Unraveling the Anti-Staining Properties of Dental Resin Cements: A Study with a Newly Developed Universal Resin Cement

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Abstract

Aim
This study examines the anti-staining properties of newly developed and existing dual-cure resin cements for prosthetic dental applications.

Materials and Methods
To replicate a common inlay restoration, a CAD/CAM inlay was machine-milled from dima Hybrid Resin Blank Plus (Kulzer Japan) for a class II cavity abutment tooth made of i-TFC Core Resin (Sun Medical). The inlay and abutment tooth were then cemented using the new universal resin cement (Kulzer Japan ZEN™ Universal Cement) and five existing resin cements (3M RelyX™ Universal Resin Cement, 3M RelyX™ Unicem 2, Ivoclar Vivadent SpeedCem® Plus, GC G-CEM ONE™ EM, and Kuraray Noritake Panavia™ SA Cement Universal). Subsequently, the specimens underwent a 24-hour immersion in red wine at 37°C, after which they were sectioned to evaluate the degree of internal staining. Recognizing that degree of monomer conversion, water sorption and water solubility are potential influencing factors, these properties of each cement were measured and compared with their respective staining tendencies. In addition, the amount of stain compounds was quantified by a new test protocol.

Results and Conclusions
There was a moderate correlation (R^2 = 0.60) between the degree of color change and water solubility, while degree of conversion and water sorption showed no correlation to the degree of color change. This suggests that the leaching of water-soluble components in resin cement may contribute to red wine staining. In addition, there was a strong correlation (R^2 = 0.94) between the amount of stain compounds and the degree of color change, indicating that the extent of staining can be effectively elucidated by a straightforward physical model, without requiring the consideration of intricate chemical interactions between stain molecules and resin composition.

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Keywords

Resin cement, stain resistance, restorative dentistry, aesthetic restoration

INTRODUCTION

Dental resin cement plays a pivotal role in contemporary dentistry by securely bonding restoration materials to prepared teeth, ensuring the functionality and longevity of dental prosthetic restorations (1). Its performance across a spectrum of crucial factors including aesthetic stability, flexural strength, film thickness, radiopacity, water sorption and solubility, and adhesion strength to various surfaces such as dentin, enamel, zirconia, porcelain, lithium disilicate, and precious metals, is paramount for successful dental prosthetic restorations (2–5). Nevertheless, the pursuit of a dental resin cement formulation that accommodates all these diverse and unique properties without significant compromise is still a pressing challenge for dental materials developers.

One of the most critical aspects of resin cement is its resistance to staining, a factor that impacts aesthetic outcomes, particularly at margin lines, and thereby directly influencing patient satisfaction. Recent investigations have shed light on the role of factors such as water sorption and solubility in affecting the stain resistance of dental composite materials. For instance, water sorption was reported to weaken the linkage between the resin matrix and the filler particles, subsequently inducing microcracks or interfacial gaps at the resin–filler interface, contributing to the color alteration of dental composites (6,7). Additionally, the presence of hydrophilic monomers, such as triethylene glycol dimethacrylate, has been implicated in increasing water sorption, and consequently, the extent of staining (8). Water solubility of resin cements were also suggested as a potential contributing factor to staining (6,9), although the specific mechanisms underlying the phenomenon remain unclear.

Parameters such as filler size and amount are also reported to influence the resistance to staining. Recent research has suggested that nanohybrid materials, characterized by lower surface roughness values, may enhance stain resistance in dental composites (8). Yet, the precise analysis of filler content and morphological distribution poses significant challenges for commercial resin cement products, and consequently, controlled experiments with well-characterized filler profiles are necessary.

The degree of monomer conversion has also been recognized as an influential factor affecting staining susceptibility. Studies have indicated that lower degrees of conversion may enhance staining attributed to increased water sorption (10,11).

Building on these previous investigations, this study examines the anti-staining properties of a newly developed resin cement (ZEN™ Universal Cement), engineered to address the multifaceted requirements of contemporary dentistry including long-lasting aesthetic stability. ZEN™ Universal Cement is a universal resin cement, which can be used as a standalone self-adhesive resin cement or can be paired with a 7th generation bonding agent (ZEN™ Universal Bond) for even greater adhesion strength.

To deconvolute potentially influencing factors and their interplay with stain resistance, this research was designed to investigate the degree of monomer conversion, water sorption, and water solubility in relation to the anti-staining properties. Utilizing inlay and disk models, both immersed in red wine – a widely consumed beverages known to cause pronounced staining compared to tea and coffee (8,12) – this study aimed to visualize and quantify staining of mechanisms of ZEN™ Universal Cement and five existing resin cements.

By employing a new test procedure, this research also examines the amount of stain molecules sorbed into resin cement. The objective of this experiment was to determine if there is a statistically significant correlation between the amount of sorbed stain molecules and the degree of staining since such relationship would potentially enable the prediction of the extent of staining through a straightforward physical model, bypassing the need for intricate consideration of chemical interactions or reactions between the stain molecules and the resin composition.

MATERIALS AND METHODS

In this study, the newly developed resin cement (Kulzer Japan ZEN™ Universal Cement) and five existing resin cements (3M RelyX™ Universal Resin Cement, 3M RelyX™ Unicem 2, Ivoclar Vivadent SpeedCem® Plus, GC G-CEM ONE™ EM, and Kuraray Noritake Panavia™ SA Cement Universal) were used to explore the anti-staining characteristics of dental resin cements. The details of the resin cements are described in Table 1.

