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Article

Clinical Performance and Bonding Mechanism of Three Resin Adhesives

Kun Wu

Department of Gastrointestinal Surgery, The Affiliated Huai'an No.1 People's Hospital of Nanjing Medical University, Huai'an 223300, China; drKunwu@163.com

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Abstract: Juxtaposed surfaces could be bond to achieve marginal sealing and adhesive of interface between different kinds of substrate. Therefore, the purpose of the current study is to investigate the bonding strength of three common resin adhesives after bonding and polymerization, and to evaluate the bonding effect. PanaviaTM-F resin adhesive, Rely 3M EPSE resin adhesive, and Kerr NX3 resin adhesive were used to prepare modules ($10 \times 10 \times 3$ mm^3). The glass permeable ceramic was made into a rectangular component ($10 \times 10 \times 10$ mm^3), ensuring all surfaces to be smooth. Three kinds of different adhesives were bonded to surface of the glass-infiltrated ceramic. Tensile tests, compression tests and shear tests were performed on different adhesives after cold and hot cycles to comprehensively evaluate the differences in the clinical properties of adhesives. After testing, the surface hardness of Kerr NX3 resin adhesive was the highest among the three, and less affected by water storage. The tensile strength and compressive capacity of Kerr NX3 resin were much stronger than those of the other two adhesives. After cold and hot cycles, Kerr NX3 resin was 39% higher than Panavia TM-F resin and 15% higher than Rely 3M EPSE resin. Scanning Electron Microscopy (SEM) observations of morphology and failure surfaces of three adhesives showed that the repairing effect of Kerr NX3 resin was the best and the bonding strength was the highest. Compared with the PanaviaTM-F and Rely 3M EPSE resin, the bonding strength of Kerr NX3 resin was the highest with best repairing effect.

Keywords: bonding strength; compression test; resin bonding; shear force; surface-dimensional hardness, tensile test

1. Introduction

Dental caries, also known as tooth decay or cavities, remains a major health concern worldwide [1]. In elderly people, edentulism is an important indicator of dental caries and a 'final marker of disease burden for oral health' [2]. In the treatment of dental caries, prosthodontics is a unique dental profession that combines aesthetics, philosophy and sciences, including reversible and irreversible treatments [3]. Wear resistance is one of the most important properties for choosing dental restorative materials [4]. The wear properties of restorative materials are very complicated, which are affected by a variety of factors, including wear of polymer matrix, loss of filler by failure of its bond with the matrix, shear of filler particle, cohesive failure through matrix, and exposure of air bubbles [5]. Glass ceramic is a relatively common dental material that is used as dental inlay, onlay, crown or other structures to fix dental problems [6]. However, it has been stated that while ceramic materials are resistant to compressive forces, they are susceptible to tensile stresses and more prone to fracture than composite materials [7]. In addition, etching silica-based ceramics with hydrofluoric acid produce insoluble by-products consisting of silica fluoride salts on the surface, and the remaining by-products can disrupt the bond strength of the resin [8]. Today, the utilization of new materials has made it feasible to fabricate all-ceramic prosthodontic restorations with superior esthetics [9].

Resin cement has been the pinnacle of direct esthetic restorations long since its discovery [10]. Resin cement is most commonly composed of bisphenol A-glycidyl methacrylate (Bis-GMA) and other dimethacrylate monomers, a filler material such as silica and in most current applications, a photoinitiator. A direct resin composite restoration based on resin cement adhesives can protect the integrity of tooth substance and achieve esthetic restoration, which significantly contributes to the realization of minimally invasive dental treatments [11]. In order to improve the aesthetic level of the final restoration, the resin composite veneer must have certain physical strength and resistance to abrasion [12]. Self-adhesive resin cement Panavia^{TM}-F is a catalyst promoting the bonding of the ceramic [13]. Self-adhesive Rely 3M EPSE is proved to have more advantages in its aesthetic properties and strength [14]. In addition, the adhesive strength of Kerr NX3 resin cement is relatively strong and can meet the clinical requirements [15]. There is evidence regarding the effect of the thickness of resin cements on the polymerization shrinkage stress [16,17], which showed that different types of resin cements play important roles in cementing ceramic restorations [15]. However, in current literature, there are few studies that compare the bonding strength and restorative effect of Panavia^{TM}-F, Rely 3M EPSE and Kerr NX3. Therefore, in this study, the bonding strength and restorative effect of the above three resin cements were evaluated by measuring their tension, compression and shear strength.

