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A laboratory study of the long-term bond strength**

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Are self-adhesive resin cements a valid alternative to conventional resin cements? A laboratory study of the long-term bond strength

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Short title: Shear bond strength of resin cements

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Abstract (250 words)

Objectives: The aim of the study was to test whether or not the shear bond strengths of six self-adhesive resin cements to dentin and to glass-ceramic, 24hours and long-term-aged, are similar to the one of a conventional resin cement. *Methods:* Human molars (N=168, n=12 per group) and silica-based glass-ceramic specimens (N=168, n=12 per group) were embedded in acrylic resin and randomly divided into 28 groups. The following resin cements were luted according to the manufacturers' instructions: Clearfil SA(CSA), G-Cem(GCM), SmartCem2(SMC), SpeedCEM(SPC), RelyX Unicem(RXU), RelyX Unicem2(RXU2) and Panavia21(control group, PAN). Shear bond strength was measured initially (24h of water storage 37°C) and after aging (24,000 thermal cycles,5/55°C). The failure types (adhesive, cohesive) were evaluated after debonding. The shear bond strength values were analysed using three-way and one-way ANOVA, followed by a post hoc Scheffé and two-sample Student's t-tests. *Results:* RXU, RXU2 and GCM showed similar after 24hours and aged shear bond strength to dentin as the control group. CSA, SMC and SPC exhibited significantly lower values. Before aging, none of the bond strength values to glass-ceramic differed significantly from the other. After thermocycling, GCM showed higher results to glass-ceramic than CSA, SMC, RXU2 and the control group. Analyzing failure types after spontaneous debonding and shear bond test at dentin, solely adhesive failures were found, while at glass-ceramic only cohesive failures occurred. *Conclusion:* Not all self-adhesive resin cements can be a valid alternative to conventional resin cements in order to bond silica-based glass-ceramics to human dentin.

Keyword: self-adhesive resin cement, dentin, shear bond strength, glass-ceramic, conventional resin cement

1. Introduction

The available cements in dentistry can be classified into water-based and resin-based polymerizing cements [1]. Water-based cements include glass-ionomer and zinc phosphate cements, whereas polymerizing cements comprise resin composites, adhesive cements and resin-modified glass ionomer cements. Chemical bonding of water-based cements to tooth tissues or restoration materials is only low (for glass-ionomer cements) or not existent (for zinc phosphate cement) [2]. In contrast, polymerizing cements constitute some chemical and mechanical connection to the tooth and to the restoration [3, 4].

The type of cementation may influence the outcome of the reconstruction depending upon restorative material the reconstruction is made out of, i.e. glass-ceramic, oxide ceramics and composites. [5, 6]. Several studies showed that silica-based glass-ceramic restorations exhibit better clinical long-term stability when luted with polymerizing resin-based cements instead of water-based cements [5, 6]. When polymerizing resin-based cements were applied, the fracture resistance of silica-based glass-ceramic crowns increased significantly [7]. Hence, this restorative materials require to be reinforced by adhesive cementation [7-9].

In order to achieve a good bonding between the polymerizing resin-based cement and the substrates, i.e. the restorative material and the tooth substance, several pre-treatment bonding steps are required. These pre-treatment steps are technique sensitive and, therefore, prone to handling errors. It has been shown that polymerizing cements are very technique sensitive. Handling problems like e.g. contamination of the substrate

with saliva or blood significantly reduce the bond strength of the respective polymerizing cement [10, 11, 12].

To facilitate the pretreatment procedures of the tooth tissue, self-adhesive resin cements were recently developed. Self-adhesive resin cements are polymerizing cements, which bond to the substrate, more specifically to dentin, without the pretreatment with bonding solutions. The first introduced and well documented self-adhesive resin cement is RelyX Unicem (3M ESPE, Germany). In order to achieve a self-adhesive reaction of this cement to the tooth structure, new methacrylate monomers with phosphoric acid groups were implemented. This results in a low pH-value and hydrophilic properties in the beginning of the setting. Subsequently, the negatively charged groups of the monomer bind to Ca^{2+} ions of the tooth and in combination with the alkaline part of the fillers a neutralization reaction follows [13]. Several in vitro and clinical studies showed promising results of RelyX Unicem with respect to bond strength [14-17]. The chemical reaction of most of the other self-adhesive cements have not been clearly announced yet by the manufacturers.