To replicate a common inlay restoration, a CAD/CAM inlay was machine-milled from dima Hybrid Resin Blank Plus (Kulzer Japan) for a class II cavity abutment tooth made of i-TFC Core Resin (Sun Medical) (Figure 1a). The inlay and abutment tooth were then cemented with the resin cements, where the excess cement was removed using the standard tack-cure procedure with a 1000 mW/cm^2 LED dental curing light (J. Morita PenCure 2000) (Figure 1b), followed by complete light curing through 10-second irradiation (Figure 1c). Subsequently, the specimens underwent a 24-hour immersion in red wine at 37 °C (Figure 1d). Following immersion, specimens were thoroughly rinsed with purified water under sonication for 60 seconds and then sectioned for evaluation of internal staining using a digital microscope (KEYENCE VHX-900) (Figure 1e-f). It should be noted that the oxygen inhibition layer of the cement line was intentionally retained, since in common clinical practices, the oxygen-inhibited layer is typically left intact during the tack cure procedure

Brand	Trade name	Shade	Product code	Composition
Kulzer Japan	ZEN™ Universal Cement (Development code: DP-003)	Translucent	ZEN	10-Methacryloyloxydecyl dihydrogen phosphate, proprietary dimethacrylate, Bioactive Monomer™, 2-hydroxyethyl methacrylate, triethylene glycol dimethacrylate, proprietary initiators, ytterbium trifluoride, silanated barium glass, silanated colloidal silica, inhibitors, stabilizers, pigments
3M	RelyX™ Universal Resin Cement	Translucent	3MRU	Dimethacrylate ester of phosphoric acid, urethane dimethacrylate, triethylene glycol dimethacrylate, 2-hydroxyethyl methacrylate, methyl methacrylate, t-amyl hydroperoxide, L-ascorbic acid 6-hexanoate, copper (II) acetate, silanated vitreous silica, ytterbium trifluoride, titanium dioxide, triphenyl phosphite, butylated hydroxytoluene
3M	RelyX™ Unicem 2	Translucent	3MU2	Dimethacrylate ester of phosphoric acid, 1,12-dodecane dimethacrylate, substituted dimethacrylate, triethylene glycol dimethacrylate, t-butyl peroxy-3,5,5-trimethylhexanoate, sodium persulfate, sodium p-toluenesulfinate, barbiturate, copper (II) acetate, silanated silica, oxide glass chemicals, calcium hydroxide, titanium dioxide
Ivoclar Vivadent	SpeedCem® Plus	Transparent	ISCP	10-Methacryloyloxydecyl dihydrogen phosphate, 1,10-decanediol dimethacrylate, urethane dimethacrylate, triethylene glycol dimethacrylate, polyethylene glycol dimethacrylate, benzoyl peroxide, ytterbium trifluoride
GC	G-CEM ONE™ EM	Translucent	GCOE	10-Methacryloyloxydecyl dihydrogen phosphate, 2-hydroxy-1,3-dimethacryloxypropane, urethan dimethacrylate, α,α-dimethylbenzyl hydroperoxide, diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide 6-tert-butyl-2,4-xylenol
Kuraray Noritake Dental	Panavia™ SA Cement Universal (SA Luting Multi)	Translucent	KNSA	10-Methacryloyloxydecyl dihydrogen phosphate, bisphenol A diglycidylmethacrylate, triethylene glycol dimethacrylate, 2-hydroxyethyl methacrylate, hydrophobic aromatic dimethacrylate, peroxide, accelerators, catalysts,
Notes: Compositional information obtained from manufacturers' safety data sheets. Some expressions may be modified for clarity or conciseness. Not every compositional detail may be included due to space limitations.				

Tab. 1 List of resin cements used in this research.

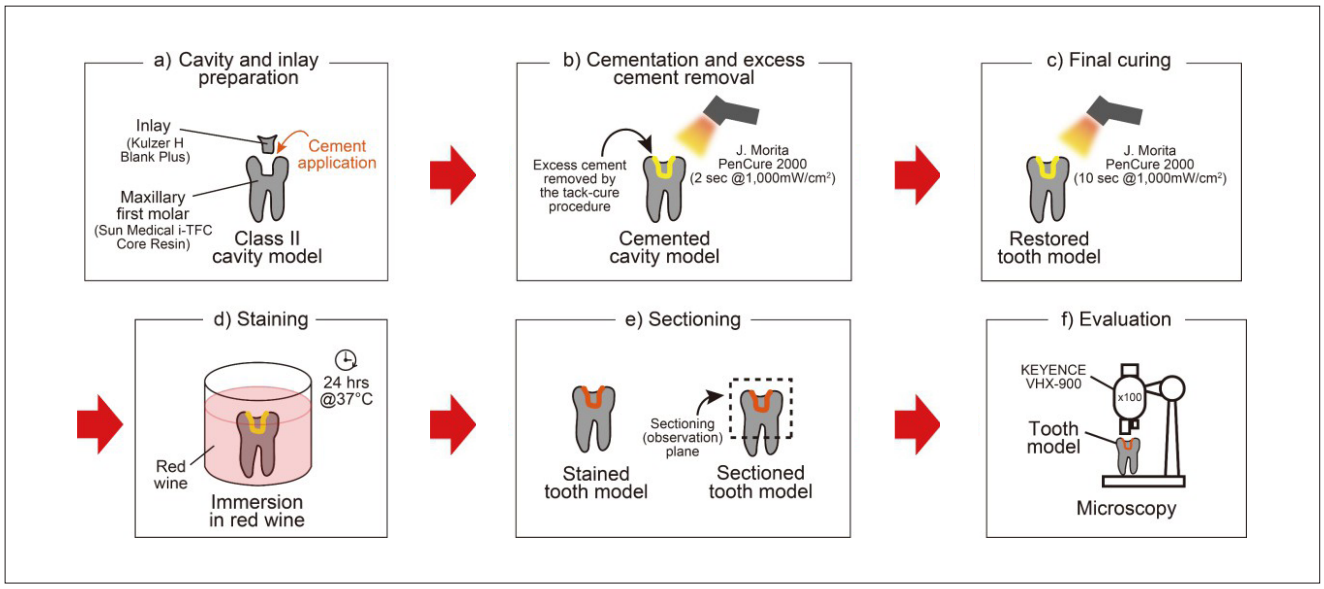

Fig. 1 Preparation and evaluation flow of stained and sectioned tooth models: (a) cavity and inlay models, (b) cementation and excess cement removal), (c) final curing of the resin cement, (d) immersion in red wine, (e) sectioning of the stained tooth model, and (f) evaluation of the degree of internal staining.

