

Effects of Different Porcelains and Shades on Light Transmission and Resin Polymerization

Keywords

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ABSTRACT

This study evaluated the effect of light-curing through two ceramics on the degree of conversion (DC) and Knoop microhardness (KHN) of resin cements. Three shades were used and light-cured directly on a Golden Gate attenuated total reflectance FT-IR plate at 30°C. KHN was tested immediately and after 24-h. Resin shade, type of ceramic, and storage time all had significant effects on the DC and KHN values. Within each shade, the choice of ceramic always had a significant effect, but the shade of the resin had a greater effect. There was an increase in KHN of all cements after 24-h of storage.

INTRODUCTION

When restoring anterior teeth, the use of ceramic veneers is considered a conservative approach compared with full-crown coverage. These veneers are bonded to the teeth using resin-based cements^{1,2} that often have a lower filler content and are less viscous than conventional resin based composites (RBCs). Resin cements exhibit improved mechanical, physical, and adhesive properties compared conventional luting agents.³ They also come in different colours to allow clinicians to select the appropriate shade for ceramic veneers. Resin cements can be categorized as chemically cured, light-activated, or dual-cured. When ceramic veneers are bonded to the tooth, it is preferable that light-activated resin cements are used, because they offer improved color stability, extended working time, and 'on demand' resin polymerization.^{4,5}

Until recently, the Quartz-Tungsten-Halogen (QTH) light-curing units were the most commonly used type of light-curing unit (LCU). Light-emitting-diode (LED) units were introduced in 2001 and have become the predominant type of curing unit used by dentists worldwide.^{6,7} Most LED units deliver light mainly in the 430 to 490 nm range while other LED units use two or more LED emitters to deliver a broader emission spectrum. Such 'dual-peak' or polywave® units deliver light below 420 nm,^{6,8,9} and are able to activate the alternative photoinitiators, e.g., Diphenyl (2,4,6-trimethylbenzoyl)-phosphine oxide (TPO), that are used in some resins.¹⁰

Achieving an adequate degree of conversion (DC) for the resin luting cement is an important factor for a successful indirect ceramic restoration. An adequate DC will improve the color stability of the resin cement, reduce microleakage, reduce pulpal irritation by reducing the amount of leached monomers, improve the physical properties of the bonded ceramic to better withstand occlusal forces, and improve adhesive strength of the bond

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between the tooth and the porcelain.^{11, 12} However, the polymerization process for resin cements may be negatively affected by their shade and amount of light transmission through the ceramic.¹²⁻¹⁵ One study reported that light transmission through ceramic may be reduced by up to 91%, depending on the type of ceramic material and its thickness.¹⁶ Another study (although not specifying the detector used) reported that the dental ceramic absorbed from 40% to 50% of the light.¹⁷ Rasetto *et al.* used the internal radiometer of Kreativ LCU to measure light and concluded that the type of LCU, the nature of the ceramic, and, most notably, the thickness of the ceramic had significant effects on the amount of light reaching the resin.¹²

In reference to the shade as a factor affecting the polymerization of resin cements, early investigations showed greater depth-of-cure in "lighter" shades when compared with darker shades.¹⁸⁻²¹ Other reports have concluded that the level of translucency and opacity of cured resins had a major impact on the amount of resin polymerization.^{18, 22, 23}

The extent of resin polymerization or the DC of dental resins can be determined by Fourier Transform-Infrared Spectroscopy (FT-IR). This technique can provide direct and both static and real-time DC test results. Other techniques used to monitor the extent of resin polymerization include both Vickers or Knoop microhardness tests or the ISO 4049 depth-of-cure test, but these tests provide only indirect results after the resin has been cured.²⁴

Since it is known that the ceramic affects the amount of light transmission, we hypothesized that resin cement polymerization would be affected by the amount of light energy delivered through different ceramic discs. The aim of this study was to evaluate the effects of three different shades of resin cement on the DC and Knoop microhardness (KHN) of a light-activated resin cement through 1.0-mm thickness of two types of ceramics that are commonly used.

The null hypotheses were that:

1. there would be no difference in both the DC and the KHN values for three different shades of the same brand of resin cement,
2. there would be no difference in DC and KHN values for resin cements cured through different ceramic materials from the same manufacturer, and
3. there would be no difference between immediate and post-curing KHN values in the three resin cements tested.

