Sirona | | | |
| | | Cercon HT | Vita Zahnfabrik | | | |
| | | Vita YZ HT | Glidewell | | | |
| | | Bruxir Full- | Tosoh | | | |
| | | Strength | Wieland Dental | | | |
| | | Zpex | Heany | | | |
| | | Zenostar T | Kuraray Noritake | | | |
| | | Luxisse Dental | Dentsply Sirona | | | |
| | | Katana HT | | | | |
| | | inCoris TZI | | | | |

| **Yttia containing zirconia** | Percentage of yttria | Material brand name | Manufacturer | Marketing name | Advantage | Disadvantage |
|---|---|---|---|---|---|---|
| **4-5Y-PSZ** | 4 - 5 mol% | Zpex 4 | Tosoh | Super-/Extra-/Ultra-translucent zirconia/ Partially stabilized zirconia (PSZ) | More translucency | May increase the LTD phenomenon, which reduce the zirconia strength |
| | | Zenostar MT | Wieland Dental | | | |
| | | Katana ST | Kuraray Noritake | | | |
| | | Lava Esthetic | 3M ESPE | | | |
| | | Vita YZ ST | Vita Zahnfabrik | | | |
| | | Vita YZ XT | Vita Zahnfabrik | | | |
| | | Cercon XT | Dentsply Sirona | | | |
| | | BruxZir Anterior | Glidewell | | | |
| | | Prettau Zirconia | Zirconzhan | | | |
| | | Katana UT | Kuraray Noritake | | | |
| | | Zpex Smile | Tosoh | | | |
| | | Luxisse + | Heany | | | |
| **8Y-CZ** | 8 mol% | Prettau Anterior | Zirconzhan | Fully stabilized zirconia (FSZ)/Cubic zirconia (CZ) | More translucency | Absence of transformation toughening induce minimizing of material strength |

**Table 1: Demonstration of types of yttria containing-zirconia.**

## Microstructure modification and characteristic of translucent monolithic zirconia

Translucent monolithic zirconia is used for the reason that considering more natural appearance tooth restoration. Both intrinsic and extrinsic factors have been reported affecting the optical properties of monolithic zirconia due to light transmission, absorption, refraction, and scattering(26). According to the differences of the refractive index (RI) between grain particles and matrix, or the unlike polymorphs of dental zirconia, as well as porosities, the light transmission thorough the zirconia material is less than internal light scattering resulting to opacity of zirconia (12, 16, 26, 27). Regarding internal light scattering, involves reflection and refraction (27), if light reflection increases, translucency of zirconia decreases (28).

To improve this appearance complication, there are several techniques which solve the refractive index mismatch: decreasing in grain size (8, 9), elimination or decreasing the concentration of some additives such as alumina (10), addition the content of 0.2% mol lanthanum oxide (11), and increasing the sintering temperature. However, at the sintering temperature above 1550°C will compromise the flexural strength and stability of zirconia. Although the reduction of oxygen vacancy and porosity is the other importance factor to handle during sintering procedure (12).

The most popular technique to reach more translucency of dental zirconia is enlargement of yttria content. Regarding translucency and Y-TZP crystalline phase (monoclinic, tetragonal, cubic), the more presence of cubic phase influences the increasing of translucency, along with the decreasing of tetragonal phase due to blocking light scattering from the grain boundaries by cubic phase (10). An addition of yttria content promotes the raising amount of cubic phase and generates more translucency (12). On the other hand, the greater cubic phase reduces mechanical properties of zirconia since the absence of transformation toughening (25).

However, translucency of monolithic zirconia is decreased by increasing its thickness (16, 26, 27). Moreover, there is a recommendation from the studies to match zirconia thickness and mean grain size, gaining translucency close dental feldspathic porcelain. For thickness of 1.3 mm, 82 nm grain size would be chosen. For thickness of 1.5 mm and 2 mm, 77 nm and 70 nm grain size are advised to increase translucency of restoration, respectively (10, 27). The recommended thickness for fixed restorations based on the studies is shown in table 2

| Type of dental ceramic | Axial clearance | Occlusal thickness |
|---|---|---|
| Conventional ceramic crown(29) | 1.5 mm | 2 mm |
| Conventional lithium disilicate bridge connector(30) | 16 mm$^2$ | |
| Translucent zirconia (in vitro)(31) | 0.5 mm | 0.5 mm |
| Implant supported restoration(32)<br>Opposing tooth is fixed implant supported restoration(32)<br>- Bridge connector(30) | 0.7 mm<br>0.8 mm<br>9 mm$^2$ | |

**Table 2: Demonstration of abutment preparation requirement for all ceramic restorations.**

## Resin cements and degree of conversion measurement

Since translucent zirconia's thickness can disturb the light transmission and polymerization of light-cured resin cements (33), dual-cured resin cements may be indicated in the clinical applications. Dual-cured resin cements merge the advantageous properties of being cured by chemical and light, by present of the self-cured and photo-initiators in the cement (34), allowing a sufficient degree of monomer conversion underneath the restoration. With dual polymerization, the portion of monomer resin that initially encountered insufficient irradiance pursues to polymerize after light exposure by means of a delayed chemical curing(35). However, post-operative complications such as early debonding of restoration, sensitivity, microleakage, and recurrent caries can be the outcomes of insufficient hardening of dual-cured resin cements, if the compensation of chemical polymerization is insufficient to reduce light polymerization caused by compromised light irradiance(36).

In clinical situation, polymerization efficiency is the important factor to determine the suitable resin cements. Adequate polymerization leads proper physical properties and a pleasant clinical performance of resin cements. The efficiency of polymerization is expressed by the percentage of reacted carbon groups (-C=C-) after polymerization and referred to "the degree of conversion". According to textbook of Phillips' science of dental material established the definition of degree of conversion as the percentage of carbon-carbon double bond (-C=C-) converted to single bond (-C--C-) during curing to form a polymeric resin (37). A low DC causes the problem of inadequate polymerization, a higher residual quantity of double bonds, the lower crucial physical properties of resin cements.

DC of resin cements can be measured by direct and indirect techniques.(38) Several equipment are available for this purpose which can determine the number of carbon-carbon double bonds (C=C) present in resin matrix qualitatively and quantitatively for example nuclear magnetic resonance (NMR), high performance liquid chromatography (HPLC), gel permeation chromatography (GPC), multiple internal reflection (MIR), infrared spectroscopy IR and FTIR spectroscopy (39-41).