Within the last years, several new self-adhesive resin cements have been introduced [18]. At present, no scientific literature is available of the newly introduced self-adhesive resin cements and their bond strength after long-term aging. Whereas studies show that aging can have a negative impact on the shear bond strength of conventional resin cements [20], the bond strength of the newly introduced self-adhesive resin cements after long-term aging has not been investigated yet. [21]. Good long-term bonding capacity, however, is desired for clinical long-term success. As mentioned before, reconstructions made out of weak silica-based ceramics need to be reinforced

by the adhesive cementation. Consequently, the self- adhesive resin cements should be to establish good long – term bonding not only to the tooth substance, but also to the ceramic. Hence, laboratory studies of the new self-adhesive resin cements are needed, which simulate the oral conditions and age the adhesive interfaces to measure the long-term bonding capacity to tooth and to the reconstruction material [19].

Therefore, the aim of this study was to test whether or not various self-adhesive resin cements exhibit similar shear bond strength to the substrates dentin and glass-ceramic as a conventional resin cement.

The null-hypothesis was that the shear bond strength of self-adhesive resin cements to both substrates is similar to the conventional cement both initially, and after long-term aging.

2. Material and Methods

Six self-adhesive resin test cements were included in the study. One conventional resin cement acted as control group. Table 1 gives detailed information of all tested cements. 168 teeth were divided into 14 groups of twelve each. Additionally 168 ceramic specimens were divided into 14 further experimental groups of twelve each.

2.1 Preparation of human dentin specimens

For this study 168 caries-free human molars were used. The teeth were cleaned from remnant soft tissue and stored in 0.5% Chloramine T at room temperature during the first 7 days after extraction and thereafter stored in distilled water at 5°C for a maximum of 6 months. They were ground flat with silicon carbide polishing paper P80 (Labo-Pol-21; Struers, Ballerup, Denmark) under water-cooling and subsequently embedded in a cylindrical form by acrylic resin (ScandiQuick, ScanDia, Hagen, Germany). The teeth were ground with SiC P500 until a dentin surface area of at least 5mm² was exposed. Immediately prior to the luting procedure, the dentin specimens of the control group were pretreated according to the respective manufacturer's recommendations (Tab. 2).

2.2 Preparation of glass-ceramic specimens

Glass-ceramic ingots (VITA Mark II, VITA Zahnfabrik, Bad Säckingen, Germany) were embedded in acrylic resin ScandiQuick (ScanDia, Hagen, Germany) and cut from cylindrical rods into slices of 5 mm thickness by a cutting machine (Accutom 50, Struers, Ballerup, Denmark). The specimens were flattened with a polishing machine with

P2400 silicon carbide polishing paper (SCAN DIA, Hagen, Germany). The surfaces of the glass-ceramic specimens were etched with 5% hydrofluoric acid for 60s (VITA Ceramics Etch; VITA Zahnfabrik, Bad Säckingen, Germany, LOT 12150), rinsed with water, cleaned with alcohol, dried with oil-free air, and silanized according to the respective manufacturer's recommendations (Tab. 2).

