

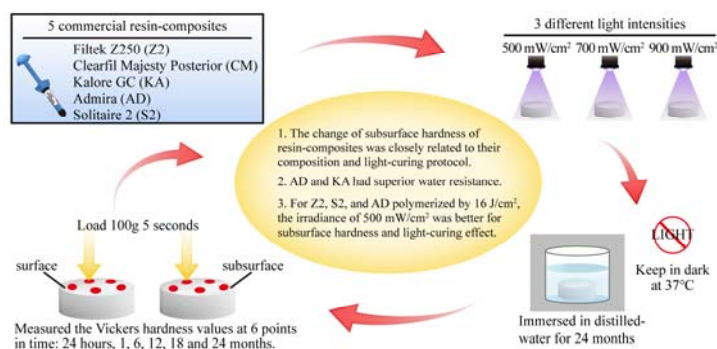
EFFECT OF LIGHT INTENSITY AND WATER AGING ON SURFACE HARDNESS OF DENTAL RESIN-COMPOSITES

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This study assessed the effect of light intensity and water aging on surface hardness of 5 resin-composites. Five specimens were developed per material. The specimens were cured by 3 different light intensities and then stored in distilled water for 24 months. A Vickers hardness tester was used to measure hardness values after different storage durations. The subsurface hardness values of Z250 (Z2) and Clearfil Majesty Posterior (CM) decreased significantly after immersion. Kalore GC (KA) and Admira (AD) showed higher hardness values compared to the baseline. As for Solitaire 2 (S2), no significant change was detected. Z2, S2, and AD showed harder subsurfaces under the irradiation of 500 mW/cm² after immersion for 24 months. The subsurface hardness values of resin-composites are closely related to composition and light-curing protocol. AD and KA showed superior water resistance. For Z2, S2, and AD, the light intensity of 500 mW/cm² was better for subsurface hardness.



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INTRODUCTION

Light-curing resin-composite has been widely used in dentistry due to its good aesthetics, biocompatibility, and mechanical properties. It is mainly composed of an organic matrix, inorganic filler, and an initiation system. The matrix undergoes a cross-linking reaction due to the effect of the initiating system, rendering the carbon-carbon double bonds of the monomer molecule broken. They are then converted into carbon-carbon single bonds (C-C) and form a polymer.^{1,2} The mechanical properties and durability of resin-composites depend on the composition of the

materials and the environment in which they are exposed to. All these will determine the clinical performance of the resin-composite. When a resin-composite is applied in the oral environment, water in the oral cavity can cause hydrolysis of inorganic filler particles, weakening of the polymer network, debonding of the filler-matrix interfaces, and ultimately lead to a decrease in the functional properties of the materials.³ As a physical characteristic, hardness is closely related to the durability of restorations. Mandikos *et al.*⁴ defined hardness as a measure of the ability of a material to resist indentation or scratching. Hardness is important in maintaining the form and stability of

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restorations, it is also correlated to the ductility, smoothness, wear resistance, and compression resistance of the materials.⁵ At present, the *in vitro* medium immersion is a common experimental method for artificial aging. Water aging is the method that is most commonly used.^{3,6} In addition, the surface hardness can reflect the degree of monomer conversion of resin-composites and changes in mechanical properties of the materials under a continuous aging process.^{7,8} Many experiments have been conducted to examine changes in the properties of resin-composites under short-term water immersion.^{3, 9, 10} However, there is a scarcity of data with regard to the changes on the surface hardness of resin-composites after long-term water aging.

It has been widely known that the clinical longevity of resin-composites is closely related to the light-curing protocol. Light intensity and exposure time affect the conversion of monomers into polymers and alter the mechanical properties of the materials.¹¹ Due to the existing need for reduction in curing exposure to minimize the chairside times of incremental filling of resin-composites restorations, some researchers support the exposure reciprocity law. This law states that the radiant energy (J/cm^2), the product of irradiance (mW/cm^2) and exposure time (s), is the main determinant of the degree of conversion and mechanical properties of resin-composites. This encourages the application of high light intensity to reduce curing time. However, other studies suggested that even though radiant energy plays an important role, the light intensity and exposure time may affect polymer chain length and degree of cross-linking. Thus, the exposure reciprocity law does not hold true all the time.¹²

In view of the conflicting opinions on this matter, this study applied 3 different light intensities, set at the same radiant energy on 5 commercial resin-composites immersed in distilled water, namely Filtek Z250, Solitaire 2, Clearfil Majesty Posterior, Kalore GC, and Admira. The hardness on the surface and the bottom of the materials were then measured at fixed intervals. The effects of water aging and light intensity on the surface hardness of resin-composites over 24 months were also investigated in this study.