MATERIALS AND METHODS

CERAMIC

Two ceramic systems were used in this study: IPS Empress Esthetic (EST), a leucite-reinforced glass-ceramic material; and IPS e.max ZirPress (ZR), a fluorapatite glass-ceramic (both ma-

terials from Ivoclar Vivadent, Amherst, NY, USA). To simulate clinical conditions, the ceramic disks were fabricated from the system ingot by the lost-wax and heat-pressed techniques as recommended by the manufacturer. For EST, a light-translucent (ETC1) ingot was selected to fabricate the disk; the ingot was pre-heated to 700°C in a calibrated furnace (EP 600, Ivoclar Vivadent) before the pressing procedure. The ceramic disk was then steam-cleaned. For ZR, a light-translucent (LT-A2) ingot was selected and used without being pre-heated. The specimen was fired at 700°C in a calibrated furnace (EP 600, Ivoclar Vivadent) for the pressing procedure. Later, the disk was immersed in IPS e.max Press Invex liquid (<1% hydrofluoric acid), cleaned in an ultrasonic cleaner (Ney, Dentsply, York, PA, USA), and finally cleaned with airborne-particle abrasion with 100 µm Al₂O₃ powder at 2-bar pressure (BEGO, ZiroDent, Cologne, Germany). The manufacturer of the ceramic recommends a minimum thickness of 1.0 mm,²⁵ and therefore both disks in this study were 1.0 mm thick and 10 mm in diameter. Each specimen was finished with 400-grit waterproof silicon carbide abrasive papers under running water until the desired thickness was confirmed with a digital micrometer (Ultra-cal mark 3, Fowler Co., Inc., Newton, MA, USA).

DEGREE OF CONVERSION AND KNOOP HARDNESS TEST

Three base shades (A1, A4, and OP) of Variolink II resin cement (Ivoclar Vivadent) and a light-emitting-diode (LED) curing light (Bluephase G2 polywave®, Ivoclar Vivadent) were used. Every specimen was exposed for 20 s with the Bluephase G2. The total power output from this light was measured using a 6-inch integrating sphere (Labsphere, North Sutton, NH, USA) connected to a fiberoptic spectrometer (USB 4000, Ocean Optics, Dunedin, FL, USA). This sphere has an internal calibration lamp and before the start of each experiment, the equipment was calibrated using this National Institute of Standards and Technology (Gaithersburg, MD, USA) traceable light source. The energy delivered by Bluephase G2 light to the specimens was recorded both without the ceramic disk and through the 1.0-mm-thick ceramic disk by means of a MARC® resin calibrator (BlueLight Analytics Inc., Halifax, NS, Canada) with MARC v 3.0 software (BlueLight Analytics Inc.).

The base part only of the Variolink II light-cured resin cement was expressed into a 0.5-mm-thick metal ring with a 6.0-mm internal diameter centered on the diamond element of a horizontal attenuated total reflectance (ATR) attachment (Golden Gate, SPECAC, Inc., Smyrna, GA, USA) at 30°C in a Fourier Transform - Infrared (FT-IR) spectrophotometer (Tensor 27, Bruker Optics Inc., Billerica, MA, USA). A Mylar® strip was placed over the resin to prevent the formation of an air-inhibited layer on the top surface and this was then further covered by the 1.0-mm-thick disc of EST, or ZR. The Bluephase G2 was activated for 20 s, and the DC was collected for 100 s after the light was turned off. The specimen was then removed and placed immediately (within a period of 5 min) on an automated knoop microhardness testing

device (Mitutoyo Canada Inc., Mississauga, ON, Canada) with a 10-gram load for 10 s. The microhardness was measured at 9 points across the bottom surface of each specimen. After KHN was measured, the specimens were stored in distilled water for 24 h in complete darkness at 37°C, and the KHN was then re-tested. In total, 60 specimens were made (three shades x 2 ceramic systems x 10 specimens in each group), and 1,080 microhardness readings were taken (60 specimens x 9 KHN measurements x 2 times).

BEAM IMAGING

The distribution of radiant power across the LCU tip end was measured by means of a laser beam profiler. As has been previously described,^{26, 27} this device uses a CCD array camera with a 50-mm-focal-length lens (USB-L070, Ophir-Spiricon, Logan, UT, USA). The camera was positioned at a fixed distance from the ceramic upon which the light guide end was placed. A filter (FIL163200, International Light, Peabody, MA, USA) was placed between the ceramic and the camera to flatten the response of the CCD sensor a filter to the different wavelengths of light from the polywave® Bluephase G2 curing light. Images of the light source were taken in the 360-420 nm and the 420-540 nm ranges through a 10-nm-wide band-pass filter centered at 405 nm (ThorLabs, Newton, NJ, USA, FB405-10) and at 460 nm (FB460-10, ThorLabs).