Fourier Transform Infrared Spectroscopy (FTIR) is one of the most popular and accurate technique, which can analyze the available carbon-carbon double bond of resins directly by obtaining a spectrum from the 450-4400 $cm^{-1}$ range (42). Recently, the development of FTIR has improved, able to measure accurately, also can be analyzed in a wide range of spectrum. Even though this technique is very precise and can be used directly, it still has the disadvantages which takes a long-time processing and requires high cost tools (43).

### Microhardness of the material: method for evaluating physical properties and degree of conversion

Surface hardness of resin cements has been supposed to be a strongly correlation with DC (18). Most previous studies have used the Knoop or Vickers hardness tests. Both methods can be done easily, quickly and take the lower cost than direct measurement with FTIR technique. Therefore, investigating the hardness of the material is an accepted method for evaluating physical properties and DC (22).

## Materials and methods

### Materials

1. Translucent $ZrO_2$ (VITA YZ T; VITA Zahnfabrik H. Rauter GmbH & Co.; shade A3)
2. High Translucent $ZrO_2$ (VITA YZ HT; VITA Zahnfabrik H. Rauter GmbH & Co.; shade A3)
3. Super Translucent $ZrO_2$ (VITA YZ ST; VITA Zahnfabrik H. Rauter GmbH & Co.; shade A3)
4. Extra Translucent $ZrO_2$ (VITA YZ XT; VITA Zahnfabrik H. Rauter GmbH & Co.; shade A3)
5. Light-cured resin cement (Variolink® N LC cement; Ivoclar Vivadent, Schaan, Liechtenstein)
6. Dual-cured resin cement (RelyX™ U200 Self-Adhesive Resin cement; 3M-ESPE St. Paul, MN, USA)

### Equipment

1. Plastic forceps
2. Plastic cement spatula
3. Digital vernier caliper (Mitutoyo series 500, Japan)
4. PVC mold
5. Gypsum Type IV (Vel-Mix™ Die Stone, Kerr Dental, USA)
6. Glass slide (1 mm and 0.15 mm in thickness)
7. Light curing unit (EliparTrilight$^{TM}$ S10, 3M-ESPE St. Paul, MN, USA)
8. Light curing cabinet (Labolight: LB-III, USA)
9. Ultrasonic bath (VGT-1990, QTD, China)
10. Microhardness tester (Future-Tech: FM-810, Japan)

### Methods

- **Sample size calculation**

According to previous study of Turkoglu and Sen(44) about Vickers hardness of the resin cement light polymerized through monolithic translucent zirconia as a reference, The calculation for number of specimens using formula as follow, $\mu_1$=65.26, $\mu_2$=71.22, $\sigma_1$=2.55, $\sigma_2$=3.28, $\alpha$=0.05, and ß=0.20 sample size for each group n=4. Then adjust the sample size for each group to n=5 per group (Figure 1).

$$n_1 = \frac{\left(z_{1-\frac{\alpha}{2}} + z_{1-\beta}\right)^2 \left[\sigma_1^2 + \frac{\sigma_2^2}{r}\right]}{\Delta^2}$$

$$r = \frac{n_2}{n_1}, \ \Delta = \mu_1 - \mu_2$$

Figure 1 Sample size calculation formula modified from Bernard, R. (2000).

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- **Monolithic zirconia preparation**

Four translucencies of translucent monolithic zirconia were used: Translucent (T), High Translucent (HT), Super Translucent (ST), and Extra Translucent (XT) (Table 3). Each zirconia was prepared in the laboratory using CAD-CAM software. The sintering procedure was processed according to the manufacturer's instructions. After cooling, each specimen was polished by a single operator with polishing sets (VITA SUPRINITY® Polishing Set; VITA Zahnfabrik H. Rauter GmbH & Co. KG), following the manufacturer's instructions. Each translucency was prepared for the final size of 10×10 ± 0.05 mm and thickness of 1 ± 0.05 mm. All measurement was done by using a digital Vernier caliper (Mitutoyo series 500, Japan). Before testing, the specimen was cleaned in an ultrasonic cleanser (ultrasonic bath, VGT-1990, QTD, China) with distilled water for 10 minutes and dried with absorbent paper. The specimens were measured for opacity percentage by using a spectrophotometer (Ultrascan PRO 74-SD-03-10; HunterLab, USA) to calibrate for proper translucency of each product. The translucency percentage of T, HT, ST, and XT was 11.3, 18.4, 19.7, and 22.9 respectively, calculating from 100 subtraction with the opacity percentage value.

Table 3 Material details and physical properties of translucent zirconia blank used in this study(45)

| Material | Manufacturer | Type | Composition (wt%); | Process | Flexural strength (MPa) | Young's modulus (GPa) | Fracture toughness (MPa m$^{-0.5}$) | CTE |
|---|---|---|---|---|---|---|---|---|
| VITA YZ T | VITA Zahnfabrik H. Rauter GmbH & Co. KG | Translucent $ZrO_2$ | $ZrO_2$ 90.4 - 94.5<br>$Y_2O_3$ 4 - 6<br>$HfO_2$ 1.5 - 2.5<br>$Al_2O_3$ 0 - 0.3<br>$Fe_2O_3$ 0 - 0.3 | Soft milling; Dry milling | 1,200 | 210 | 4.5 | 10.5 (20-500°C) $10^{-6}$/K |
| VITA YZ HT | VITA Zahnfabrik H. Rauter GmbH & Co. KG | High translucent $ZrO_2$ | $ZrO_2$ 90.4 - 94.5<br>$Y_2O_3$ 4 - 6<br>$HfO_2$ 1.5 - 2.5<br>$Al_2O_3$ 0 - 0.3<br>$Er_2O_3$ 0 - 0.5<br>$Fe_2O_3$ 0 - 0.3 | Soft milling; Dry milling | 1,200 | 210 | 4.5 | 10.5 (20-500°C) $10^{-6}$/K |
| VITA YZ ST | VITA Zahnfabrik H. Rauter GmbH & Co. KG | Super translucent $ZrO_2$ | $ZrO_2$ 88.4 – 92.5<br>$Y_2O_3$ 6 – 8<br>$HfO_2$ 1.5-2.5<br>Other oxides≤1 | Soft milling; Dry milling | >850 | 210 | 3.5 | 10.3 (20-500°C) $10^{-6}$/K |
| VITA YZ XT | VITA Zahnfabrik H. Rauter GmbH & Co. KG | Extra translucent $ZrO_2$ | $ZrO_2$ 86.4 - 90.5<br>$Y_2O_3$ 8 – 10<br>$HfO_2$ 1.5-2.5<br>Other oxides≤1 | Soft milling; Dry milling | >600 | 210 | 2.5 | 10 (20-500°C) $10^{-6}$/K |