RESULTS

The results showed that the ratio of the subsurface/surface hardness of each resin-composite exceeded 80%, indicating that all the

materials had been fully cured. Therefore, only the subsurface hardness was analyzed. The measured hardness values of the five resin-composites were shown in Table 2. Hardness values prior to immersion were used as a baseline (24 h). Regardless of the light intensity, the subsurface hardness values of Z2 and CM decreased significantly after 24 months of immersion ($p < 0.05$). On the contrary, KA and AD were found to have harder subsurface values compared to the baseline ($p < 0.05$). As for S2, no significant change was detected between the same storage period. However, S2 and AD had harder surfaces when they were cured by $500 \text{ mW}/\text{cm}^2$ prior to immersion. After the 24-month immersion, the subsurface hardness values of Z2 and S2 irradiated by $500 \text{ mW}/\text{cm}^2$ were significantly higher than that of $900 \text{ mW}/\text{cm}^2$ ($p < 0.05$), but the values were not significantly different from that of $700 \text{ mW}/\text{cm}^2$. The subsurface hardness value of AD cured by $500 \text{ mW}/\text{cm}^2$ were significantly higher than that of $700 \text{ mW}/\text{cm}^2$ and $900 \text{ mW}/\text{cm}^2$ ($p < 0.05$). Nevertheless, the hardness values of the other resin-composites were not significantly different under the 3 different light intensities.

DISCUSSION

It is generally considered that resin-composite with a thickness of 2 mm could be fully polymerized when irradiated by a light curing unit at the irradiance of $400 \text{ mW}/\text{cm}^2$ with wavelengths in the range of 450-500 nm for 40 seconds.¹³ Therefore, in this study, the thickness of all the resin-composites specimens were standardized at 2 mm, whereas light intensity was set as $500 \text{ mW}/\text{cm}^2$, $700 \text{ mW}/\text{cm}^2$, and $900 \text{ mW}/\text{cm}^2$ respectively with the aim to evaluate which setting is more favorable for the full curing of resin-composites at the same radiant energy, thereby providing the much needed information for clinical application. In a previous study, we found that $16 \text{ J}/\text{cm}^2$ of radiant energy was better at polymerizing most of the resin-composites compared with $12 \text{ J}/\text{cm}^2$.¹⁴ Therefore, the radiant energy for this experiment was set as $16 \text{ J}/\text{cm}^2$. Distilled water was used instead of artificial saliva in this study because studies have shown that there was no significant difference in the effect of distilled water and artificial saliva on the aging behavior of resin-composites.¹⁵ Equivalent degradation of resin-composites was also described by Yap *et al.* after aging in either distilled water or artificial saliva.¹⁶

Table 1
Technical profiles of resin-composites investigated in the study

Materials	Code	Type	Matrix	Filler type	Filler content (vol%/wt%)	Manufacturer	Batch number
Filtek Z250	Z2	Universal	Bis-GMA, Bis-EMA, UDMA, TEGDMA	Zirconia, Silica	60/82	3M ESPE, St. Paul, MN, America	N657490
Solitaire2	S2	Universal	Bis-EMA, UDMA, TEGDMA	Aluminum silicate glass, Barium fluorosilicate, Porous silica	>90/-	Heraeus Kulzer Dental Ltd, Wehrheim, Hesse, Germany	30603
Clearfil Majesty Posterior	CM	Low-Shrinkage	Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate	Surface treated alumina micro filler, Silanated glass ceramics	82/92	Kuraray Noritake Dental Inc, Kurashiki, Okayama, Japan	AQ0002
Kalore GC	KA	Low-shrinkage	DX-511, UDMA, Dimethacrylate comonomer	Pre-polymerized filler (lanthanide fluoride), Fluoroaluminum silicate glass, Silica strontium / barium glass,	69/82	GC Corporation, Kasugai, Aichi, Japan	1309242
Admira	AD	Low-shrinkage	Organic modified ceramics, Aliphatic and aromatic dimethacrylates	Barium-aluminum glass, Silica	56/78	Voco GmbH, Lower Saxony, Cuxhaven, Germany	1413134