The photonic count received by each camera pixel was calibrated with specific software (BeamGage v.6.1, Ophir-Spiricon). Prior to each image collection, the level of ambient light was subtracted to normalize all pixels to a similar baseline value (UltraCal, BeamGage). The LCU was then activated and allowed 5 s to stabilize, and the resulting image and data were recorded on a personal computer equipped with the appropriate software (BeamGage v6.1, Ophir-Spiricon). The appropriate average radiant power obtained using the integrating sphere was then applied to the recorded images to produce color-coded, calibrated, localized levels of irradiance values on the image.

STATISTICAL ANALYSIS

The effects of the two types of ceramic discs on the DC of three different resin cement shades, the immediate, and the 24-hour KHN were analyzed first with two-way analysis of variance (ANOVA) and then with Fisher's Protected Least Significant Difference (PLSD) post-hoc multiple comparison tests at $\alpha = 0.05$.

RESULTS

IRRADIANCE AND RADIANT EXPOSURE DELIVERED THROUGH THE CERAMIC.

The beam imaging (irradiance uniformity and light distribution) across the tip end of the LCU and through the two ceramics is shown in Figure 1a and b. The radiant exposures delivered to the

specimens are shown in Figure 2. The samples cured through the EST ceramic received more energy. After 20 s, EST samples received 9.8 J/cm² of energy, compared with 6.3 J/cm² for ZR; this represented a 55.6% increase. Conversely, the radiant exposure without the ceramic disc between the LCU and the sensor was 20.15 J/cm².

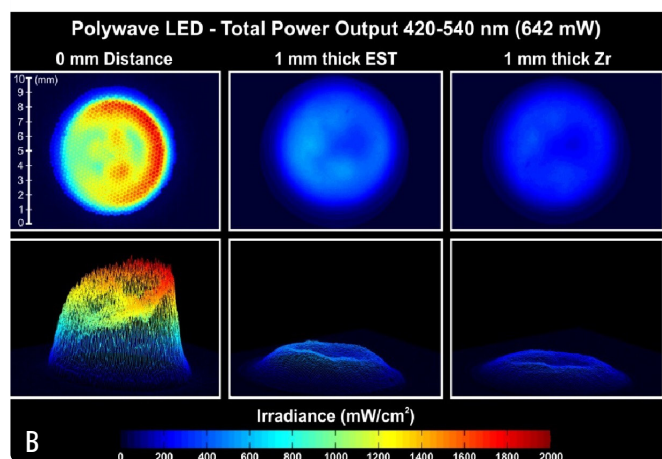
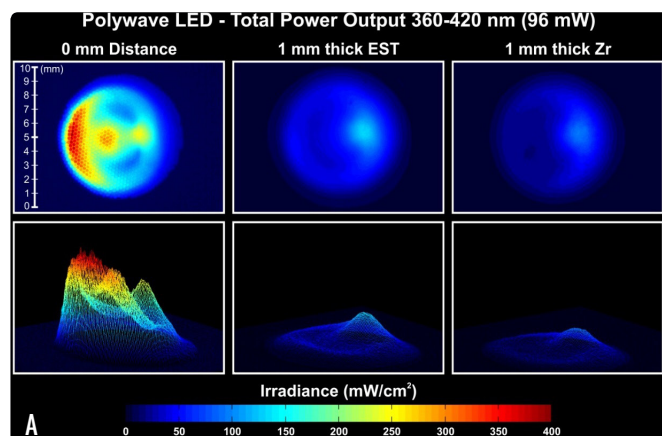


Figure 1a-b: Irradiance (mW/cm²) distribution image shows the beam profile at the LCU tip and through 1-mm-thick EST and ZR ceramics in wavelength ranges of (a) 360-420 nm, and (b) 420-540 nm.