**Table 4 Resin cements used in this study.**

| Material | Manufacturer | Type | Shade | Composition |
|---|---|---|---|---|
| Variolink® N LC Cement | Ivoclar Vivadent, Schaan, Liechtenstein | Light-cured resin cement | Transparent (Base paste only) | Barium glass filler and mixed oxide (48.4%), dimethacrylates (26.3%), ytterbium trifluoride (25.0%), initiators and stabilizers (0.3%), pigments (<0.1%) |
| RelyX™ U200 | 3M-ESPE St. Paul, MN, USA | Dual-cured Resin cement | Translucent | Base: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives<br>Catalyst: methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, rheological additives, pigments |

LC: Light Curing

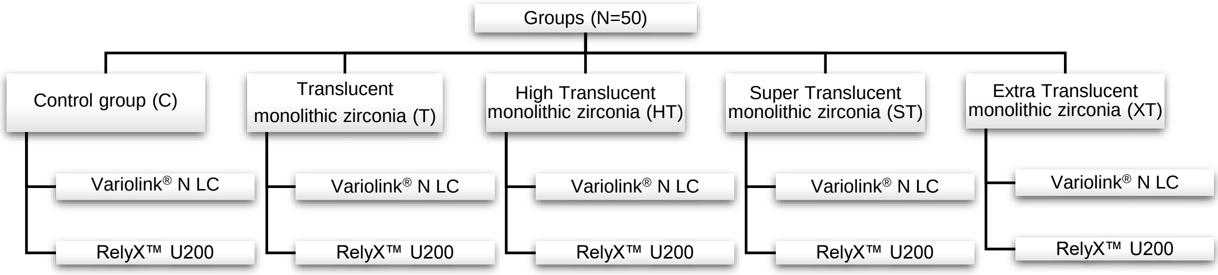

Figure 2 Diagram showing group division in this study.

- **Resin cements sample preparation**

The light-cured resin cement (Variolink® N LC) and the dual-cured resin cement (RelyX™ U200) were used in this study (Table 2). Each resin cement was tested with four translucencies of zirconia and a control group with glass slide (1-mm thick) resulted in eight experimental groups and two control groups (n=5) of total 50 testing specimens (Figure 2). The resin cement was prepared by mixing the cement followed the manufacturers recommendation and loading into a space (2 mm in height and 5 mm in diameter), in a customized mold made of gypsum type IV in a PVC block (Figure 3). After loading, a transparent glass slide (0.15 mm in thickness) was placed over the mold to avoid the inhibition of polymerization by oxygen. The tip of the light curing unit (EliparTrilight™ S10, 3M-ESPE St. Paul, MN, USA) was placed over the 1-mm thick glass slide in the control groups, while in the experimental groups, the tip was placed over the zirconia specimen. The light curing procedure were done with a wavelength of 430-480 nm and a power density of 800 mW/cm$^2$ for 20 s in a light curing cabinet (Labolight: LB-III, USA) (Figure 4). The curing device was calibrated to check for the power of light density before each curing. Subsequently, the specimens were stored dry in dark at 37°C for 24 h.

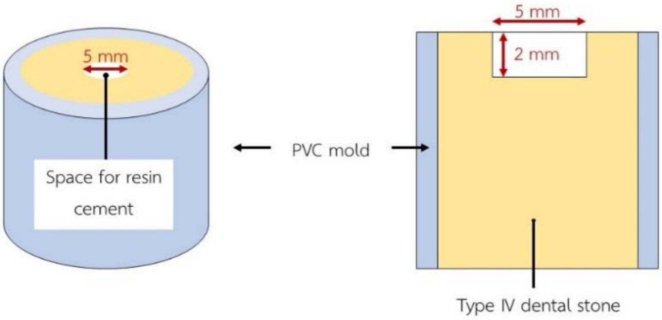

Figure 3 illustration of a customized mold for resin cements sample.

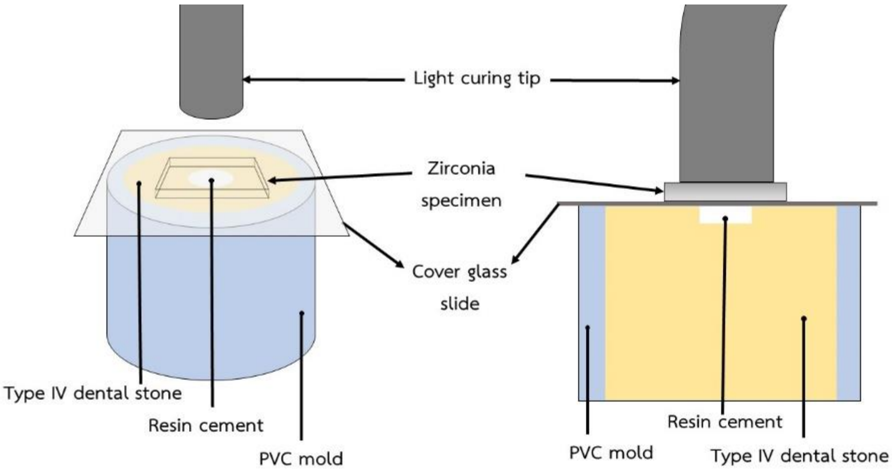

Figure 4 illustration of method of resin cements sample preparation

- **Vickers hardness testing**

The specimen was placed on the platform of a Vickers microhardness tester (Future-Tech: FM-810, Japan) by the resin cement surface was positioned to face the diamond indenter. The Vickers hardness number (VHN) is calculated from the software of the microhardness tester on the basis of surface area of the indentation by using the following formula:

$$\text{VHN (kgf/mm}^2\text{)} = 1.854(F/D^2);$$

whereas F is the applied load (kgf) and $D^2$ is the area of the indentation ($mm^2$). The mean VHN was obtained from 5 measurement of each sample in a vertical pattern. The interface of the resin cement and the gypsum surface of a customized mold was marked as a zero point. The first indentation was 1 mm apart from the zero point, followed by the other 4 points at 0.75 mm apart from the other by setting the software of the tester (Figure 4). Each indentation was tested with 300 g of load for 15 second.