Bis-GMA: bisphenol A glycol dimethacrylate;

Bis-EMA: ethoxylated bisphenol-A dimethacrylate;

UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate

Table 2

Mean (standard deviation) for Vickers hardness (kg/mm^2) of five resin-composites cured by different light intensities at different water aging durations and percentage change

Materials	Light intensity (mW/cm^2)	Immersion time						Percentage change (%) [*]
		24 h	1 mon	6 mon	12 mon	18 mon	24 mon	
Z2	500	82.28(1.94) ^{Aa}	79.34(2.59) ^{ABab}	79.24(1.21) ^{ABa}	79.14(1.99) ^{ABa}	75.12(2.26) ^{Ba}	77.58(3.41) ^{Ba}	-5.71
	700	81.00(1.35) ^{Aa}	76.22(0.97) ^{BCa}	76.76(0.74) ^{Bb}	74.42(0.98) ^{BCb}	73.70(2.44) ^{Ca}	74.74(1.67) ^{BCab}	-7.73
	900	83.20(2.84) ^{Aa}	79.92(2.50) ^{ABb}	76.30(1.09) ^{BCb}	73.18(1.64) ^{Cb}	72.92(1.74) ^{Ca}	72.04(4.04) ^{Cb}	-13.41
S2	500	44.26(1.11) ^{Aa}	42.64(1.53) ^{Aa}	43.34(1.01) ^{Aa}	41.74(1.04) ^{Aa}	41.44(2.68) ^{Aa}	43.76(0.83) ^{Aa}	-1.13
	700	44.00(1.39) ^{Aa}	40.78(0.60) ^{Bb}	44.16(1.11) ^{Aa}	42.06(1.16) ^{ABa}	41.22(1.78) ^{Ba}	43.36(1.71) ^{ABa}	-1.45
	900	40.42(1.79) ^{Ab}	37.48(0.88) ^{BCc}	37.08(1.48) ^{BCb}	35.04(1.26) ^{Cb}	35.40(1.77) ^{Cb}	38.52(1.47) ^{ABb}	-4.70
CM	500	111.40(1.34) ^{Aa}	105.00(2.00) ^{Ba}	99.76(1.34) ^{Ca}	101.00(1.58) ^{Ca}	99.20(0.90) ^{Ca}	106.62(2.04) ^{Ba}	-4.29
	700	110.80(1.30) ^{Aa}	103.80(0.84) ^{Bab}	99.50(2.04) ^{Ca}	100.52(1.91) ^{Ca}	98.96(1.33) ^{Ca}	104.32(1.63) ^{Ba}	-5.85
	900	112.00(0.71) ^{Aa}	102.52(1.08) ^{Bb}	101.40(1.35) ^{Ba}	101.60(1.67) ^{Ba}	99.74(0.85) ^{Ba}	105.80(2.59) ^{Ca}	-5.54
KA	500	35.76(0.79) ^{Aa}	37.36(0.38) ^{Aa}	41.30(0.72) ^{Ba}	39.30(1.21) ^{Ca}	39.92(0.72) ^{BCa}	39.32(1.46) ^{Ca}	9.96
	700	34.28(2.07) ^{Aa}	39.66(0.84) ^{Bb}	41.54(0.96) ^{Ba}	40.08(1.14) ^{Ba}	41.00(1.01) ^{Ba}	40.70(2.47) ^{Ba}	18.73
	900	34.24(1.26) ^{Aa}	36.16(0.85) ^{Aa}	39.42(0.60) ^{Bb}	40.84(1.47) ^{Ba}	40.44(1.40) ^{Ba}	39.12(1.67) ^{Ba}	14.25
AD	500	45.38(0.44) ^{Aa}	48.26(0.80) ^{Ba}	47.90(0.42) ^{Ba}	47.06(0.42) ^{Ba}	45.48(1.05) ^{Aa}	47.08(0.89) ^{Ba}	3.75
	700	41.60(1.31) ^{Ab}	44.70(1.28) ^{Bb}	44.74(0.77) ^{Bb}	45.34(0.85) ^{Bb}	44.88(0.77) ^{Ba}	44.64(1.27) ^{Bb}	7.31
	900	42.30(1.12) ^{Ab}	45.94(0.93) ^{BCb}	47.36(0.44) ^{Ca}	46.40(0.88) ^{BCab}	45.02(0.59) ^{Ba}	44.76(1.75) ^{Bb}	5.82

*Percentage change are expressed as the percentage decrease or the percentage increase of subsurface hardness values between 24 h and 24 mon.