2.3 Resin cement luting

The embedded specimens (human teeth and glass-ceramic) were randomly divided in the test- or control groups. In order to apply the different cements to the bonding area, the specimens were fixed in a special holding device to retain the surface parallel to the bench. An acrylic cylinder with an inner diameter of 2.9 mm (D+R Tec, Birmensdorf, Switzerland) was fixed on the specimen surface by means of a custom-made device. Therefore the procedure for the preparation of the specimens will only be briefly summarized. The cements were mixed according to the manufacturers' recommendations and applied into the opening of the cylinders. A steel screw with an inner diameter matching to the acrylic cylinders was inserted parallel to the axis of the cylinders and loaded with 1N. The excess cement was removed with foam pellets. By using this device it could be ensured to attain a thickness of the cement of 1mm evenly. The specimens were light polymerized by an LED polymerization light with a light intensity of 1200 mW/cm² (Bluephase G2; Ivoclar Vivadent GmbH, Schaan, Liechtenstein) according to the manufacturers' recommendations. To achieve a constant light polymerization, the output tip has been kept in contact to the acrylic cylinder from two opposed sites for 30 seconds each per side. All specimens were carefully removed and stored in distilled water at 37°C for 24 h. Subsequently, half of all specimens was

subjected to long-term thermocycling during 24'000 cycles at 5° and 55°C with a dwelling time of 20s.

2.4 Shear bond strength measurements

The shear bond strength was measured in a Universal Testing Machine (Z 010; Zwick, Ulm, Germany). The specimens were positioned in the sample holder with the bonding surface parallel to the loading piston. The loading piston had a chisel configuration and the load was applied with a crosshead speed of 1 mm/min. The load was applied at the outer surface of the cylinder in a distance of 300 µm to the specimen surface. The maximal load was measured before de-bonding occurred. The shear bond strength values were calculated with the following formula: fracture load/bond area = N/mm² = MPa.

2.5 Failure types analysis

The de-bonding surface was examined by two operators under a binocular microscope (Wild M3B, Heerbrugg, Switzerland) and the failure were classified into the following three different failure types: i) adhesive (no cement remnants on the polished specimen surface), ii) cohesive (fracture totally into the ceramic/dentin), and iii) mixed (cement remnants and polished specimen surface exposed).

2.6 Statistical analysis

The statistical package for Social Science Version 19 (SPSS Inc., Chicago, IL, US) was used for the statistical analysis. After a first screening of the results, the values for dentin showed a normal distribution of 71% and for glass ceramic 86%. Hence, the

shear bond strength values were analyzed based on the assumption of normal distribution. Three-way ANOVA between resin cement vs. aging type vs. bond area and one-way ANOVA testing the impact of resin cement and the impact of aging level on bond strength has been performed followed by a post hoc Scheffé test. In addition, a two-sample Student's t-test was computed. All results from the statistical analysis with a p-value <5% were considered as statistically significant.

3. Results

The results of the shear bond strength measurements on dentin and glass-ceramic, initially and after long-term thermocycling, are shown in Table 3 and Fig. 2. The results of three-way ANOVA are presented in Table 4. All of the tested self-adhesive resin cements exhibited similar shear bond strength to glass-ceramic as a conventional resin cement. To the substrate dentin only some cements resulted in similar values (GCM, RXU, RXU2) whereas others resulted in significantly lower data (CSA, SMC, SPC) comparing to the control group. The resin cement ($p < 0.001$), the bonding area ($p < 0.001$) and the aging mode ($p = 0.003$) had a significant effect on the shear bond strength.

3.1 Shear bond strength to dentin

At dentin, two of six self-adhesive resin cements, CSA ($p = 0.003$) and SMC ($p = 0.003$), showed significantly lower initial shear bond strength compared to the control group (conventional cement PAN). The self-adhesive resin cements, CSA and SMC, presented significantly lower data than RXU ($p < 0.001$) and GCM ($p \leq 0.001$). The values of SPC were significantly lower compared to RXU ($p = 0.029$).

After long-term thermocycling, three of six self-adhesive resin cements, CSA ($p < 0.001$), SMC ($p < 0.001$) and SPC ($p < 0.001$), resulted in significantly lower shear bond strength values than the conventional cement PAN. No statistical difference was found between the three self-adhesive resin cements CSA, SMC and SPC. ($p < 0.001$). RXU revealed significantly higher values than all the other self-adhesive resin cements ($p < 0.001$ - 0.008), but not statistically different to control group PAN ($p = 0.499$). CSA, SMC and SPC showed significantly lower data than GCM ($p < 0.001$), RXU ($p < 0.001$) and RXU2

($p < 0.001-0.003$). All of the tested specimens of the group with SMC resulted in complete debonding after thermal fatigue.