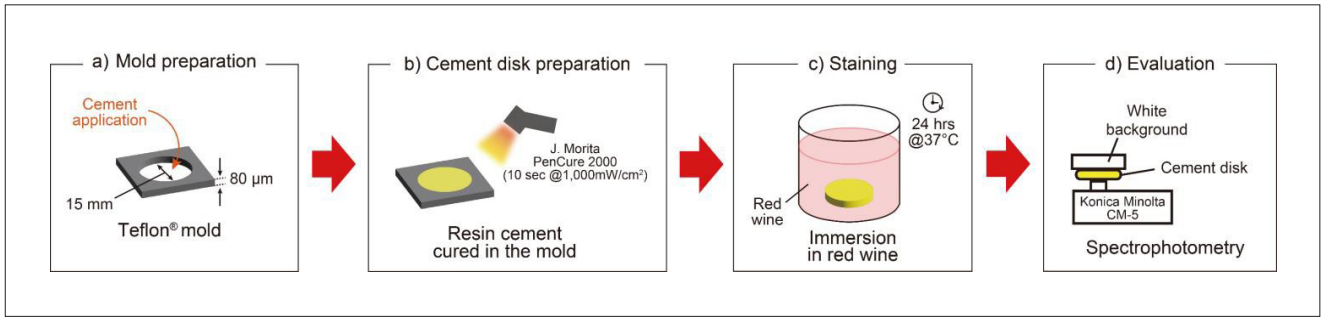

Fig. 2 Preparation and evaluation of stained cement disks: (a) Teflon® mold and application of resin cement, (b) curing of resin cement, (c) immersion in red wine, and (d) evaluation of the degree of staining.

but is known to signify staining (13,14).

Next, given the inherent difficulty in quantifying color changes within the narrow cement lines (< 100 µm), the approach chosen for characterizing the extent of color alteration entailed evaluating cement disks, each possessing a diameter of 15 mm and a thickness of 80 µm. Importantly, it should be noted that assuming the equivalence of color change between the inlay and disk models became necessary for this alternative approach. The preparation of these disks involved curing resin cements within a Teflon® mold, as illustrated in Figure 2a-b.

The assessment procedure unfolded as follows: The cement disks underwent a 24-hour immersion in red wine at 37°C, as illustrated in Figure 2c. Following this staining period, the specimens were removed from the solution, subjected to a thorough rinsing with purified water under sonication for 60 seconds. Subsequently, the color values of the cement disks were measured both before and after immersion in the staining solution, utilizing a spectrophotometer (Konica Minolta CM-5) in the CIELAB color space (Figure 2d). The degree of color change (ΔE*ab) was then calculated by using the formula (15):

$$\Delta E^*ab = \sqrt{(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2}$$

where L^*_1 and L^*_2 are the lightness values of the cement disk before and after staining, respectively; a^*_1 and a^*_2 are the green-magenta opponent color values before and after staining, respectively; and b^*_1 and b^*_2 are the blue-yellow opponent color values before and after staining, respectively.

Recognizing the potential impact of monomer conversion, water sorption, and water solubility on staining tendencies, comprehensive assessments of these properties were conducted for each resin cement and compared accordingly. The degree of monomer conversion was determined using Fourier-transformed infrared spectroscopy (FT-IR) with a Perkin Elmer UATR 2. The resin cement was applied to the sample stage, and the spectra were acquired in the range of 1000-4000 cm^{-1}, serving as the initial data. After exposure to a 1,000 mW/cm^2 LED dental curing unit (J. Morita PenCure 2000) for 10 seconds and a 2-hour waiting period at the room temperature, post-curing data were acquired through another scan. The degree of conversion (DC) was then calculated by using the formula (16):

$$DC = 100 \times \left[\frac{CC_2/CO_2}{CC_1/CO_1}\right]$$

Here, CC1 and CC2 represent the peak intensities of aliphatic C=C bonds before and after curing (1634 cm^{-1}), reflecting the polymerization of methacrylate double bonds. CO_1 and CO_2 denote the peak intensities of carbonyl C=O bonds before and after curing (1717 cm^{-1}), providing a reference band for spectra normalization (16). The correlation between DC and the extent of staining (ΔE*ab) was then tested using a commercially available statistical analysis software (StatCrunch®) with a simple linear regression model.

The measurements of the water sorption and solubility of the resin cements were performed according to the ISO standard 4049:2019 (Figure 3), with a sample size of 5. In short, bone-dry dual-cured disk samples, each with a diameter of 15 mm and a thickness of 1 mm, were immersed in 10 mL of purified water at 37°C. After 7 days, the samples were removed from water and dried with absorbent paper, and placed in a desiccator until the samples acquired a stable weight. Water sorption and solubility values (µg/mm^3) for each resin cement were then calculated using the formulas (5):

$$WS = \frac{m_2 - m_3}{V}$$

$$WSL = \frac{m_1 - m_3}{V}$$

where m_1 is the initial, bone-dry mass; m_2 is the

mass post-sorption; and m_3 is mass after the final dry. The correlation between water sorption and staining (ΔE*ab), and between water solubility and staining, was respectively assessed using StatCrunch® with simple linear regression models. To further explore the combined influence of multiple factors on staining, multiple regression analysis was conducted to investigate the interplay between degree of conversion, water sorption, water solubility, and staining.

Another critical parameter is the extent to which stain molecules are sorbed into resin cement. By assessing this sorption, one can draw an important conclusion: if there is a statistically significant correlation between the quantity of stain molecules sorbed into the resin cement and the degree of staining, it suggests that the extent of staining can be predicted using a simple physical model, without requiring the consideration of intricate chemical interactions or reactions between the stain molecules and the resin composition.