Means followed by distinct capital letters in the same row and distinct lowercase letters in the same column within the same materials indicate significant differences ($p < 0.05$).

In this study, CM was found to record the highest hardness values, followed by Z2, S2, KA, and AD. The difference in hardness is commonly related to the content, size, distribution of filler particles, the type of matrix, and degree of conversion.^{17,18} Although Z2 had a higher hardness value, it was reduced by 5.71% to 13.41% after 24 months of water aging. This was found to be the greatest decrease in hardness values among all materials. Our study echoed the findings of de Moraes *et al.*¹⁹ which immersed Z2 in water for 1 year was found to have a significant reduction in the subsurface hardness values. As a typical methacrylate resin, Z2 undergoes a universal hardness change of resin in water. Water sorption in resin-composites is considered a diffusion-controlled process. When water molecules enter the matrix of resin, the polymer networks are softened due to the swelling of resin matrix and a reduction in the frictional forces between the polymer chains. This results in the release of unreacted monomers and by-products of polymer networks into the solution.^{3,20} The degradation of resin-composites, induced by water, mainly happens via 2 mechanisms. Firstly, the effect of water on the silane interface would result in the hydrolytic cleavage of the silane bonds. Secondly, the water also renders a plasticizing effect to soften the resin-composites. As a result, the mechanical properties of the materials are degraded.⁹ In other words, water sorption of resin-composites will cast a great influence on the surface hardness, and it is mainly determined by the composition of resin matrix.²¹ Bis-GMA-based materials have been found to undergo more water sorption than urethane-based resins.²² The increase in the TEGDMA content in resin matrix leads to an increase in water sorption because TEGDMA monomer exhibits higher hydrophilicity than Bis-GMA and UDMA.²³ Z2 is mainly composed of the above monomers, which explains the significant decrease in the surface hardness values of Z2 upon water sorption. In addition, water sorption of the materials also depends on inorganic fillers. Water diffuses through the resin matrix and reacts with the fillers at the filler-matrix interface, so the differences in water uptake may be related to the nature of the filler particle and the coupling agent.²¹ Yap *et al.*¹⁶ reported that zirconia glass fillers were susceptible to water.

Among all the resin-composites, CM showed the highest initial hardness value, because it is a nano-hybrid material with a very high proportion of fillers (82% by volume, 92% by weight).

Furthermore, it has been found that Vickers hardness is also dependent on the quantity of inorganic fillers in the resin; an increase in fillers results in a higher hardness value.¹⁷ As compared with micro-filled resin-composites, nano-filled resin-composites have larger contact areas with the organic matrix, leading to a higher wetting of fillers by the resin, thus improving the hardness of the material. Moreover, the physical properties of resin-composites are related to the inter-particle spacing in the material. The smaller the distance between particles, the higher the hardness value of the resin-composites. Nano-filled resins can reduce inter-particle spacing and improve the hardness by decreasing the particle size and increasing the filler content.¹⁸ The matrix of CM is mainly composed of Bis-GMA and TEGDMA with a strong water sorption ability. Nano-fillers have been linked with an increase in water uptake due to their large surface to volume ratio.⁹ This may explain why the hardness values of CM decreased significantly after immersion in water for 24 months in this study. Although the hardness values showed a downward trend, CM still maintained as high as 95% of initial surface hardness. This is because CM also contains hydrophobic aromatic dimethacrylate monomer. Moreover, resin-composites containing high filler loading have fewer micropores, thus resulting in less space for water molecules and reduced water uptake of materials.