2. Material and Methods

2.1. Materials

Each of 60 components ($10 \times 10 \times 3$ mm^3) was prepared from three different adhesives (Kerr NX3, Panavia™-F and Rely 3M EPSE). The surface was demanded to be smooth and the surface roughness (Ra) with no more than 0.10 μm. Twenty components ($10 \times 10 \times 10$ mm^3). Smooth surface were obtained from glass-infiltrated ceramic. Under a stereoscope, the surface structure of each component was uniform. According to the manufacturer's instructions, 20 adhesive composites ($10 \times 10 \times 13$ mm^3) were obtained from the three kinds of resin cements bonding to the glass-infiltrated ceramics.

2.2. Grouping

The prepared Panavia™-F was designated as group A1; Rely 3M EPSE was designated as group A2; and Kerr NX3 was designated as group A3. Besides, Panavia™-F bonding to glass-infiltrated ceramic was selected as group B1; Rely 3M EPSE bonding to glass-infiltrated ceramic was selected as group B2; and the Kerr NX3 bonding to glass-infiltrated ceramic was selected as group B3.

2.3. Vickers Hardness (HV) Test

Each of five components were randomly selected from group A1, A2 and A3 and independently immersed in distilled water for 24 h, 1 week, 2 weeks, 4 weeks and 12 weeks. Then the HV value of each group was calculated.

2.4. Tensile Testing

Five components were randomly selected from group A1, A2 and A3. The components were sliced vertically along its longitudinal axis using low-speed saw in cooling water, to prepare a specimen with a length of 10 mm and cross-sectional area 3×3 mm^2. The micro-tensile bond strength of each specimen was measured using a universal testing machine (UTM). Furthermore, 5 components were randomly selected from group B1, B2 and B3. Then the components were sliced vertically along its longitudinal axis using low-speed saw in cooling water, to prepare a specimen with a length of 13 mm and cross-sectional area 3×3 mm^2. The micro-tensile bond strength of each specimen was tested using a UTM.

2.5. Compression Test

Five components were randomly selected from group A1, A2 and A3. The compressive strength was measured using a UTM at a crosshead *speed* of 1 mm/min. Moreover, five components were randomly selected from group B1, B2 and B3. The compressive strength was measured using a UTM and the parameter condition was consistent with group A.

2.6. Thermal-Cold Cycling and Shear Strength Testing

Each of five components were randomly selected from group A1, A2 and A3, which were sliced vertically along its longitudinal axis using a low-speed saw in cooling water, to prepare a specimen with a length of 10 mm and cross-sectional area 5×5 mm^2. Additionally, five monomers were randomly chosen from group B1, B2 and B3. The specimen with a length of 13 mm and cross-sectional area 5×5 mm^2 was obtained by the same way. All specimens were placed in the thermal shock chamber. And the specimens from the six groups were immersed in water bath and then were thermocycled at 5°C and 55°C for 5000 cycles, 60 seconds for each cycle. Next, the shear strength of the samples was calculated using a UTM at a crosshead *speed* of 0.5 mm/min.

2.7. Scanning Electron Microscopy (SEM)

Five components were randomly selected from group B1, B2 and B3 and were sectioned perpendicularly to the bonded surface to expose the adhesive interface to obtain 1 mm specimens. The specimens were subsequently wet-polished using 500 to 2000 grit waterproof abrasive paper. The ultrastructure of bonded surface in group B1, B2 and B3 were observed by SEM. Moreover, all specimens were rinsed with distilled water and placed in an ultrasonic bath for several minutes and were dried by gentle blotting using absorbent paper (Kimwipes; Kimberly-Clark Professional, Roswell, GA, USA). After gold sputtering, the specimens were viewed using SEM. Next, specimens were selected from group B1, B2 and B3 after the shear testing. The damaged surface of resin cements bonding to glass-infiltrated ceramic was observed by SEM.

2.8. Statistical Analysis

All the data were processed using SPSS 19.0 statistical software (SPSS Inc., Chicago, IL, USA). The homogeneity of variance was examined using the shear and bonding strength of the three resin composites. Statistical analysis was performed using analysis of variance test. Comparisons between paired groups were performed using Last Significant Difference (LSD) tests for two groups, and the data were presented as mean ± standard deviation. A p value of < 0.05 was considered statistically significant.

3. Results

3.1. Kerr NX3 Possesses Higher HV

Initially, HV test was adopted to explore the HV of resin. As shown in Table 1, cement types and the water storage time had a significant effect on the HV. Specifically, the HV of components in the A1 group was not affected by water storage time. Then, the HV of the components in the A2 group was decreased after long-term water immersion. Furthermore, the components in the A3 group showed significantly higher HV and were less affected by water storage time. $p < 0.05$ indicated statistically significant. These results demonstrated that HV of Kerr NX3 is higher and less affected by water storage time.