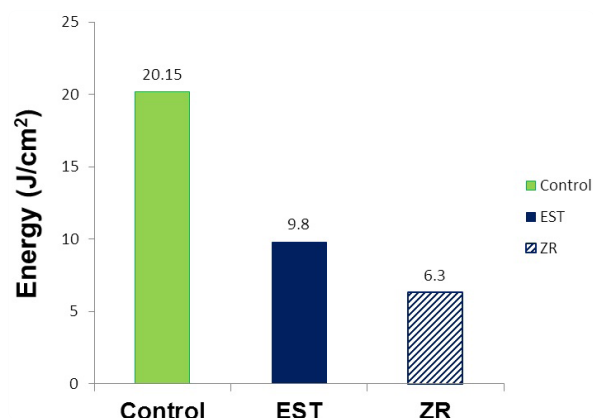


Figure 2: Radiant exposure (J/cm²) delivered at the tip control and through EST and ZR ceramics after 20 s of light exposure.

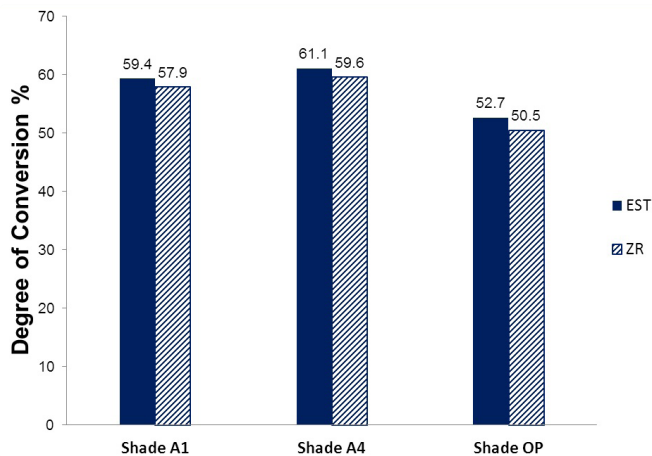


Figure 3: Degree of conversion (%) of three resin cements for each shade and type of ceramic.

DEGREE OF CONVERSION (DC)

The DC measurements for resin specimens are shown in Table 1 and Figure 3. In general, the specimens cured through the EST ceramic achieved a higher DC compared to those cured through ZR. Shade A4 specimen had the highest values (61.1% for EST and 59.6% for ZR), whereas the OP shade samples recorded the lowest

DC values (52.7% for EST and 50.5% for ZR). All values were highly statistically significant according to Fisher's PLSD test ($P < 0.0001$).

KNOOP MICROHARDNESS NUMBER (KHN)

The results for the immediate and 24-hour KHN values are shown in Table 2 and Figure 4. Both two-way ANOVA and Fisher's PLSD tests showed that effects of light curing for different shades were very significantly different for the resin cured through both ceramics ($P < 0.0001$). For all specimens, the microhardness values of the resin cement after 24 h of storage were also very significantly greater compared with their immediate KHN values ($P < 0.0001$). When light-cured through both ceramics, with shade A4, the KHN values were higher compared with those of shade A1. The KHN was 13.1% greater when cured through EST, with a larger 30% increase when cured through ZR. Conversely, the KHN values of the OP samples recorded the lowest values [1.94 (EST) and 1.72 (ZR)].

Table 2 also shows that the resin specimens (in all 3 shades) light-cured through the EST ceramic had higher values compared with those cured through the ZR ceramic. Fisher's PLSD test showed a highly statistically significant difference between specimens cured through ZR and those cured through EST ($P < 0.0001$). There was a linear relationship between DC and KHN ($R^2 = 0.99$) within the range of DC and KHN values achieved in this study.

Table 1. Mean values and standard deviations (SD) for degree of conversion (DC) of resin cements for each shade and type of ceramic. All comparisons were highly significant.

Ceramic	Resin shades	DC % Mean (SD)	Two-way ANOVA test	Fisher's PLSD test		
EST	A1	59.4 (0.01)	< 0.0001	EST	ZR	0.0002
	A4	61.1 (0.01)		A1	A4	0.0023
	OP	52.7 (0.02)		A1	OP	< 0.0001
ZR	A1	57.9 (0.01)	< 0.0001	A4	OP	< 0.0001
	A4	59.6 (0.01)				
	OP	50.5 (0.01)				

Table 2. Mean values and standard deviations (SD) for immediate and delayed (24 h) KHN for resin cements for each shade and type of ceramic. All comparisons were highly significant.