Although the hardness values of S2 decreased after water aging, the result was not statistically significant. This is because S2 matrix contains Bis-EMA but not Bis-GMA. Bis-EMA is a monomer similar to Bis-GMA but it lacks 2 hydrophilic hydroxyl groups. Cornelio *et al.*²⁴ found that the substitution of bis-GMA by bis-EMA resulted in reduced values for water sorption and solubility of the experimental resin. Moreover, S2 displays the highest filler loading among all the investigated materials, which translates into a decrease in water uptake. Krüger *et al.*²⁵ found that the effect of *in-vitro* aging on Vickers hardness was mainly influenced by filler content in which an increase in filler content would improve the resistance of the material against chemical degradation process. AD is a resin-composite produced from organically modified ceramics (ORMOCERS), which is a hybrid structure generated by special processing process using molecular scale technology in which the sol-gel method is adopted to combine organic and inorganic components at a nanoscopic scale. The main characteristic of this material is the

linking of organic groups to the inorganic backbone by hydrolysis and condensation of alkoxides.²⁶ Based on our findings, the hardness value of AD slightly increased after long-term immersion in water, which was consistent with the previous study by Cavalcante.²⁶ This may be due to the fact that the organic-inorganic network in ORMOCERS is different from the organic network that is present in the dimethacrylate resins. The inorganic backbone of ORMOCERS results in high hydrophobic behavior, thus decreasing the water uptake of materials and improving its resistance to chemical degradation processes.^{26,27}

KA also is a nano-hybrid resin-composite, but its initial hardness value was the lowest among all materials. This is likely due to the use of pre-polymerized fillers. Blackham *et al.*²⁸ reported that the incorporation of pre-polymerized fillers to a resin-composite might lead to a decrease in its mechanical properties. The bond between polymer matrix and pre-polymerized fillers is considered to be a weak spot, and the use of pre-polymerized fillers may result in a decrease in the actual percentage of the anorganic phase and eventually compromising its mechanical properties.⁹ In this study, the hardness value of KA increased significantly by 10%-20% after artificial aging of 24 months. Due to the stiff structure of its matrix, KA has been found to have a lower water sorption value after immersion in water when compared with other resin-composites.²⁹ The matrix of KA is mainly composed of UDMA and DX-511. DX-511 is a high molecular-based monomer based on the chemistry of urethane dimethacrylates and owns a stiff molecular core. As for the monomer UDMA, it has been reported to show lower long-term water sorption than Bis-GMA and TEGDMA.³⁰ Moreover, KA does not contain Bis-GMA and TEGDMA, but instead contains a small amount of Bis-EMA, thus the water uptake is further reduced. In addition, the silica surfaces in KA are treated with dimethyl constituents for hydrophobic treatment, which is more stable than methacryloxysilane-treated silica, improving service life and reducing the risk of softening during storage.³¹ Unlike the other three resin-composites, the hardness values of AD and KA increased during the initial artificial aging process but began to decrease after reaching the peak. However, the final hardness values were still higher than the initial pre-aging values. Apart from the underlying composition of AD and KA, the occurrence of additional post-curing cross-linking reactions in the resin also contributed to the

increase in hardness values. The post-curing stage refers to the effect of free radical polymerization in a monomer system that can last for several days to months after stopping the illumination.³²

In the comparison of the subsurface hardness of resin-composites before and after aging, the hardness values of S2 and AD were found to be higher under the irradiance of 500 mW/cm² at 24 h. However, the values of the other three resins were not significantly different under different light intensities. Z2, S2, and AD recorded the highest hardness values under the irradiation of 500 mW/cm² after aging for 24 months, while there was no significant difference in hardness values between different light intensities for the other 2 resin-composites. While a high power intensity with short curing time can save the chairside times, it is vital to take into account that rapid polymerization could also induce shorter polymer chain lengths and lower average molecular weight of polymers, resulting in an incomplete polymerization and ultimately a weaker physical and mechanical properties of the materials.³³ In addition, free radical reaction times are shorter during curing process under high light intensity. When this happens, a cross-linked network structure would rapidly form on the surfaces of the materials, which can hinder the transmission of light and result in the incomplete curing on the subsurfaces.^{33,34} In contrast, the low light intensity with long period exposure will not cause rapid polymerization of resin-composites. They will be able to receive more photons for reaction and achieve a sufficient flow before the vitrification stage, thereby reducing the reaction rate and stress on the materials.^{26,35} In this study, Z2, CM, and S2 reported a decrease in hardness values, the percentage decrease of subsurface hardness values was lowest when they were irradiated by 500 mW/cm² after water immersion, which result in a difference in the effect of light intensity on hardness before and after aging of Z2. This phenomenon might be due to the incomplete polymerization generated by high power intensity which has the unintended and unwanted effect of increased water sorption and the solubility of unreacted monomers.³³ As for AD and KA, the percentage increase of hardness values was lowest under the irradiation of 500 mW/cm² after aging. A possible reason for this might be the large burst of photon supply within a short period of time due to the irradiation of high light intensity. This results in an increased temperature, which might have a positive effect on post-curing reactions.