3.2 Shear bond strength to glass-ceramic

Among glass-ceramic specimens, the initial shear bond strength of all tested self-adhesive resin cements ($p = 0.708-0.999$) did not differ significantly from the control group PAN. Within the self-adhesive resin cements, SPC showed significantly higher shear bond strength than SMC ($p = 0.042$) and RXU2 ($p = 0.049$).

After long-term thermocycling, GCM presented significantly higher shear bond strength values than the conventional cement PAN ($p = 0.008$) and the self-adhesive resin cements CSA ($p < 0.001$), SMC ($p = 0.005$) and RXU2 ($p < 0.001$) on glass-ceramic. CSA revealed significantly lower measurements than SPC ($p = 0.014$) and RXU ($p < 0.001$).

3.3 Impact of thermocycling

Within the self-adhesive resin cements, the negative impact of thermocycling on the shear bond strength to dentin was significant for CSA ($p = 0.015$), SMC ($p < 0.001$) and SPC ($p < 0.001$). GCM ($p < 0.001$), SMC ($p = 0.002$), RXU ($p = 0.013$) and RXU2 ($p = 0.046$) showed significantly higher values for shear bond strength on glass-ceramic after long-term thermocycling. Within the control group PAN no impact of aging on the shear bond strength was observed for both bonding areas (dentin: $p = 0.224$, glass-ceramic $p = 0.087$).

No correlation of the shear bond strength of all resin cements between the two bonding areas dentin and glass-ceramic was found ($R^2 = 0.2274-0.28478$).

3.4 Failure type

Within the dentin specimens, only adhesive failures (no cement remnants on the polished specimen surface) occurred. All glass-ceramic specimens showed cohesive failures (fracture totally into the ceramic). Mixed failure types were not observed at all.

4. Discussion

The results of the present study showed that the null hypothesis had to be rejected.

The self-adhesive resin cements exhibited highly differing bonding performances to dentin. While some of the self-adhesive resin cements (RXU, RXU2, GCM) had similar bond strength values as the control cement PAN, other resulted in significantly lower values before (CSA, SMC) and after (CSA, SMC, SPC) long-term ageing. More consistent bonding performance of the self-adhesive resin cements was observed at glass-ceramics, resembling the bonding performance of the control cement. Finally, the bond strength of self-adhesive cements to dentin was lower than the bond strength to glass-ceramics. More consistent bonding performance of the self-adhesive resin cements was observed at glass-ceramics. With one exception (GCM), all tested self-adhesive resin cements revealed similar shear bond strength values on glass-ceramics as the control group PAN. GCM exhibited significantly higher values than PAN. In general, bonding to dentin lead to lower bond strength values than bonding to glass-ceramics.

Due to the difference of the bond strength of the various self-adhesive resin cements, an overall judgement of the bonding capacity of this new type of cement is limited. Due to the different chemical compositions of the tested self-adhesive resin cements, different bonding mechanisms occur. All self-adhesive resin cements contain multifunctional monomers that react with aid of acid groups. Albeit, the precise chemical bonding reaction of the respective cements is still not clarified. More comparative studies are needed to classify the different groups of cements.

The bonding effectiveness on dentin is still the bigger challenge than bonding to glass-ceramics. The failure types on dentin showed only adhesive failures. At conventional resin cements during dentin pre-treatment the dentin tubules are opened and a hybrid layer with resin tags into the dentin can be observed [10] or the smear layer is merely altered and the intertubular collagen only mild demineralised by the bonding agent as in the control group PAN [10]. Measurements of pH values of RXU and GCM after light curing showed values of 5.0 or less [22]. Therefore this cements seem to have technically a potential of decalcification of the dentin. But, neither demineralization nor infiltration of dentin can be shown [10]. In addition, pretreatment of the dentin facilitates to create a real hybrid layer which occurred with PAN but not with RXU [4]. Furthermore, differences in shrinkage behaviour [24], physical properties [25, 26], pH values, and film thickness [22] of different self-adhesive resin cements were shown in a number of studies. In a study by Kitzmüller [24], shrinkage behaviour was depended on the temperature of the cement and the curing mode. In this study, RXU revealed significantly less shrinkage strains when self-cured compared to dual-cured setting. In the study by Han [22] , the pH value for RXU was 7.0 48 hours after cementation, while