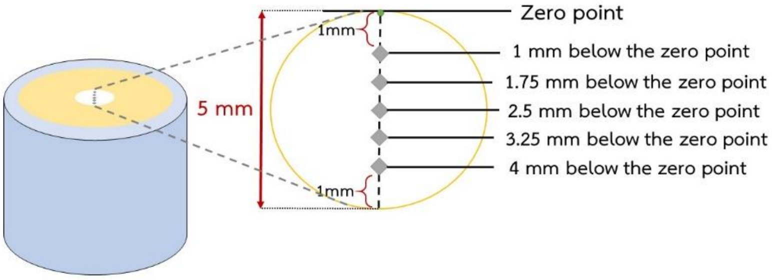

Figure 4 Schematic showing the determination of hardness indentation points on resin cement in a customized mold.

- **Statistics analysis**

The data was analyzed by IBM SPSS Statistic for windows version 22.0 software (SPSS Statistics, IBM Corporation, Chicago, IL, USA). Two-way ANOVA was performed to determine the effect of main factors including translucency of zirconia and type of resin cement and their interactions on the VHN. One-way ANOVA with Tukey post hoc analysis, Welch ANOVA with Games Howell post hoc analysis and independent t-test were used to analyze the difference among groups. All *p*-value < 0.05 were considered statistically significant.

## Results

The results of two-way ANOVA on VHN of the resin cements were shown in Table 5. The VHN was affected by all independent variables (resin cement and translucency) and two-way interactions of all parameters were statistically significant.

**Table 5 Results of Two-way ANOVA on the VHN**

| Source | Type III Sum of Squares | *df* | Mean squares | *F* | *p* |
|---|---|---|---|---|---|
| Intercept | 134126.778 | 1 | 134126.778 | 632231.231* | <0.05 |
| Translucency | 222.385 | 4 | 55.596 | 26.210* | <0.05 |
| Cement | 1417.291 | 1 | 1417.291 | 668.152* | <0.05 |
| Cement*Translucency | 27.585 | 4 | 6.896 | 3.251* | <0.05 |

**Table 6 Results of all measurements of Variolink® N LC and RelyX™ U200**

| Translucency | Translucency percentage | VHN (kgf/$mm^2$) | |
|---|---|---|---|
| | | Variolink® N LC Mean (S.D.) | RelyX™ U200 Mean (S.D.) |
| T | 11.3 | 44.03 (0.59) $^{a, A}$ | 53.38 (1.12) $^{a, B}$ |
| HT | 18.4 | 46.05 (0.88) $^{b, A}$ | 55.95 (1.88) $^{b, B}$ |
| ST | 19.7 | 47.52 (1.12) $^{b, A}$ | 56.39 (1.10) $^{b, B}$ |
| XT | 22.9 | 47.60 (0.64) $^{b, A}$ | 58.01 (1.04) $^{b, B}$ |
| C | 100 | 48.32 (1.87) $^{b, A}$ | 61.86 (0.77) $^{c, B}$ |

Note: The same lower-case letters indicate no significant differences from Welch ANOVA (for Variolink® N LC) and One-way ANOVA (for RelyX™ U200) in column ($p$>0.05). The same upper-case letters indicate no significant differences form independent t-test in row ($p$>0.05).

S.D.: Standard deviation.

T: Translucent; HT: High Translucent; ST: Super Translucent; XT: Extra Translucent, C: Control.

The mean and standard deviation of VHN for Variolink® N LC and RelyX™ U200 were shown in Table 6. For both resin cements Variolink® N LC, the VHN increased by order of translucency as followed: XT $\geq$ ST $\geq$ HT > T. The T group showed significantly lower VHN than those of the all other groups. ($p$<0.05) (Table 6). The VHN value of RelyX™ U200 were significantly higher than those of Variolink® N LC with the same translucency ($p$<0.05) (Table 6) (Figure 5).

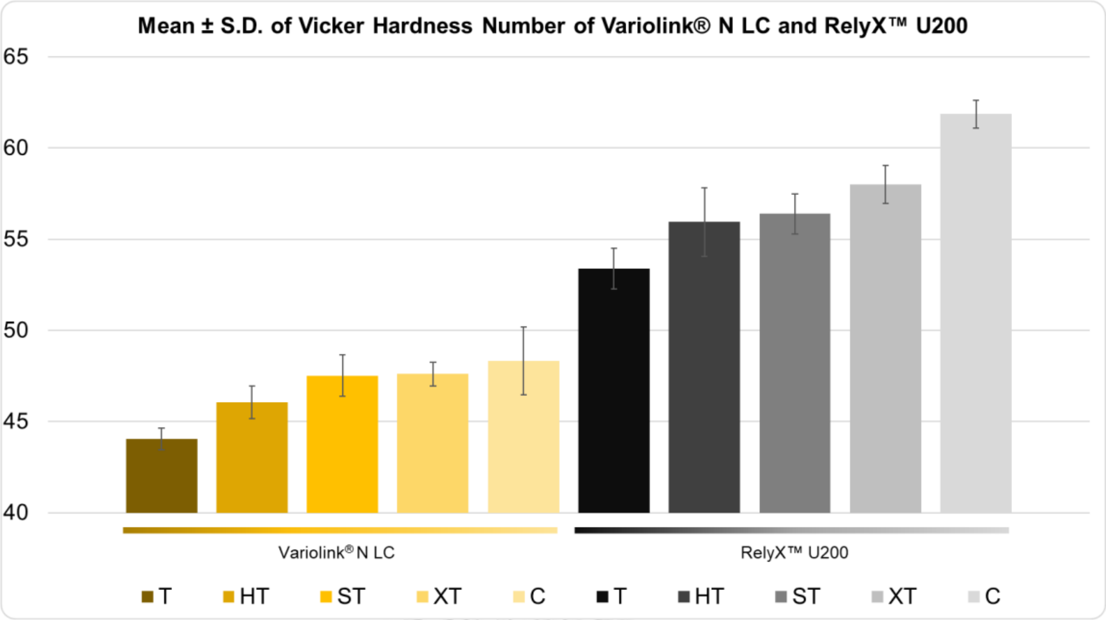

Figure 5 Mean ± S.D. of VHN of Variolink® N LC and RelyX™ U200

(T: Translucent; HT: High Translucent; ST: Super Translucent; XT: Extra Translucent, C: Control)

Table 7 Percentage of decreased VHN in the experimental groups compared to the control groups.

| Zirconia | Translucency percentage | $\Delta$ Mean VHN (kgf/mm$^2$) | | Percentage of VHN (%) | |
|---|---|---|---|---|---|
| | | Variolink® N LC | RelyX™ U200 | Variolink® N LC | RelyX™ U200 |
| T | 11.3 | 4.29 | 4.63 | 91.12 | 86.29 |
| HT | 18.4 | 2.27 | 5.91 | 95.30 | 90.45 |
| ST | 19.7 | 0.80 | 5.47 | 98.34 | 91.16 |
| XT | 22.9 | 0.72 | 3.85 | 98.51 | 93.78 |
| C | 100 | 0 | 0 | 100 | 100 |

To compare the VHN value with the control group of each resin cement, $\Delta$mean VHN and VHN percentage of Variolink® N LC and RelyX™ U200 were calculated and shown in Table 7.