EXPERIMENTAL

Composite materials and instruments

In this study, 5 commercially available resin-composites from different manufacturers were investigated. The types and composition of materials are outlined in Table 1. Filtek Z250 (Z2) and Solitaire 2 (S2) are considered universal resin-composites. Clearfil Majesty Posterior (CM), Kalore GC (KA), and Admira (AD) are recommended as low-shrinkage resin-composites. The radiant energy of all resin-composites specimens in this experiment was set at 16 J/cm². Based on this, 3 light-emitting diode (LED) curing lights were used to irradiate at 3 different intensities: 500 mW/cm² for 32 seconds (Ski LED Curing Light, Duoyimei Medical Instrument Corporation, Foshan, Guangdong, China), 700 mW/cm² for 23 seconds (Rainbow LED Curing Light, Yaling Dental Instrument Corporation, Guangzhou, Guangdong, China), and 900 mW/cm² for 18 seconds (SLC-VIII A Curing Light, Hangzhou Sifang Instrument Corporation, Hangzhou, Zhejiang, China). The power intensity was measured by a LED radiometer (model 644726, Dentsply International, York, PA, USA). The surface of its sensing window was coated with a polyester film to simulate the illumination of the surface of resin-composites during the curing process. The hardness was measured using a Vickers hardness measuring instrument (HMV-G21ST, Shimadzu Corporation, Tyoko, Japan).

Measurement process

Preparation of specimens

The resin-composites were first placed into a cylindrical-shaped copper mould (inner diameter 6 mm and thickness 2 mm). A polyester film and a glass slide were placed against the top and bottom surfaces to prevent the formation of an anaerobic layer during the curing process. Any excessive resin was gently squeezed out to avoid the formation of bubbles before the glass slides were removed. The resin-composites specimens were cured using 3 LED curing lights. The light source was directed in a perpendicular direction to the surface of the specimen, with the tip placed 1 mm away from the surface of the resin-composites. At the end of the irradiation, the specimens were removed from the mould, immersed in distilled water, and stored at 37°C in dark. Following that, the resin-composites were divided into 15 groups according to 3 light-curing protocols. Five specimens were made per material, totalling 75 specimens.

Vickers hardness test

During the storage period, the specimens were stored in distilled water for 24 months and the water was changed weekly. The surface and subsurface Vickers hardness values of specimens were measured by HMV-G21ST at 6 points in time; 24 hours, 1 month, 6 months, 12 months, 18 months, and 24 months. For each specimen, a load of 100 g was applied for a dwell time of 5 seconds. This created 5 indentations on the surface and subsurface and they were separated by at least 1 mm. The hardness values were then calculated by measuring the indentations. The subsurface/surface hardness of each experimental group was calculated after each measurement. The Vickers hardness percentage of each group = (average subsurface hardness of each group at a certain point of time)/(the maximum surface hardness of each group at the same time) × 100. If the ratio exceeded 80%, the resin was considered to have been fully cured.

Statistical analysis

Data analysis was performed using statistical software of SPSS ver. 19.0 (SPSS Inc., Chicago, IL, USA). One-way ANOVA and Tukey HSD tests were conducted. The data normality was confirmed using Shapiro-Wilk's test, whereas homoscedasticity was established using the homogeneity test. Differences in the hardness values among different aging durations for each material, as well as differences in hardness values among light intensities of 500 mW/cm², 700 mW/cm², and 900 mW/cm² for each material were examined. The significance level for all tests carried out was set at $\alpha = 0.05$.

CONCLUSION

Within the limitations of the study, the following conclusions were drawn:

1. The subsurface hardness of resin-composites showed various changes after immersion in water, which was closely related to their composition and light-curing protocol.
2. As low-shrinkage resin-composites, AD and KA had superior water resistance.
3. For Z2, S2, and AD polymerized by 16 J/cm², the irradiance of 500 mW/cm² was better for subsurface hardness and light-curing effect.

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