GCM (pH 3.6) and SMC (pH 4.0) maintained in a significant lower pH level. Similar results have been shown in a study by Saskalauskaite [26]. Further studies are needed to determine if a low pH level influences the adhesion of cements to dentin. The fact that in the present study some of the tested self-adhesive cements resulted in similar values of the shear bond strength on dentin as control group whereas other self-adhesive resin cements showed significantly lower results than control group might be caused by this brand-specific differences. This result conforms to other studies that conclude that self-adhesive capacity depend on product-specific factors [23]. The study of Scherrer [27] demonstrates that the different examiners may influence bond strength values even when similar test methods and sample preparation are used. The comparison of the absolute bond strength values can only be drawn within the same study.

Other variables like the quality of dentin and the proximity to the pulp of the tested dentin can impact the bonding effectiveness [28]. Interestingly the new developed RXU2 [29] resulted in lower strong bond strength values than the primarily investigated RXU. The reason leading to smaller bond strength requires to be clarified in further studies.

On glass-ceramic only cohesive failure types were observed. Therefore, it can be concluded that the bonding effectiveness of all cements is higher than the fracture resistance of glass-ceramics. Nevertheless the results on glass-ceramic differ significantly. This can be explained by the differences of the self-adhesive resin cements concerning the viscosity or the weight percentage of cement particles. The study of Han [22] showed that there are significant differences in the particle content between GCM and RXU or SMC. An increase of particle content results in an increase of viscosity. As a consequence a cement with a lower viscosity penetrates deeper in the acid modified

glass-ceramic surface. This might be the reason why GCM with a low particle content results in higher shear bond strength in the present study than RXU with a higher particle content.

Long-term aging with thermocycling exhibited a significant impact on shear bond strength for some of the tested cements whereas no impact was observed for the control group. This finding is in accordance to the results of other studies [20, 30]. Water absorption of different self-adhesive resin cements varies [31]. In a recent study, GCM showed significantly higher water absorption than PAN, RXU or CSA [31]. While absorption of water results in an expansion of the material, the anchorage of the cement in the acid etched ceramic roughness is expected to be higher. This may be the reason why GCM results in higher shear bond strength values on glass-ceramic after long-term ageing than the other cements.

5. Conclusion

Based on the findings of this study, it can be concluded within its limitations that not all self-adhesive resin cements can be a valid alternative to conventional resin cements to bond silica-based glass ceramics to dentin. Among the tested cements only GCM, RXU and RXU2 showed similar bond strength on dentin and glass-ceramics simultaneously compared to the conventional resin cement PAN.

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References:

- [1] Edelhoff D, Ozcan M. To what extent does the longevity of fixed dental prostheses depend on the function of the cement? Working Group 4 materials: cementation. *Clin Oral Implants Res* 2007 Jun;18 Suppl 3:193-204.
- [2] Burke FJ. Trends in indirect dentistry: 3. Luting materials. *Dent Update* 2005 Jun;32(5):251-4, 7-8, 60.
- [3] Peumans M, Van Meerbeek B, Yoshida Y, Lambrechts P, Vanherle G. Porcelain veneers bonded to tooth structure: an ultra-morphological FE-SEM examination of the adhesive interface. *Dent Mater* 1999 Mar;15(2):105-19.
- [4] De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, et al. A critical review of the durability of adhesion to tooth tissue: methods and results. *J Dent Res* 2005 Feb;84(2):118-32.
- [5] Sadowsky SJ. An overview of treatment considerations for esthetic restorations: a review of the literature. *J Prosthet Dent* 2006 Dec;96(6):433-42.
- [6] Kramer N, Lohbauer U, Frankenberger R. Adhesive luting of indirect restorations. *Am J Dent* 2000 Nov;13(Spec No):60D-76D.
- [7] Bindl A, Luthy H, Mormann WH. Thin-wall ceramic CAD/CAM crown copings: strength and fracture pattern. *J Oral Rehabil* 2006 Jul;33(7):520-8.
- [8] Jensen ME, Sheth JJ, Tolliver D. Etched-porcelain resin-bonded full-veneer crowns: in vitro fracture resistance. *Compendium* 1989 Jun;10(6):336-8, 40-1, 44-7.
- [9] Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. *J Prosthet Dent* 2003 Mar;89(3):268-74.