Based on the assumption that red wine is composed of volatile (e.g., solvent) and non-volatile (stain) compounds, the quantification of stain molecules was conducted in the following manner. First, five dual-cured disk specimens of each resin cement, prepared in accordance with the ISO 4049:2019 standard for water sorption and solubility, were completely dried and then immersed in 10 mL of red wine at 37°C. After 7 days, the samples were extracted from red wine and rinsed with purified water for 60 seconds under sonication, followed by drying with absorbent paper. Subsequently, the samples were placed in a desiccator until a stable weight was achieved, in accordance with the requirements outlined by the ISO 4049:2019 standard for water sorption and solubility. The apparent dissolution (RLS_{ISO}) was then calculated using the formula:

$$WSL = \frac{m_1 - m_4}{V}$$

where m_1 is the initial (bone-dry) mass, and m_4 is the mass after the final drying process.

As illustrated in Figure 4, the relationship between this apparent dissolution and the amount of sorbed stain compounds (S) can be expressed as:

$$RSL = RSL_{ISO} + S$$

where RLS represents the actual dissolution into red wine. Now, by assuming that the type of solvent, whether purified water or red wine, does not significantly influence the dissolution from resin cements since both solvents can be categorized as polar solvents with water as their main components, then:

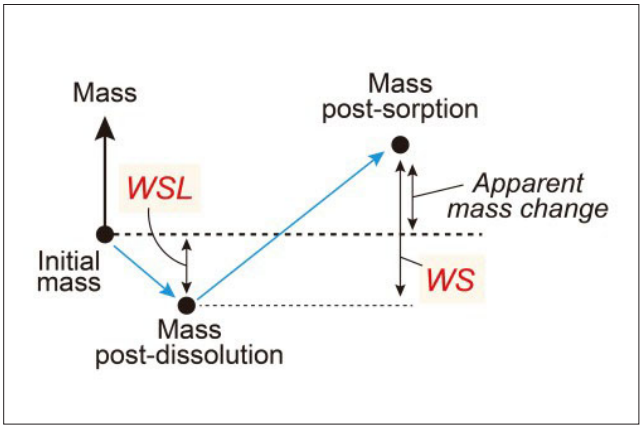

Fig. 3 Water sorption and solubility as defined by the ISO standard 4049:2019.

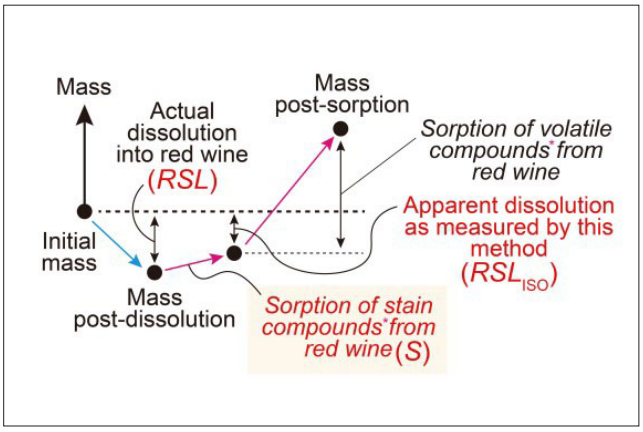

Fig. 4 Quantification of the stain molecule (S) sorbed into resin cement.

$$RSL \sim WSL$$

Therefore, Equation (6) can be rewritten as:

$$WSL = RSL_{ISO} + S$$

$$S = WSL - RSL_{ISO}$$

Hence, by using Equation (8), the amount of stain molecules sorbed into resin cement was calculated, and the correlation with the extent of staining was tested using StatCrunch® with a simple linear regression model.

RESULTS AND DISCUSSION

The fabricated inlay models are given in Figure 5. The cement lines of these inlay models were nearly invisible even under magnification. Figure 6 displays cross-sectional views of the inlay models, further confirming this imperceptible nature of the cement

lines in untreated inlay models.
Inlay models after red wine immersion are shown in Figure 7. After immersion in red wine, the existing resin cements showed varying degree of staining, while the new resin cement (ZEN) remained unstained. From the cross-sectional observation (Figure 8), it was found that the stain tends to progress inwards, implying a significant transport of stain compounds within the existing resin cements.
The disk samples before or after immersion in red wine are shown in Figure 9. As quantified by the spectrophotometer (Figure 10), it was evident that exposure to red wine significantly increased Δb* in the most existing resin cements, except for 3MU2, thereby deteriorating their overall appearance (ΔE*ab). In contrast, the new resin cement (ZEN) exhibited the smallest Δb* as well as Δa*, resulting in the minimum impact on the overall aesthetic appearance (ΔE*ab).
The measured values for the degree of conversion, water sorption, and solubility of each resin cement are provided in Table 2. From the statistical test results visualized in Figure 11, it turned out that while the degree of conversion and water sorption did not show a correlation with the extent of staining, respectively, water solubility exhibited a mild correlation (= 0.60) with the staining tendencies.
Next, the multiple linear regression analysis revealed that the R^2 value was not significantly improved R^2 value was not significantly improved (R^2 = 0.62 with adjusted R^2 = 0.07) even when all the three independent variables (DC, WS and WSL) were considered, indicating that the additional terms (DC and WS) does not contribute to the explanation of the extent of staining in resin cements.
Therefore, it is concluded that enhancement of the surface area due to leaching of water-soluble