Ceramic	Resin shades	Immediate-KHN Mean (SD)	24-hour KHN Mean (SD)	Two-way ANOVA test	Fisher's PLSD test		
EST	A1	2.98 (0.23)	8.1 (0.42)	< 0.0001	EST	ZR	< 0.0001
	A4	3.37 (0.17)	9.09 (0.52)		A1	A4	< 0.0001
	OP	1.94 (0.16)	5.63 (0.46)		A1	OP	< 0.0001
ZR	A1	2.51 (0.15)	7.03 (0.41)	< 0.0001	A4	OP	< 0.0001
	A4	3.27 (0.16)	7.62 (0.39)		Immediate	24-hour	< 0.0001
	OP	1.72 (0.12)	4.15 (0.84)				

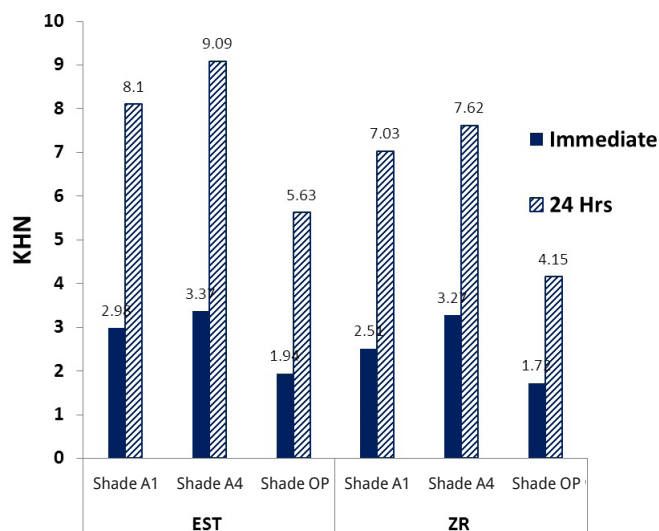


Figure 4: Immediate and 24-hour KHN values for the resin cements for each shade and type of ceramic.

DISCUSSION

Our findings showed that there were highly significant differences in both KHN and DC values between all three shades of resin cement. Thus, the first hypothesis was rejected. When the effects of curing different shades of resin cements through different ceramic materials were compared, there was a large and statistically significant difference. Thus, the second hypothesis was rejected. Also, there was a highly statistically significant increase for all tested specimen values when the microhardness was re-tested after 24 h. Thus, the third hypothesis was rejected.

Adequate polymerization of resin cements is essential to produce of an acceptable ceramic restoration. The preferred requirement of energy delivered for the adequate cure of a 1- to 2-mm thickness of resin-based composite increment has been reported to range between 16 and 24 J/cm².²⁸⁻³⁰ The manufacturer's instructions (Ivoclar Vivadent) for the ceramic and resin used in this study recommend the use of their Bluephase G2 light on the high-power mode for 10 s for each mm of ceramic thickness to cure Variolink II resin cement. In the present study, the Bluephase G2 was used in the high-power mode for 20 s, thus doubling the recommended time to simulate clinical protocols and ensure that sufficient energy was delivered to the specimens. The radiant exposure delivered by the LCU was 20.15 J/cm². This value was reduced to 9.8 J/cm² after passing through EST and was reduced to 6.3 J/cm² after passing through the ZR ceramic. The 51% reduction in light transmission through EST and 69% reduction through ZR were expected.¹⁶ Peixoto *et al.* used a digital power meter (Newport Optical Power Meter) to measure light transmission and also confirmed power reduction when light passed through different thickness of ceramics. The present findings clearly showed that more light passed through EST compared with ZR ceramic material, and almost 50% more energy was delivered to the resin in the same time from the same

LCU. These findings were in agreement with those reported from other studies.^{31, 32} The two ceramics have different refractive indices (an average number of 1.53 for EST and 2.18 for ZR),^{33, 34} and that could explain why the EST ceramic allowed more light to pass through.

A ceramic veneer is considered a conservative procedure when compared with partial or full-crown coverage. The amount tooth reduction ranges from 0.5 to 1.0 mm, depending on the clinical condition and patient requirements; both the ceramic material and resin cement shade help to deliver the optimum final esthetic shade. For example, dark substrate (discolored dentine or a metallic core) could require both resin cement with an opaque shade and a less translucent ceramic material coverage to mask the dark substructure. Also, the use of a lighter shade of resin cement can help to lighten the final outcome of a ceramic restoration shade. In our study, the OP specimen had the lowest DC and KHN values, probably due to the opaque within this cement that caused greater absorption and attenuation of light. This is in agreement with previous studies.^{19, 23, 35}

When other shades were evaluated, the darker shade (A4) was harder and achieved a greater conversion when compared with the lighter A1 shade. Previous reports in the dental literature supported the need for extended light exposure time for darker-shade RBCs to achieve the same hardness and mechanical properties as compared with lighter shades of RBCs. This is supported by several studies.³⁶⁻³⁹ Conversely, more recent studies support the contention that it is the opacity, and not the darkness or lightness of the resin, that affects resin photopolymerization.^{23, 31, 35, 40-42} These studies were in agreement with our findings that showed the darker shade could be equivalent to or harder than lighter resin shade when cured with the same LCUs. The explanation for these outcomes could be due to the fact that the A4 resin cement specimen may contain a greater concentration of the camphorquinone photoinitiator that has a yellow colour.