Table 1. The Vickers hardness (HV) of Panavia^{TM}-F, Rely 3M EPSE and Kerr NX3

Resin cement	**24 h/HV**	**1 w/HV**	**2 w/HV**	**4 w/HV**	**12 w/HV**
Panavia^{TM}-F	46.32 ± 4.45	45.67 ± 4.21	46.01 ± 3.89	44.56 ± 3.96	43.82 ± 4.45
Rely 3M EPSE	49.54 ± 4.37	42.91 ± 3.84	40.35 ± 1.14*	37.72 ± 1.09*	34.81 ± 1.35*
Kerr NX3	53.27 ± 1.78*	50.82 ± 5.29$^{\triangle}$	49.56 ± 1.67$^{\triangle}$	48.78 ± 1.74$^{\triangle}$	47.96 ± 2.01$^{\triangle}$

Notes: h, hour; w, week; HV, Vickers hardness; *, $p < 0.05$ compared with Panavia^{TM}-F; $^{\triangle}$, $p < 0.05$ compared with Rely 3M EPSE.

3.2. Kerr NX3 Possesses Increased Tensile Strength

A UTM was employed to measure micro-tensile bond strength. In addition, the side length of each sample was measured by a vernier caliper and the cross-sectional area (mm^2) was calculated. The micro-tensile bond strength (MPa = N/mm^2) was derived by dividing the imposed force (N) at the time of fracture. The results of tensile testing showed that, compared with resin cements bonding to glass-infiltrated ceramic in group B1, B2 and B3, pure resin material in group A1, A2 and A3 possessed a better tensile strength. Moreover, the micro-tensile bond strength of the specimens in group A3 and B3 were significantly higher than that in the other two categories of groups (all $p < 0.05$, Table 2). Thus, tensile strength is better in Kerr NX3.

Table 2. The micro-tensile bond strengths of Panavia^{TM}-F, Rely 3M EPSE and Kerr NX3 in the six groups (n = 5)

	Panavia^{TM}-F	Rely 3M EPSE	Kerr NX3
	(A1, B1)/MPa	(A2, B2)/MPa	(A3, B3)/MPa
Resin cement	167.90 ± 15.69	212.77 ± 22.13*	297.88 ± 11.53$^{*\triangle}$
Resin cement bonding to glass-infiltrated ceramic	128.60 ± 13.01$^{\#}$	176.07 ± 13.63$^{*\#}$	265.58 ± 21.85$^{*\triangle\#}$

Notes: A1, Panavia^{TM}-F; A2, Rely 3M EPSE; A3, Kerr NX3; B1, Panavia^{TM}-F bonding to glass-infiltrated ceramic; B2, Rely 3M EPSE bonding to glass-infiltrated ceramic; B3, Kerr NX3 bonding to glass-infiltrated ceramic; *, $p < 0.05$ compared with A1 and B1; $^{\triangle}$, $p < 0.05$ as compared with A2 and B2; $^{\#}$, $p < 0.05$ compared with the pure resin materials in the homogeneous group.

3.3. Kerr NX3 Yields Increased Compressive Strength

Compressive strength was measured by compression test using a UTM. As shown in Table 3, a pairwise comparison for obtained data was considered statistically significant ($p < 0.05$). Pure resin material in group A1, A2 and A3 showed a higher compressive strength than resin cements bonding to glass-infiltrated ceramic in group B1, B2 and B3； the specimens in group A3 and B3 showed a higher compressive strength than that in the other two categories of groups. All above results implied that Kerr NX3 has better compressive strength.

Table 3. The compressive strength of Panavia^{TM}-F, Rely 3M EPSE and Kerr NX3 in the six groups

	Panavia^{TM}-F	Rely 3M EPSE	Kerr NX3
	(A1, B1)/N	(A2, B2)/N	(A3, B3)/N
Resin cement	4269.90 ± 375.69	5566.77 ± 262.13*	6813.88 ± 321.53$^{*\triangle}$
Resin cement bonding to glass-infiltrated ceramic	3488.60 ± 439.01$^{\#}$	4820.07 ± 273.63$^{*\#}$	6025.58 ± 321.85$^{*\triangle\#}$