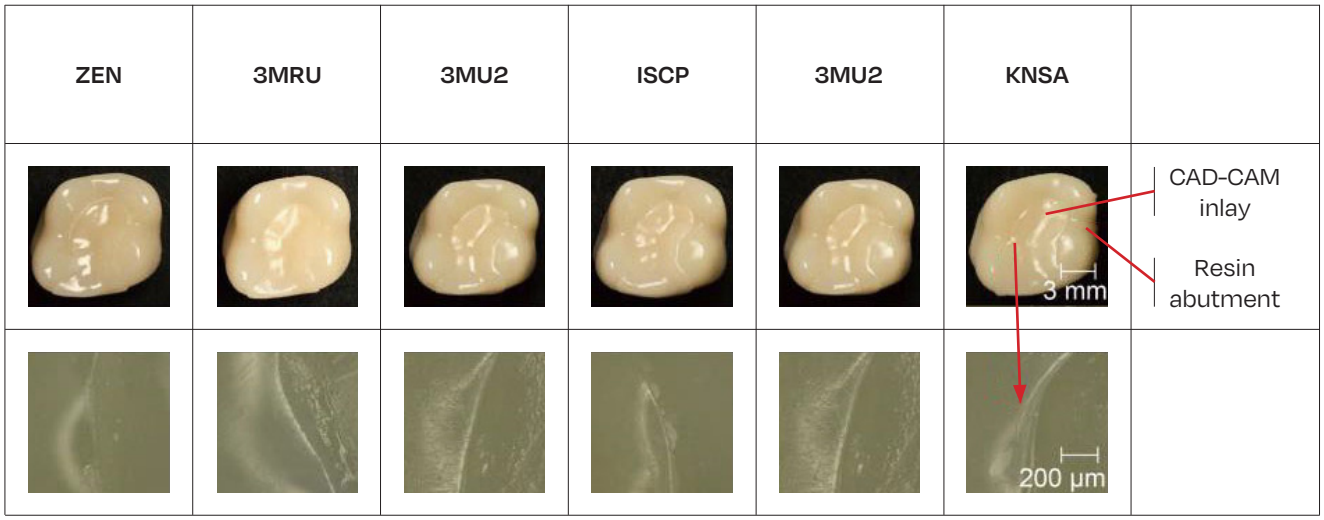

Fig. 5 Inlay models cemented with the resin cements (occlusal plane).

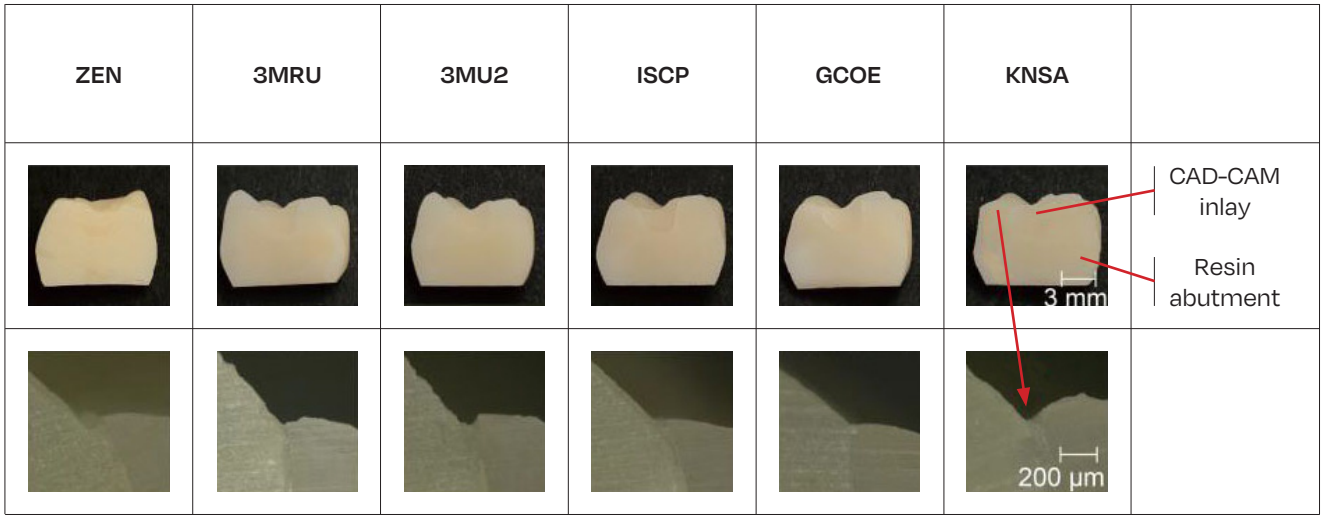

Fig. 6 Crosscuts of the inlay models given in Figure 6 (transverse plane).

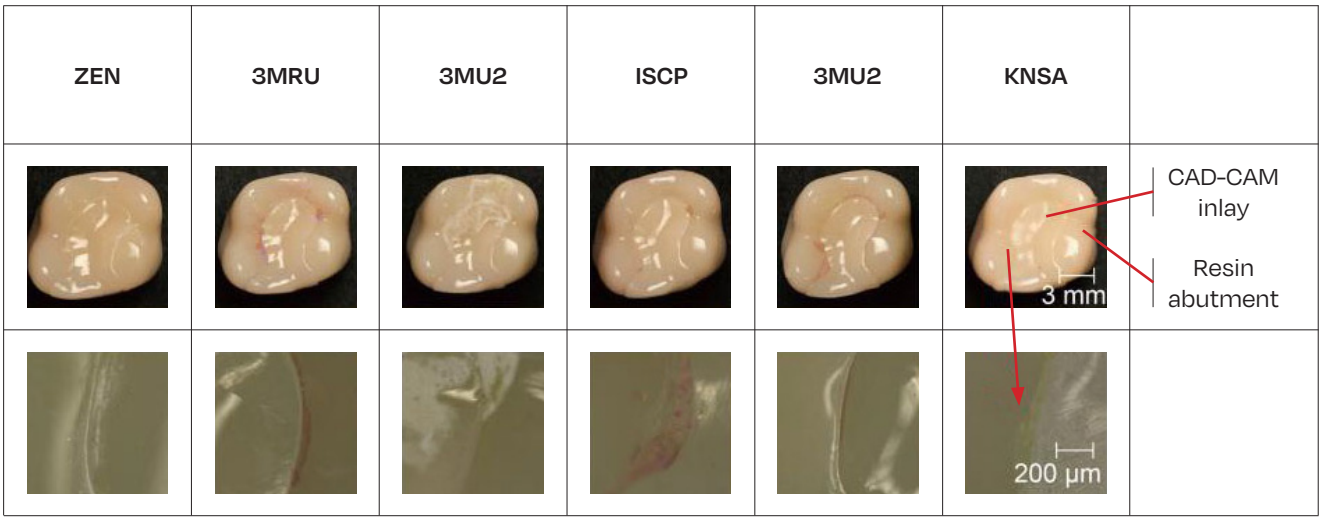

Fig. 7 Inlay models after immersion in red wine, showing varying degrees of staining (occlusal plane).