- [10] Monticelli F, Osorio R, Mazzitelli C, Ferrari M, Toledano M. Limited decalcification/diffusion of self-adhesive cements into dentin. *J Dent Res* 2008 Oct;87(10):974-9.
- [11] Frankenberger R, Reinelt C, Petschelt A, Kramer N. Operator vs. material influence on clinical outcome of bonded ceramic inlays. *Dent Mater* 2009 Aug;25(8):960-8.
- [12] Frankenberger R, Kramer N, Petschelt A. Technique sensitivity of dentin bonding: effect of application mistakes on bond strength and marginal adaptation. *Oper Dent* 2000 Jul-Aug;25(4):324-30.
- [13] 3M E. RelyX Unicem self-adhesive universal resin cement – technical product profile. 2009.
- [14] Aschenbrenner CM, Lang R, Handel G, Behr M. Analysis of marginal adaptation and sealing to enamel and dentin of four self-adhesive resin cements. *Clin Oral Investig* 2011 Feb 16.
- [15] Piwowarczyk A, Lauer HC, Sorensen JA. In vitro shear bond strength of cementing agents to fixed prosthodontic restorative materials. *J Prosthet Dent* 2004 Sep;92(3):265-73.
- [16] Mormann W, Wolf D, Ender A, Bindl A, Gohring T, Attin T. Effect of two self-adhesive cements on marginal adaptation and strength of esthetic ceramic CAD/CAM molar crowns. *J Prosthodont* 2009 Jul;18(5):403-10.
- [17] Fabianelli A, Goracci C, Bertelli E, Monticelli F, Grandini S, Ferrari M. In vitro evaluation of wall-to-wall adaptation of a self-adhesive resin cement used for luting gold and ceramic inlays. *J Adhes Dent* 2005 Spring;7(1):33-40.

- [18] Radovic I, Monticelli F, Goracci C, Vulicevic ZR, Ferrari M. Self-adhesive resin cements: a literature review. *J Adhes Dent* 2008 Aug;10(4):251-8.
- [19] Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. *J Dent* 1999 Feb;27(2):89-99.
- [20] Holderegger C, Sailer I, Schuhmacher C, Schlapfer R, Hammerle C, Fischer J. Shear bond strength of resin cements to human dentin. *Dent Mater* 2008 Jul;24(7):944-50.
- [21] Liu Q, Meng X, Yoshida K, Luo X. Bond degradation behavior of self-adhesive cement and conventional resin cements bonded to silanized ceramic. *J Prosthet Dent* Mar;105(3):177-84.
- [22] Han L, Okamoto A, Fukushima M, Okiji T. Evaluation of physical properties and surface degradation of self-adhesive resin cements. *Dent Mater J* 2007 Nov;26(6):906-14.
- [23] Sarr M, Mine A, De Munck J, Cardoso MV, Kane AW, Vreven J, et al. Immediate bonding effectiveness of contemporary composite cements to dentin. *Clin Oral Investig* 2009 Aug 25.
- [24] Kitzmuller K, Graf A, Watts D, Schedle A. Setting kinetics and shrinkage of self-adhesive resin cements depend on cure-mode and temperature. *Dent Mater* 2011 Jun;27(6):544-51.
- [25] Giraldez I, Ceballos L, Garrido MA, Rodriguez J. Early Hardness of Self-Adhesive Resin Cements Cured under Indirect Resin Composite Restorations. *J Esthet Restor Dent* 2011 Apr;23(2):116-24.
- [26] Saskalauskaite E, Tam LE, McComb D. Flexural strength, elastic modulus, and pH profile of self-etch resin luting cements. *J Prosthodont* 2008 Jun;17(4):262-8.