Notes: A1, Panavia^{TM}-F; A2, Rely 3M EPSE; A3, Kerr NX3; B1, Panavia^{TM}-F bonding to glass-infiltrated ceramic; B2, Rely 3M EPSE bonding to glass-infiltrated ceramic; B3, Kerr NX3 bonding to glass-infiltrated ceramic; *, $p < 0.05$ as compared with A1 and B1; $^\triangle$, $p < 0.05$ as compared with the A2 and B2 groups; $^\#$, $p < 0.05$ compared with the pure resin materials in the homogeneous group

3.4. Kerr NX3 Offers Shear Strength

Shear strength of Panavia^{TM}-F, Rely 3M EPSE and Kerr NX3 was measured by thermal-cold cycling and a UTM. As shown in Table 4, the specimens in group A1 and B1 had relatively low shear strength. However, the specimens in group A3 and B3 showed significantly higher shear strength than that in the other two categories of groups, and there was significant difference between the homogeneous groups ($p < 0.05$), demonstrating that shear strength is higher in Kerr NX3.

Table 4. The shear strength of Panavia^{TM}-F, Rely 3M EPSE and Kerr NX3 in the six groups

	Panavia^{TM}-F (A1, B1)/MPa	Rely 3M EPSE (A2, B2)/MPa	Kerr NX3 (A3, B3)/MPa
Resin cement	26.40 ± 1.99	31.85 ± 2.48*	36.83 ± 3.23$^{*\triangle}$
Resin cement bonding to glass-infiltrated ceramic	22.97 ± 1.52$^\#$	27.69 ± 3.10$^{*\#}$	32.05 ± 2.16$^{*\triangle\#}$

Notes: A1, Panavia^{TM}-F; A2, Rely 3M EPSE; A3, Kerr NX3; B1, Panavia^{TM}-F bonding to glass-infiltrated ceramic; B2, Rely 3M EPSE bonding to glass-infiltrated ceramic; B3, Kerr NX3 bonding to glass-infiltrated ceramic; *, $p < 0.05$ as compared with A1 and B1; $^\triangle$, $p < 0.05$ as compared with A2 and B2; $^\#$, $p < 0.05$ compared with the pure resin materials in the homogeneous group.

3.5. Properties and Adhesive Strength Were Higher in Kerr NX3

To explore properties and adhesive strength of Panavia^{TM}-F, Rely 3M EPSE and Kerr NX3, SEM was used to observe their morphology. As shown in Figure 1C, the bonding interface of Kerr NX3 bonding to glass-infiltrated ceramic was found to be intact and without any voids, while the relatively large cracks, as well as long and dense resin tags were observed in Panavia^{TM}-F bonding to glass-infiltrated ceramic and Rely 3M EPSE bonding to glass-infiltrated ceramic. The results of interface damage were shown in Table 5. The predominant failure mode of the three kinds of resin cements bonding to glass-infiltrated ceramic was quasi-cleavage fracture (53.33%) and only 26.67% samples showed adhesive failure. And the failure mode of the glass infiltrated ceramics was the least, indicating that the cohesive force of the three kinds of resin cements was lower than that of the glass-infiltrated ceramic. In addition, Kerr NX3 showed no evidence of self-broken, which indicated that better properties and adhesive strength may be possessed by Kerr NX3.

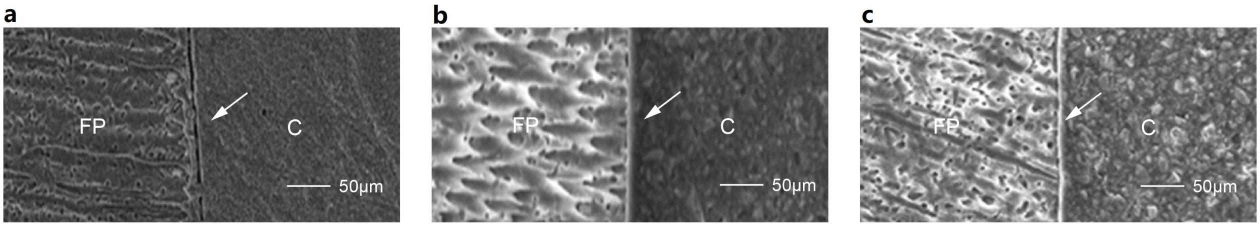

Figure 1. Morphology of Panavia^{TM}-F, Rely 3M EPSE and Kerr NX3 in the B1, B2 and B3 groups was observed by SEM. Notes: **a**, morphology of Panavia^{TM}-F; **b**, morphology of Rely 3M EPSE; **c**, morphology of Kerr NX3; the white arrows indicated cracks and the black arrows indicated resin tags in Panavia^{TM}-F; the white letter C indicated the resin cement and the letters FP indicated the glass-infiltrated ceramics; the magnification of the SEM was ×500; B1, Panavia^{TM}-F bonding to glass-infiltrated ceramic; B2, Rely 3M EPSE bonding to glass-infiltrated ceramic; B3, Kerr NX3 bonding to glass-infiltrated ceramic; SEM, scanning electron microscopy.