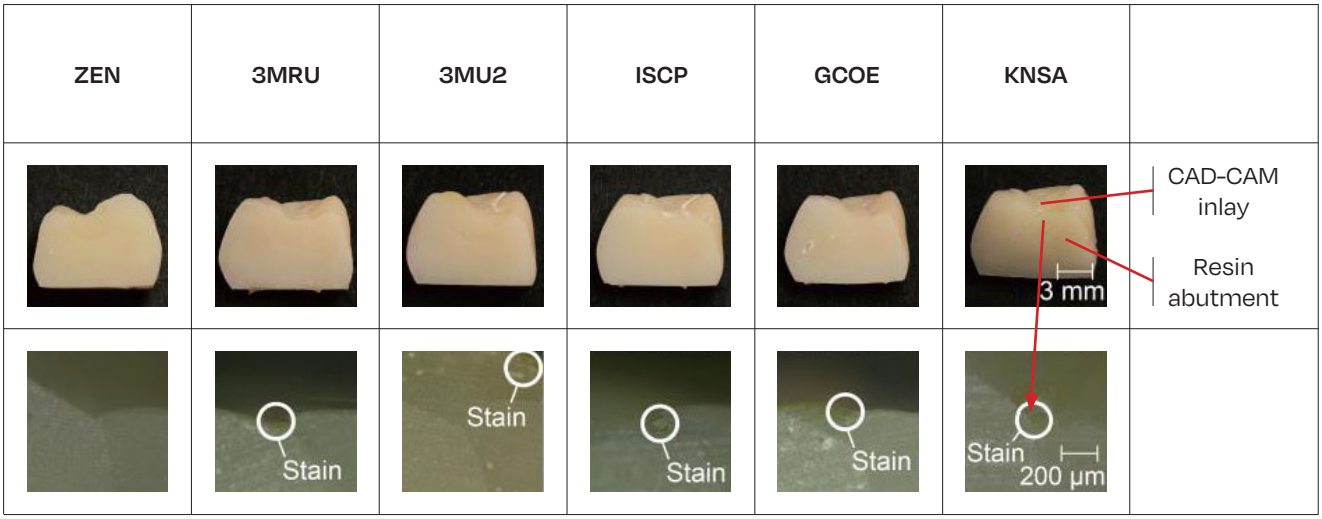

Fig. 8 Crosscuts of inlay models given in Figure 8 (transverse plane).

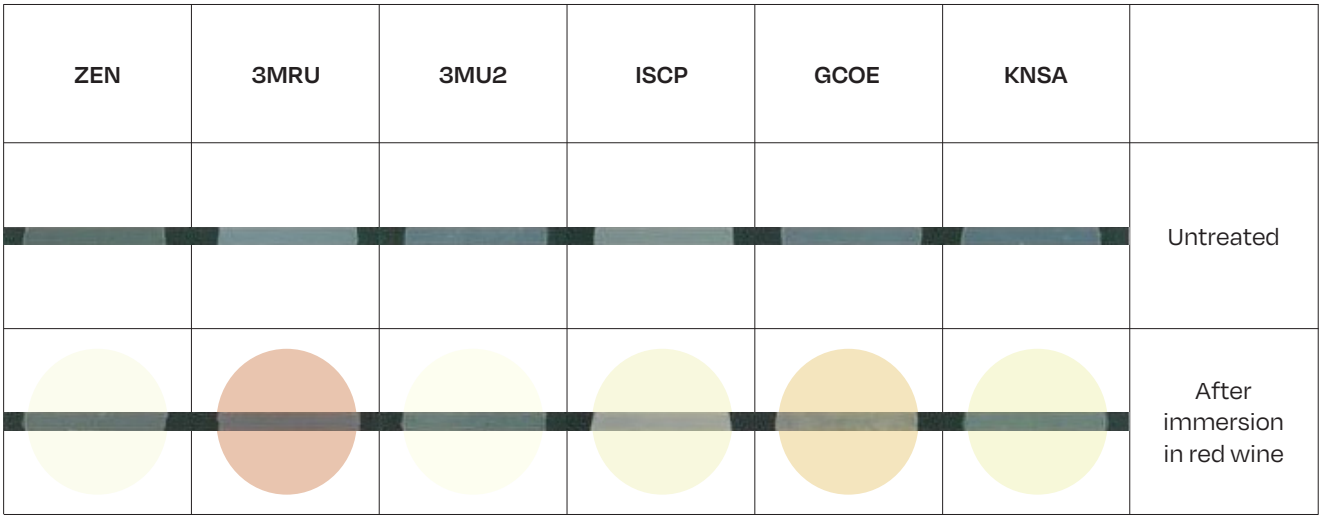

Fig. 9 Untreated and stained cement disks prepared for spectrophotometry.