For Variolink® N LC, the T group showed significantly lower VHN than the control group ($p<0.05$), whereas the other groups (HT, ST, and XT) showed no significant difference in VHN with the control group. ($p>0.05$) (Table 6). The VHN were 91.12%, 95.30%, 98.34%, and 98.51% for T, HT, ST, and XT respectively when comparing to the control group (Table 7).

For RelyX™ U200, all the experimental groups (T, HT, ST, and XT) showed significantly lower VHN than the control group ($p<0.05$) (Table 6). The VHN were 86.29%, 90.45%, 91.16%, and 93.78% for T, HT, ST, and XT respectively when comparing to the control group. The ΔMean VHN of these groups were higher than those of Variolink® N LC (Table 7).

## Discussion

Surface hardness of resin cement has been supposed to correlate with its degree of conversion (19). Therefore, microhardness test is used in many studies as a reliable indirect method to evaluate the degree of conversion of resin-based luting cements (19, 20, 23). When the cross-link polymer increased, the conversion degree and relatedly the higher hardness of the resin material will be shown (20). In this present study, the light-cured (Variolink® N LC) and the dual-cured (RelyX™ U200) resin cements were used to determine the effect of translucent monolithic zirconia's translucency on the VHN of resin cements.

According to the effect of various translucencies of zirconia on VHN of the resin cements, the low-translucency monolithic zirconia (T) provided lower VHN than the other groups (XT, ST, and HT) in both resin cement; however there was no significant difference among the XT, ST, and HT groups. Therefore, the first null hypothesis was rejected.

Previously, it was reported that different brands of yttria-stabilized monolithic zirconia exhibited different translucency properties (26). According to the same study, monolithic zirconia with the higher amounts of yttria (<12 %wt) was reported to have higher translucency parameter values than the other tested groups with lower yttria (4-6 %wt). The results of this study showed that the VHN of resin cements were higher when cured under monolithic zirconia with the increased yttria (8-10 %wt in XT, and 6-8 %wt in ST) compared to those of monolithic zirconia with lower yttria (4-6 %wt in T). The translucency of monolithic zirconia is associated with its microstructural characterization and chemical composition (46). Sen and Isler (47) investigated the crystalline phases of VITA YZ HT, ST, and XT which also used in this study and found that after sintering, XRD analysis demonstrated that the increase in $Y_2O_3$ content leading to the higher amounts of cubic phase in zirconia. Consequently, the rising amount of cubic phase generates more translucency in the yttria-stabilized zirconia (12). Thus, it can be stated that the higher cubic phase in XT, ST, and HT groups leaded to an increased translucency, consequently enhancing the polymerization efficacy of resin cements below.

According to the manufacturer's information, T and HT contained the same amounts of yttria content (4-6 %wt). However, the results of VHN of both resin cements in this study, was significantly different between HT and T group. This result may arise from the different microstructural characterization and chemical composition of the monolithic zirconia. Nakamura et al. (48) reported that the addition of 0.5 %wt erbium oxide ($Er_2O_3$) to yttria-stabilized zirconia showed a significant increase in the cubic phase. Consisting of $Er_2O_3$ (<0.5 %wt) in HT was seemed to be an influential factor for the rising of the cubic phase.

RelyX™ U200 which is a dual-cured resin cement showed higher VHN than Variolink® N LC which was polymerized by light-cured alone, therefore, the second null hypothesis was rejected. This was in agreement with a previous study of Hofmann et al. (21) which reported that light-cured resin cements showed lower VHN than dual-cured resin cements (Variolink® II, Cerec Vita DuoCement, SonoCem, Nexus, and Panavia 21) when cured under leucite-reinforced glass-ceramic specimens. According to Alovisi et al. (19), the different brands of cement with the same curing mode (RelyX™ Ultimate and Panavia SA) also influenced the VHN. However, they also stated that the absolute VHN value should not be used to compare the DC between the different resin cements. Note that the results of this study showed a significant difference of VHN between the two different cements tested, which may depend on variation in filler particles and initiators (21, 49). The RelyX™ U200 resin cement (72%wt filler) contains a similar proportion of filler to Variolink® N LC cement (73.4%wt filler) (50, 51). According to the manufacturer’s information, RelyX™ U200 resin cement (12.5 µm) had the bigger filler size than Variolink® N LC (0.04-3 µm). This was in agreement with the study of Kundie et al. (52) who reported that the higher filler size exhibited the increased surface hardness of the resin-based materials. Accordingly, it might be the reason that the RelyX™ U200 group showed a higher VHN value than the Variolink® N LC group. However, according to the results of this study, Variolink® N LC cured under HT, ST and XT zirconia specimens showed no significant different VHN with the control group which cured under a transparent glass. In contrast, all experimental groups of RelyX™ U200 showed a significant difference in the VHN value with their control group. Thus HT, ST, and XT groups may provide the sufficient polymerization of Variolink® N LC resin cements underneath, whereas RelyX™ U200 might not. This might be explained by the delay procedure of the chemical curing process in the dual-cure resin cement, while in the light-cured resin cement the specimen in the translucency percentage above 18.4 (HT, ST, and XT) could be effective by light curing procedure.

Reges et al. (53) reported that the shade of the cement also affected its microhardness. The darker shade of resin provided a decrease in microhardness in case of direct light exposure (54). Transparent shade of resin cement exhibited more light absorption than the opaque shade, providing increasing curing depth and microhardness value (55). In this study, the transparent shade of Variolink® N LC and the translucent shade of RelyX™ U200 were used to minimize the impact of shade of cement on the VHN and focus on the effect of zirconia’s translucency on the polymerization of the resin cements. However, in clinical situations, clinicians should mind that the tested resin cements may have lower hardness value when the darker shade is chosen.