- [27] Scherrer SS, Cesar PF, Swain MV. Direct comparison of the bond strength results of the different test methods: a critical literature review. *Dent Mater* 2010 Feb;26(2):e78-93.
- [28] Leloup G, D'Hoore W, Bouter D, Degrange M, Vreven J. Meta-analytical review of factors involved in dentin adherence. *J Dent Res* 2001 Jul;80(7):1605-14.
- [29] 3M E. RelyX Unicem 2 Automix - Technical Data Sheet. ESPE 3M; 2011.
- [30] Mazzitelli C, Monticelli F, Toledano M, Ferrari M, Osorio R. Effect of thermal cycling on the bond strength of self-adhesive cements to fiber posts. *Clin Oral Investig* 2011 Jun 14.
- [31] Nakamura T, Wakabayashi K, Kinuta S, Nishida H, Miyamae M, Yatani H. Mechanical properties of new self-adhesive resin-based cement. *J Prosthodont Res* 2010 Apr;54(2):59-64.

Tables

Table 1. The brands, batch numbers, abbreviations, manufacturers and chemical composition of the tested materials.

Cement and bonding agents	Abbreviation	Manufacturers	Batch	Composition
Panavia21 + ED Primer A/B + Clearfil Ceramic Primer	PAN	Kuraray Dental Co. Ltd., Osaka, Japan	408CA 00283A/00143E 00009C	MDP, Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, fillers, BPO, hydrophilic liphatic dimethacrylate, hydrophilic dimethacrylate, DEPT, sodium aromatic sulfonate HEMA, MDP, 5-NMSA, water, accelerator, ethanol, 3-methacryloxypropyl tris(trimethylsiloxy)silane MPTS, initiator MDP, ethanol, MPTS
Clearfil SA + Clearfil Ceramic Primer	CSA	Kuraray Dental Co. Ltd., Osaka, Japan	033BBA 00009C	MDP, Bis-GMA, TEGDMA, other methacrylate monomers, silanated barium glass filler, silanated colloidal silica, dl-camphorquinone, benzoyl peroxide, initiator, surface treated sodium fluoride, accelerators, pigments MDP, ethanol, MPTS
G-Cem + GC Ceramic Primer	GCM	GC, Leuven, Belgium	810241 901272	Fluoro-alumino-silicate glass, initiator, pigments, 4-META, phosphoric acid ester monomer, water, UDMA, Dimethacrylate, silica powder, initiator, stabilizer Ethanol, Methyl methacrylate, 2-HEMA
SmartCem2	SMC	Dentsply DeTrey GmbH,	809231	PENTA,UDMA, EBPADMA, Di-and trifunctional diluents,

+ Calibra Silane Coupling Agent		Konstanz, Germany	812051	Photoinitiating system, self-cure initiating system Acetone, Ethyl Alcohol, Organo Silane
SpeedCEM	SPC	Ivoclar Vivadent, Schaan, Liechtenstein	627590	Acidic monomers, Dimethacrylates, barium glass, ytterbium trifluoride, co-polymer, silicon dioxides, catalysts, stabilizers, pigments
+ Monobund Plus			626221	Ethanol, water, 3-methacryloxy propyl-trimethoxysilane
RelyX Unicem (Aplicap)	RXU	3M ESPE, Seefeld, Germany	363991	Methacrylate monomers containing phosphoric acid groups, alkaline fillers, silanated fillers, initiator components, pigments, methacrylate monomers, initiator components, stabilizers
+ RelyX Ceramic Primer			7XY	Ethanol, water, methacrylacid-3-trimethoxysilypropylester
RelyX Unicem2 (Automix)	RXU2	3M ESPE, Seefeld, Germany	421455	Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizer components, rheologic additives, alkaline fillers, pigments, rheologic additives
+ RelyX Ceramic Primer			7XY	Ethanol, water, methacrylacid-3-trimethoxysilypropylester