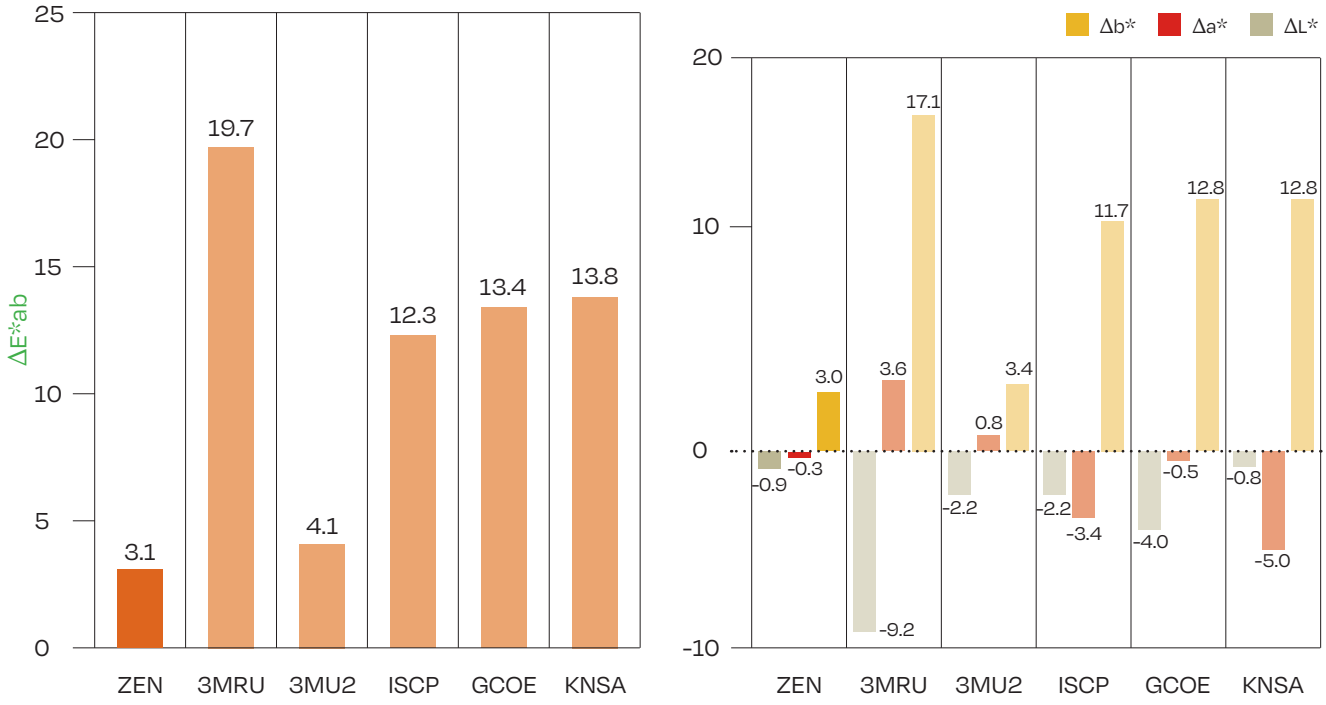

Fig. 10 Color changes of each resin cement quantified in the CIELAB color space: (a) ΔE*ab and (b) ΔE*, a* and b*, as measured by the spectrophotometer.

Product code	ΔE*ab	DC (%)	WS (μg/cm³)	WSL (μg/cm³)
ZEN	3.1	61.4	36.6	1.6
3MRU	19.7	63.6	42.0	4.4
3MU2	3.9	48.2	31.6	0.5
ISCP	12.3	73.8	32.9	3.1
GCOE	13.4	61.1	29.3	1.8
KNSA	13.8	57.0	32.6	5.2
Note: The water sorption and water solubility values shown here represent the averages of five specimens.				

Tab. 2 Measured values for the degree of conversion, water sorption and water solubility of each resin cement.

components (e.g., monomers, initiators, and other additives) leads to more pronounced staining in the scheme depicted in Figure 12. It is worth noting that models such as those represented in Figure 13 is unlikely, given that the inclusion of additional terms (DC and WS) did not enhance the model's accuracy, as confirmed by the multiple linear regression analysis.
It is noteworthy that previous studies have underscored the significance of both the degree of monomer conversion and water sorption for the extent of staining. However, within the scope of this research, these variables did not emerge as primary influencers. Regarding the degree of monomer conversion, while this parameter holds potential to impact stain

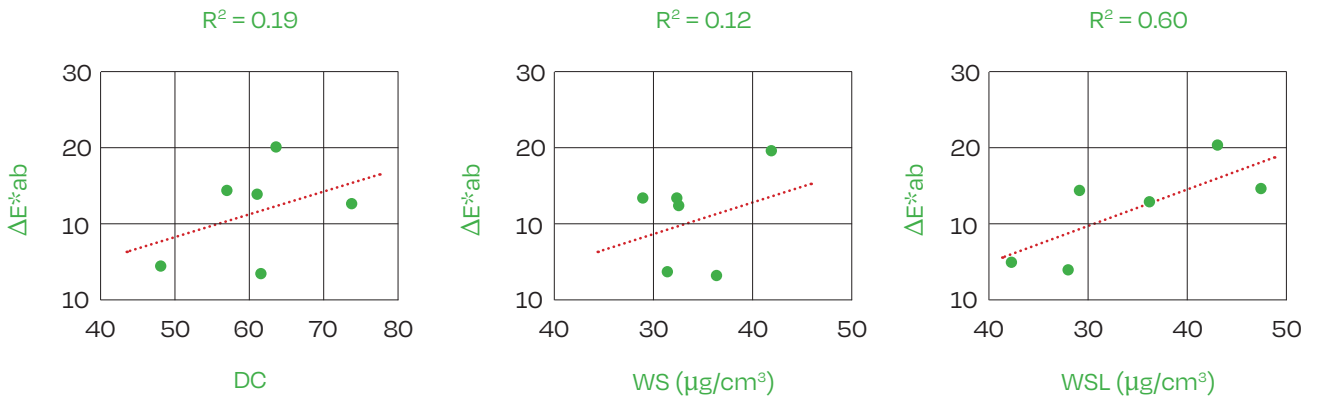

Fig. 11 Simple linear regression analysis results for the degree of monomer conversion, water sorption and water solubility as independent variables.