In this present study, the condition of 24-hour storage was determined according to the reported of Yan et al. (56) that the polymerization reaction and VHN value of resin cement were significantly increased at 24 h, but were not significantly increased thereafter. However, other studies reported the significantly increased degree of conversion and VHN value according to the storage time (after 7 days) (57-59). Thus, further study of a longer period of curing of resin cement and in the oral environment are needed.

In this study, a low translucent monolithic zirconia (T) exhibited the lowest VHN value in both resin cements. The decrease in translucency of monolithic zirconia tends to result in the reduction of the VHN values of resin cements. Clinicians should consider that a decrease in the translucency of monolithic zirconia restorations might result in reducing a light penetration, which could affect the polymerization efficacy of the resin cement underneath and long-term durability of the restoration.

## Conclusions

Within the limitations of this study, it can be concluded that:

1. Dual-cured RelyX™ U200 had higher VHN value than light-cured Variolink® N LC after light transmission through the all different translucencies of monolithic zirconia.
2. The VHN of both RelyX™ U200 by dual-cured mode and Variolink® N LC by light- cured mode were influenced by different translucencies of monolithic zirconia.
3. For both resin cements, under the lowest translucency of T zirconia showed significantly lower VHN than the HT, ST and XT groups.

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# VITA

| | |
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# Appendix

Table 8 Raw data of VHN of RelyX™ U200 groups

| | T | HT | ST | XT | C |
|---|---|---|---|---|---|
| | 53.778 | 56.354 | 57.352 | 58.454 | 61.102 |
| | 51.736 | 56.756 | 55.622 | 56.750 | 62.608 |
| | 54.570 | 54.970 | 54.932 | 57.072 | 61.640 |
| | 54.002 | 58.316 | 57.456 | 58.658 | 61.216 |
| | 52.814 | 53.348 | 56.580 | 59.112 | 62.734 |
| Mean ± S.D. | **53.38 ± 1.12** | **55.95 ± 1.88** | **56.39 ± 1.10** | **58.01 ± 1.04** | **61.86 ± 0.77** |

Table 9 Raw data of VHN of Variolink® N LC groups

| | T | HT | ST | XT | C |
|---|---|---|---|---|---|
| | 44.604 | 46.154 | 46.352 | 46.682 | 49.064 |
| | 43.486 | 44.544 | 47.992 | 47.174 | 48.416 |
| | 44.714 | 46.216 | 48.894 | 47.996 | 51.002 |
| | 43.866 | 46.552 | 47.992 | 48.11 | 46.602 |
| | 43.498 | 46.776 | 46.353 | 48.062 | 46.516 |
| Mean ± S.D. | **44.03 ± 0.59** | **46.05 ± 0.88** | **47.52 ± 1.12** | **47.60 ± 0.64** | **48.32 ± 1.87** |

Table 10 Test of Normality

| Type | | Shapiro-Wilk | | |
|---|---|---|---|---|
| | | Statistic | df | Sig. |
| VHN | T-VL | .836 | 5 | .154 |
| | HT-VL | .809 | 5 | .095 |
| | ST-VL | .859 | 5 | .226 |
| | XT-VL | .818 | 5 | .113 |
| | C-VL | .916 | 5 | .503 |
| | T-RU | .945 | 5 | .701 |
| | HT-RU | .985 | 5 | .961 |
| | ST-RU | .912 | 5 | .479 |
| | XT-RU | .887 | 5 | .342 |
| | C-RU | .855 | 5 | .210 |

VL: Variolink® N LC

RU: RelyX™ U200

**Independent Factor: Translucency of zirconia**

**Table 11 Test of Homogeneity of Variance of Variolink® N LC groups**

| Levene Statistic | df1 | df2 | Sig. |
|---|---|---|---|
| 3.918 | 4 | 20 | 0.017 |

**Table 12 ANOVA of Variolink® N LC groups**

| | Sum of Squares | df | Mean Square | F | Sig. |
|---|---|---|---|---|---|
| Between Groups | 58.220 | 4 | 14.555 | 11.568 | .000 |
| Within Groups | 25.164 | 20 | 1.258 | | |
| Total | 83.385 | 24 | | | |

**Table 13 Robust Tests of Equality of Means of Variolink® N LC groups**

| | Statistic[a] | df1 | df2 | Sig. |
|---|---|---|---|---|
| Welch | 21.377 | 4 | 9.756 | .000 |

a. Asymptotically F distributed.

**Table 14 Multiple Comparisons of Variolink® N LC groups**

| | | | Mean | | | 95% Confidence Interval | |
|---|---|---|---|---|---|---|---|
| (I) Type | | | Difference (I-J) | Std. Error | Sig. | Lower Bound | Upper Bound |
| Tukey HSD | T-VL | HT-VL | -2.014800 | .709427 | .068 | -4.13767 | .10807 |
| | | ST-VL | -3.483000* | .709427 | .001 | -5.60587 | -1.36013 |
| | | XT-VL | -3.571200* | .709427 | .001 | -5.69407 | -1.44833 |
| | | C-VL | -4.286400* | .709427 | .000 | -6.40927 | -2.16353 |
| | HT-VL | T-VL | 2.014800 | .709427 | .068 | -.10807 | 4.13767 |
| | | ST-VL | -1.468200 | .709427 | .271 | -3.59107 | .65467 |
| | | XT-VL | -1.556400 | .709427 | .222 | -3.67927 | .56647 |
| | | C-VL | -2.271600* | .709427 | .032 | -4.39447 | -.14873 |
| | ST-VL | T-VL | 3.483000* | .709427 | .001 | 1.36013 | 5.60587 |
| | | HT-VL | 1.468200 | .709427 | .271 | -.65467 | 3.59107 |
| | | XT-VL | -.088200 | .709427 | 1.000 | -2.21107 | 2.03467 |
| | | C-VL | -.803400 | .709427 | .788 | -2.92627 | 1.31947 |
| | XT-VL | T-VL | 3.571200* | .709427 | .001 | 1.44833 | 5.69407 |
| | | HT-VL | 1.556400 | .709427 | .222 | -.56647 | 3.67927 |
| | | ST-VL | .088200 | .709427 | 1.000 | -2.03467 | 2.21107 |
| | | C-VL | -.715200 | .709427 | .849 | -2.83807 | 1.40767 |
| | C-VL | T-VL | 4.286400* | .709427 | .000 | 2.16353 | 6.40927 |
| | | HT-VL | 2.271600* | .709427 | .032 | .14873 | 4.39447 |
| | | ST-VL | .803400 | .709427 | .788 | -1.31947 | 2.92627 |
| | | XT-VL | .715200 | .709427 | .849 | -1.40767 | 2.83807 |