Table 5. The morphological observation of Panavia^{TM}-F, Rely 3M EPSE and Kerr NX3 in the B1, B2 and B3 groups by SEM (n = 5)

	B1/MPa	**B2/MPa**	**B3/MPa**
Type 1	1	1	0
Type 2	0	1	0
Type 3	2	1	1
Type 4	2	2	4

Notes: B1, Panavia™-F bonding to glass-infiltrated ceramic; B2, Rely 3M EPSE bonding to glass-infiltrated ceramic; B3, Kerr NX3 bonding to glass-infiltrated ceramic; Type 1, cohesive failures of resin cements; Type 2, failures of the glass-infiltrated ceramics; Type 3, adhesive failures of resin cements bonding to glass-infiltrated ceramic; Type 4, quasi-cleavage fracture; SEM, scanning electron microscopy.

4. Discussion

Resin cements can determine the success of fixed dental prostheses by bonding juxtaposed surfaces together to achieve marginal sealing and adhesive of interface between different kinds of substrate, as well as adequate preservation and resistance [18]. Generally, two mechanical factors should be considered as for the bonding of ceramics to the tooth: the adhesive forces at the resin-ceramic interface and at the resin-tooth interface [19]. One study showed that the resin cement combined with the ceramic can significantly enhance the micro-shear bond strength [20]. This study was designed to investigate the bonding strength and restorative effect of Panavia™-F, Rely 3M EPSE and Kerr NX3, finding that Kerr NX3 may have higher bond strength values and better restorative effect than Panavia™-F and Rely 3M EPSE. The main component of resin composite is the resin matrix and an inorganic filler, wherein the inorganic filler is uniformly dispersed in the resin matrix, and after curing the resin composite essentially becomes a polymer composite reinforced by the inorganic filler; the resin composite matrix consists of a resin basis and the dilution monomers that are primarily methacrylate monomers [20]. Furthermore, Ishikiriama et al. have reported that the degree of wear in NX3 is relatively small, indicating that the hardness of this material is higher than that of an average adhesive resin [21]. Lambade et al. also showed that Nexus NX3 has the highest shear strength [22]. In line with previous studies, it has been found that NX3 can solve the incompatibility problems during the bonding process and has the highest bond strength and best restorative effect in this paper.

In this study, it was found that the HV of Panavia™-F was almost free from the impact of water storage, while the HV of Rely 3M EPSE changed more obviously after the long-term water storage. The Kerr NX3 not only showed higher HV, but was also less impacted by water storage than Rely 3M EPSE. Water adsorption as well as polymer chain hydrolysis and plastification can seriously affect the resin composite in the mechanically mixed layer, thus causing the aging of morphologic integrity and affecting the bonding strength of resin [23]. Therefore, this study simulated the oral environment and stored the resin cements in water to figure out whether the adhesive strength of three resin composites would be affected. Moreover, Panavia™-F was a self-etching adhesive system that can be solidified by a dual mode of optical and chemical processes, thus providing excellent engineering features with wear resistances; [24]. Since Rely-X Unicem (3M EPSE) was a self-adhesive resin and the water storage could reduce the durability of self-adhesive bond strength by the slow degradation of the unprotected collagens, it indicated that 3M EPSE was more affected by water storage and decreased its adhesion strength [25,26]. Due to its unique amine free redox system, Nexus NX3 was resistant to the acidic monomers in the air inhibition layer of the light cured adhesive material so that the adhesive bonding strength increased, suggesting it was less impacted by water storage [22].

In summary, the tensile testing, compression test, thermal-cold cycling and shear strength testing for the three different resin cements showed that as compared to Panavia™-F and Rely 3M EPSE, Kerr NX3 showed higher micro-tensile bond strength, compressive strength and shear strength, and less interface damage and quasi-cleavage fracture in the Kerr NX3 bonding to glass-infiltrated ceramics. However, there are still some limitations in this study. For example, the clinical efficacy of Kerr NX3 was not assessed in this study and hence requiring further evaluation. And due to less evidences showing how the glass-infiltrated ceramic would be affected by Kerr NX3 resin cement, more advanced technology should be used for researching the clinical efficacy of Kerr NX3.

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