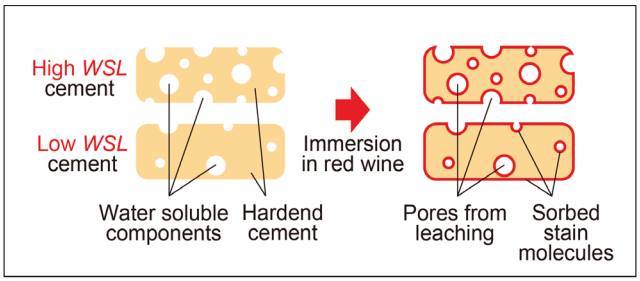

Fig. 12 Possible staining scheme for dental resin cements.

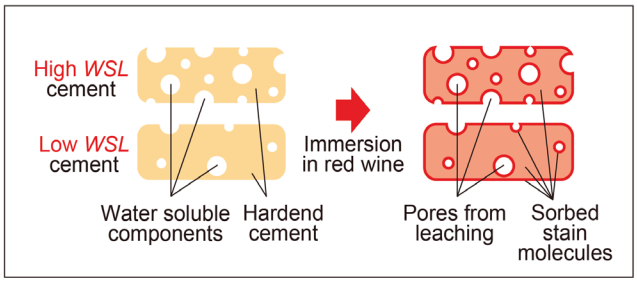

Fig. 13 Improbable staining mechanism, where WSL is the primary factor for staining, with WS and DC as secondary factors affecting stain penetration (diffusion) into the resin matrix.

Product code	ΔRSL_{ISO} (μg/cm³)	S (μg/cm³)
ZEN	0.0	1.6
3MRU	−1.4	5.8
3MU2	−1.8	2.3
ISCP	−0.7	3.8
GCOE	−3.3	5.1
KNSA	−1.0	4.2
Note: The values are the averages of five specimens.		

Tab. 3 The amounts of apparent dissolution and sorbed stain compounds of each resin cement.

resistance, the diverse monomer profiles inherent in the investigated resin cements—varying in reactivity, hydrophilicity, and hydrophobicity—were not controlled for. Consequently, the expected correlation between the degree of monomer conversion and staining was not observed.

As for water sorption, it is probable that even if similar levels of water sorption are present in two resin cements, the accessibility of stain molecules (such as anthocyanins and tannins (17)) - which are notably larger than water molecules - could behave differently during diffusion through polymer networks. This behavior could be, for instance, affected by the chemical properties of the polymer networks including the functional groups and cross linkers, potentially leading to a lack of statistical significance between water sorption and the extent of staining when resin cements of dissimilar monomer compositions are compared. These complexities underline the necessity for further exploration to elucidate the relationship between these variables with resin cements controlled for their polymer network properties.

The amount of apparent dissolution (RSL_{ISO}) from the resin cements, as well as the amount of sorbed stain compounds (S) by the resin cements are given in Table 3. As plotted in Figure 14, the simple linear regression analysis yielded a high R^2 value of 0.94, indicating a strong positive correlation between the amount of sorbed stain molecules and the degree of color change (ΔE*ab). This suggests that the extent of staining can be effectively elucidated by a straightforward physical model, without requiring the consideration of intricate chemical interactions between stain molecules and the resin composition.

CONCLUSIONS

This study investigated the stain resistance of various resin cements for dental prosthetic applications. While conventional resin cements exhibited varying degrees

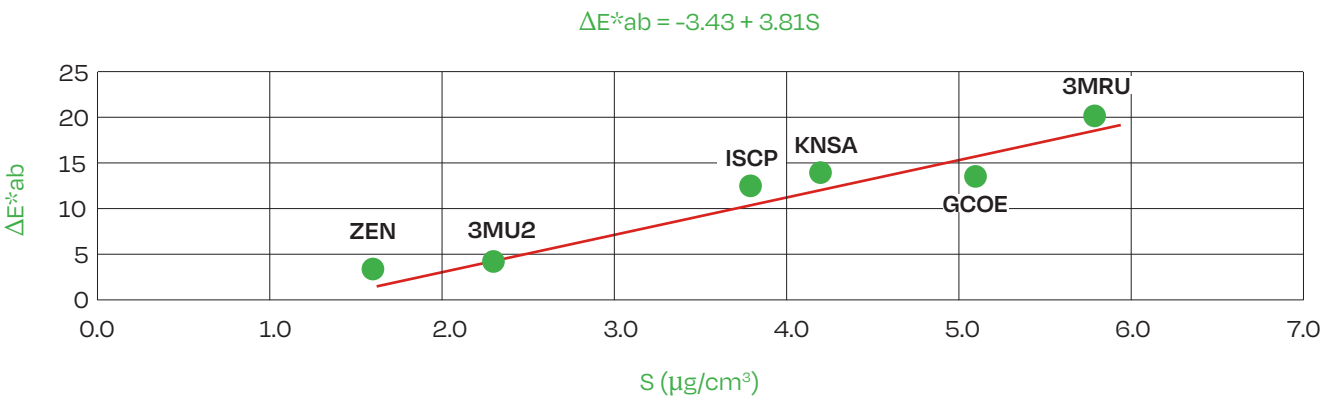

Fig. 14 Amount of sorbed stain compounds plotted against the degree of staining, indicating a strong positive correlation (R^2 = 0.94).

of staining upon exposure to red wine, resulting in compromised aesthetic outcomes, ZEN™ Universal Cement demonstrated minimal color alteration (ΔE*ab). This emphasizes the importance of selecting resin cement with robust anti-staining properties to achieve long-term aesthetic success and ensure the highest level of patient satisfaction.

Statistical analysis unveiled that while factors such as monomer conversion and water sorption did not directly correlate with the extent of staining, the presence of water-soluble compounds significantly influenced the anti-staining properties. Notably, ZEN™ Universal Cement shows minimal water solubility and thus least discoloration. Moreover, a strong positive correlation (R^2 = 0.94) was observed between the amount of stain molecules sorbed into resin cements and the level of staining, suggesting that the extent of color change can be well-predicted by a straightforward (linear) relationship. These findings suggest that the use of low-water-soluble and anti-staining resin cements has considerable advantages for the future of aesthetic dentistry.

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Conflicts of Interest

The authors are employees of Sun Medical Co., Ltd.

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