| (I) Type | | | Mean Difference (I-J) | Std. Error | Sig. | 95% Confidence Interval Lower Bound | 95% Confidence Interval Upper Bound |
|---|---|---|---|---|---|---|---|
| Games-Howell | T-VL | HT-VL | -2.014800* | .473740 | .022 | -3.70886 | -.32074 |
| | | ST-VL | -3.483000* | .568446 | .005 | -5.60828 | -1.35772 |
| | | XT-VL | -3.571200* | .390997 | .000 | -4.92423 | -2.21817 |
| | | C-VL | -4.286400* | .876497 | .025 | -7.86387 | -.70893 |
| | HT-VL | T-VL | 2.014800* | .473740 | .022 | .32074 | 3.70886 |
| | | ST-VL | -1.468200 | .638160 | .242 | -3.70481 | .76841 |
| | | XT-VL | -1.556400 | .486822 | .077 | -3.27623 | .16343 |
| | | C-VL | -2.271600 | .923236 | .224 | -5.79923 | 1.25603 |
| | ST-VL | T-VL | 3.483000* | .568446 | .005 | 1.35772 | 5.60828 |
| | | HT-VL | 1.468200 | .638160 | .242 | -.76841 | 3.70481 |
| | | XT-VL | -.088200 | .579394 | 1.000 | -2.22078 | 2.04438 |
| | | C-VL | -.803400 | .975226 | .915 | -4.35909 | 2.75229 |
| | XT-VL | T-VL | 3.571200* | .390997 | .000 | 2.21817 | 4.92423 |
| | | HT-VL | 1.556400 | .486822 | .077 | -.16343 | 3.27623 |
| | | ST-VL | .088200 | .579394 | 1.000 | -2.04438 | 2.22078 |
| | | C-VL | -.715200 | .883636 | .917 | -4.27897 | 2.84857 |
| | C-VL | T-VL | 4.286400* | .876497 | .025 | .70893 | 7.86387 |
| | | HT-VL | 2.271600 | .923236 | .224 | -1.25603 | 5.79923 |
| | | ST-VL | .803400 | .975226 | .915 | -2.75229 | 4.35909 |
| | | XT-VL | .715200 | .883636 | .917 | -2.84857 | 4.27897 |

*. The mean difference is significant at the 0.05 level.

**Table 15 Test of Homogeneity of Variance of RelyX™ U200 groups**

| Levene Statistic | df1 | df2 | Sig. |
|---|---|---|---|
| 1.287 | 4 | 20 | .308 |

**Table 16 ANOVA of RelyX™ U200 groups**

| | Sum of Squares | df | Mean Square | F | Sig. |
|---|---|---|---|---|---|
| Between Groups | 195.764 | 4 | 48.941 | 31.988 | .000 |
| Within Groups | 30.600 | 20 | 1.530 | | |
| Total | 226.363 | 24 | | | |

Table 17 Robust Tests of Equality of Means of RelyX™ U200 groups

| | Statistic[a] | df1 | df2 | Sig. |
|---|---|---|---|---|
| Welch | 47.307 | 4 | 9.847 | .000 |

a. Asymptotically F distributed.

Table 18 Multiple Comparisons of RelyX™ U200 groups

| (I) Type | | | Mean Difference (I-J) | Std. Error | Sig. | 95% Confidence Interval | |
|---|---|---|---|---|---|---|---|
| | | | | | | Lower Bound | Upper Bound |
| Tukey HSD | T-RU | HT-RU | -2.568800* | .782298 | .027 | -4.90973 | -.22787 |
| | | ST-RU | -3.008400* | .782298 | .008 | -5.34933 | -.66747 |
| | | XT-RU | -4.629200* | .782298 | .000 | -6.97013 | -2.28827 |
| | | C-RU | -8.480000* | .782298 | .000 | -10.82093 | -6.13907 |
| | HT-RU | T-RU | 2.568800* | .782298 | .027 | .22787 | 4.90973 |
| | | ST-RU | -.439600 | .782298 | .979 | -2.78053 | 1.90133 |
| | | XT-RU | -2.060400 | .782298 | .102 | -4.40133 | .28053 |
| | | C-RU | -5.911200* | .782298 | .000 | -8.25213 | -3.57027 |
| | ST-RU | T-RU | 3.008400* | .782298 | .008 | .66747 | 5.34933 |
| | | HT-RU | .439600 | .782298 | .979 | -1.90133 | 2.78053 |
| | | XT-RU | -1.620800 | .782298 | .270 | -3.96173 | .72013 |
| | | C-RU | -5.471600* | .782298 | .000 | -7.81253 | -3.13067 |
| | XT-RU | T-RU | 4.629200* | .782298 | .000 | 2.28827 | 6.97013 |
| | | HT-RU | 2.060400 | .782298 | .102 | -.28053 | 4.40133 |
| | | ST-RU | 1.620800 | .782298 | .270 | -.72013 | 3.96173 |
| | | C-RU | -3.850800* | .782298 | .001 | -6.19173 | -1.50987 |
| | C-RU | T-RU | 8.480000* | .782298 | .000 | 6.13907 | 10.82093 |
| | | HT-RU | 5.911200* | .782298 | .000 | 3.57027 | 8.25213 |
| | | ST-RU | 5.471600* | .782298 | .000 | 3.13067 | 7.81253 |
| | | XT-RU | 3.850800* | .782298 | .001 | 1.50987 | 6.19173 |
| Games-Howell | T-RU | HT-RU | -2.568800 | .977908 | .172 | -6.14328 | 1.00568 |
| | | ST-RU | -3.008400* | .699927 | .016 | -5.42665 | -.59015 |
| | | XT-RU | -4.629200* | .681299 | .001 | -6.98606 | -2.27234 |
| | | C-RU | -8.480000* | .606035 | .000 | -10.64024 | -6.31976 |
| | HT-RU | T-RU | 2.568800 | .977908 | .172 | -1.00568 | 6.14328 |
| | | ST-RU | -.439600 | .973527 | .989 | -4.00952 | 3.13032 |
| | | XT-RU | -2.060400 | .960221 | .308 | -5.61910 | 1.49830 |
| | | C-RU | -5.911200* | .908369 | .006 | -9.47270 | -2.34970 |