Almost all previous studies based their conclusions on microhardness ISO tests (ISO 4545) or on the classic depth-of-cure test (ISO 4049). Our findings were collected based on both an indirect method (KHN) and a direct method that measured the DC after photopolymerization at a clinically relevant temperature of 30°C. Previous studies either were conducted at room temperature, or there was no mention of the temperature conditions. In the present study, a 30°C temperature setting was used to simulate clinical conditions, because it has been shown that polymerizing resin at mouth temperature will result in greater polymerization and improved physical properties.⁴³ Higher temperatures will increase molecular mobility, leading to an increase in monomer conversion.^{44, 45} This may explain why our values were different from those of others.

The highly statistically significant difference between the immediate and delayed (24 h) KHN, supports previous studies.^{5, 46, 47} Thus, the polymerization process continues after exposure, and the patient should be cautioned not to chew vigorously on light-activated resin-bonded ceramic restorations for 24 h.

CONCLUSIONS

In reference to our findings, we conclude the following:

- The opaque shade of the resin cement investigated produced the lowest DC and KHN values.
- After 24 h of dark storage in water at 37°C, there was a significant and marked increase in KHN for all three resin cements.
- The resin cements reached greater DC and hardness values through the EST ceramic than through the ZR ceramic.

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The winners of the 2016 Frechette Awards were recently presented at the International Association for Dental Research annual meeting in Seoul, Republic of Korea. The Frechette is the annual research competition for new investigators sponsored by the Prosthodontics Group of the International Association for Dental Research and honoring the memory of Arthur R. Frechette who served as Secretary-Treasurer of the IADR from 1968 until 1977. The winners were Dr Emilio Hara, Okayama University, Okayama-shi, Japan for "Chondrocyte Burst Promotes Space for Mineral Expansion During Initial Mineralization" and Dr Candida Parisi, University of Bologna, Bologna, Italy for "Translucency of Monolithic Hybrid Cubic/Tetragonal Zirconia Crowns: A Comparative Study". The individuals pictured are (left-to-right) Dr David Bartlett, President IADR Prosthodontics Group; Dr Jamila Almuhamadi, Barts and the London, School of Dentistry, London, UK, Frechette Award Finalist; Dr Hara, Frechette Award Winner; Dr Parisi, Frechette Award Winner; Dr Lianyi Xu, Ninth People's Hospital, Shanghai Jiao Tong University, School of Medicine, Shanghai, China, Frechette Award Finalist; and Dr. Stephen F. Rosenstiel, Frechette Coordinator IADR Prosthodontics Group. In addition to the plaques Drs. Hara and Parisi each received a check for \$1000 thanks to the generous support of the Whip Mix Corporation.

New investigators in Prosthodontics are encouraged to apply for one of two 2017 Arthur R. Frechette Research Awards in Prosthodontics. Each Award recognizes original research by new investigators and is sponsored by the Prosthodontics Group of the IADR and supported by the Whip Mix Corporation. One award is for Prosthodontic research undertaken with a Materials Science or Bioengineering oriented protocol and the other is for Prosthodontic research undertaken with a Biological Sciences and Tissue Engineering oriented protocol. Researchers carrying out original research are eligible for the Frechette Award if they have been the primary author of no more than three articles published in refereed dental journals. Each award carries a cash prize of \$1,000.

Research submitted for the IADR meeting in San Francisco, California is eligible for consideration for the Frechette Award, provided it has not been published elsewhere or will not be under consideration for another award. Abstracts must be submitted to the Prosthodontics Group of the IADR by the IADR deadline with the appropriate boxes on the on-line submission checked.

The abstracts will be judged for (1) originality and scientific design of the investigation, (2) suitability of the methods of analysis, and (3) scientific value of the work. Selected finalists will be judged at the 2017 IADR meeting in San Francisco, California (March 22-25, 2017) and the winner will be announced at the Prosthodontics Group Business Meeting.