| (I) Type | | | Mean Difference (I-J) | Std. Error | Sig. | 95% Confidence Interval | |
|---|---|---|---|---|---|---|---|
| | | | | | | Lower Bound | Upper Bound |
| Games-Howell | ST-RU | T-RU | 3.008400* | .699927 | .016 | .59015 | 5.42665 |
| | | HT-RU | .439600 | .973527 | .989 | -3.13032 | 4.00952 |
| | | XT-RU | -1.620800 | .674995 | .209 | -3.95456 | .71296 |
| | | C-RU | -5.471600* | .598939 | .000 | -7.60092 | -3.34228 |
| | XT-RU | T-RU | 4.629200* | .681299 | .001 | 2.27234 | 6.98606 |
| | | HT-RU | 2.060400 | .960221 | .308 | -1.49830 | 5.61910 |
| | | ST-RU | 1.620800 | .674995 | .209 | -.71296 | 3.95456 |
| | | C-RU | -3.850800* | .577060 | .001 | -5.88617 | -1.81543 |
| | C-RU | T-RU | 8.480000* | .606035 | .000 | 6.31976 | 10.64024 |
| | | HT-RU | 5.911200* | .908369 | .006 | 2.34970 | 9.47270 |
| | | ST-RU | 5.471600* | .598939 | .000 | 3.34228 | 7.60092 |
| | | XT-RU | 3.850800* | .577060 | .001 | 1.81543 | 5.88617 |

*. The mean difference is significant at the 0.05 level.

**Independent Factor: Resin cement**

**Table 19 Group Statistics of the Control groups (C).**

| Cement | | N | Mean | Std. Deviation | Std. Error Mean |
|---|---|---|---|---|---|
| 24 h | Rely X™ U200 | 5 | 61.86000 | .768290 | .343590 |
| | Variolink® N LC | 5 | 48.32000 | 1.868276 | .835518 |

**Table 20 Independent Samples Test of the Control groups (C).**

| | | Levene's Test for Equality of Variances | | t-test for Equality of Means | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | 95% Confidence Interval of the Difference | |
| | | F | Sig. | t | df | Sig. (2-tailed) | Mean Difference | Std. Error Difference | Lower | Upper |
| 24 h | Equal variances assumed | 2.690 | .140 | 14.988 | 8 | .000 | 13.540000 | .903407 | 11.456739 | 15.623261 |
| | Equal variances not assumed | | | 14.988 | 5.315 | .000 | 13.540000 | .903407 | 11.258542 | 15.821458 |

Table 21 Group Statistics of the Translucent groups (T).

| Cement | | N | Mean | Std. Deviation | Std. Error Mean |
|---|---|---|---|---|---|
| 24 h | Rely X™ U200 | 5 | 53.38000 | 1.116297 | .499223 |
| | Variolink® N LC | 5 | 44.03360 | .592268 | .264870 |

Table 22 Independent Samples Test of the Translucent groups (T).

| | | Levene's Test for Equality of Variances | | t-test for Equality of Means | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | 95% Confidence Interval of the Difference | |
| | | F | Sig. | t | df | Sig. (2-tailed) | Mean Difference | Std. Error Difference | Lower | Upper |
| 24 h | Equal variances assumed | 2.396 | .160 | 16.538 | 8 | .000 | 9.346400 | .565137 | 8.043191 | 10.649609 |
| | Equal variances not assumed | | | 16.538 | 6.087 | .000 | 9.346400 | .565137 | 7.968316 | 10.724484 |

Table 23 Group Statistics of the High Translucent groups (HT).

| Cement | | N | Mean | Std. Deviation | Std. Error Mean |
|---|---|---|---|---|---|
| 24 h | Rely X™ U200 | 5 | 55.94880 | 1.880266 | .840881 |
| | Variolink® N LC | 5 | 46.04840 | .878276 | .392777 |

Table 24 Independent Samples Test of the High Translucent groups (HT).

| | | Levene's Test for Equality of Variances | | t-test for Equality of Means | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | 95% Confidence Interval of the Difference | |
| | | F | Sig. | t | df | Sig. (2-tailed) | Mean Difference | Std. Error Difference | Lower | Upper |
| 24 h | Equal variances assumed | 2.668 | .141 | 10.667 | 8 | .000 | 9.900400 | .928092 | 7.760217 | 12.040583 |
| | Equal variances not assumed | | | 10.667 | 5.666 | .000 | 9.900400 | .928092 | 7.596610 | 12.204190 |

Table 25 Group Statistics of the Super Translucent groups (ST).

| Cement | | N | Mean | Std. Deviation | Std. Error Mean |
|---|---|---|---|---|---|
| 24 h | Rely X™ U200 | 5 | 56.38840 | 1.096982 | .490585 |
| | Variolink® N LC | 5 | 47.51660 | 1.124667 | .502966 |

Table 26 Independent Samples Test of the Super Translucent groups (ST).

| | | Levene's Test for Equality of Variances | | t-test for Equality of Means | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | 95% Confidence Interval of the Difference | |
| | | F | Sig. | t | df | Sig. (2-tailed) | Mean Difference | Std. Error Difference | Lower | Upper |
| 24 h | Equal variances assumed | .022 | .885 | 12.627 | 8 | .000 | 8.871800 | .702601 | 7.251598 | 10.492002 |
| | Equal variances not assumed | | | 12.627 | 7.995 | .000 | 8.871800 | .702601 | 7.251423 | 10.492177 |

Table 27 Group Statistics of the Extra Translucent groups (XT).

| Cement | | N | Mean | Std. Deviation | Std. Error Mean |
|---|---|---|---|---|---|
| 24 h | Rely X™ U200 | 5 | 58.00920 | 1.036688 | .463621 |
| | Variolink® N LC | 5 | 47.60480 | .643126 | .287615 |

Table 28 Independent Samples Test of the Extra Translucent groups (XT).

| | | Levene's Test for Equality of Variances | | t-test for Equality of Means | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | 95% Confidence Interval of the Difference | |
| | | F | Sig. | t | df | Sig. (2-tailed) | Mean Difference | Std. Error Difference | Lower | Upper |
| 24 h | Equal variances assumed | 3.618 | .094 | 19.070 | 8 | .000 | 10.404400 | .545588 | 9.146271 | 11.662529 |
| | Equal variances not assumed | | | 19.070 | 6.682 | .000 | 10.404400 | .545588 | 9.101723 | 11.707077 |