Table 2. Pretreatment of dentin and glass-ceramic

	Pretreatment of dentin	Pretreatment of glass-ceramic
Control group	Dispense one drop of ED Primer liquids A and B, stir for 5 s, apply to dentin with a sponge pledget, leave for 60s, dry gently by oil-free air	Etching with 5% hydrofluoric acid 60 s, rinse off with water spray, drying with alcohol 98%, application of the silane (Clearfil Ceramic Primer)
Test groups	No pretreatment	Etching with 5% hydrofluoric acid 60 s, rinse off with water spray, drying with alcohol 98%, application of the silane recommended by the manufacturer of the cement

Table 3. Mean (SD) shear bond strength with confidence intervals (95% CI) and significantly differences between tested cements.

Group	Mean (SD) MPa	95% CI MPa	p-value initial vs. aged	Mean (SD) MPa	95% CI MPa
	24 h H ₂ O (37°)			24 h H ₂ O + TC	
Dentin					
PAN	7.4 (3.9) ^{b,c}	(4.9;9.9)	0.224	9.0 (2.3) ^{b,c}	(7.5;10.6)
CSA	3.3 (1.2) ^a	(2.5;4.1)	0.015	1.9 (1.3) ^a	(1.0;2.8)
GCM	7.9 (2.1) ^{b,c}	(6.5;9.3)	0.531	7.2 (2.9) ^b	(5.3;9.2)
SMC	3.3 (1.5) ^a	(2.3;4.3)	<0.001	0.0 ^a	-
SPC	5.7 (1.8) ^{a,b}	(4.5;7.0)	<0.001	2.1 (1.3) ^a	(1.2;3.0)
RXU	9.1 (2.1) ^c	(7.7;10.5)	0.069	11.1 (2.9) ^c	(9.2 ;12.0)
RXU2	6.1 (1.2) ^{a,b,c}	(5.3 ;6.9)	0.821	6.3 (2.6) ^b	(4.6 ;8.0)
Glass-ceramic					
PAN	13.0 (2.4) ^{a,b}	(11.5;14.4)	0.087	14.4 (1.8) ^{a,b}	(13.3;15.6)
CSA	11.8 (2.4) ^a	(10.2;13.4)	0.368	10.9 (2.6) ^a	(9.1;12.6)
GCM	12.4 (1.6) ^{a,b}	(11.4;13.5)	<0.001	19.0 (3.2) ^c	(16.9;21.0)
SMC	11.2 (1.7) ^a	(10.1;12.3)	0.002	14.3 (2.5) ^{a,b}	(12.6;15.9)
SPC	14.7 (3.2) ^b	(12.7;16.7)	0.670	15.2 (1.9) ^{b,c}	(13.9;16.4)
RXU	13.7 (2.2) ^{a,b}	(12.2;15.1)	0.013	16.7 (3.2) ^{b,c}	(14.6;18.7)
RXU2	11.2 (2.6) ^a	(9.5;13.0)	0.046	13.4 (2.3) ^{a,b}	(11.9;14.9)

Table 4. Results of three-way ANOVA interaction between resin cement vs. aging type vs. bond area.

	Sum of squares	df	Mean squares	F	p values
Constant parameters	31949	1	31949	6004	<0.001
Resin cement	1350	6	225	42.3	<0.001
Bond area	5217	1	5317	999	<0.001
Aging level	48.9	1	48.9	9.2	0.003
Resin cement vs. aging area	457	6	76.2	14.3	<0.001
Resin cement vs. aging level	221	6	36.9	6.9	<0.001
Bond area vs. aging level	189	1	189	35.4	<0.001
Resin cement vs. bond area vs. aging level	158	6	26	4.9	<0.001
Error	1650	310	5.3		
Total	41477	338			

Figures

Figure 1.

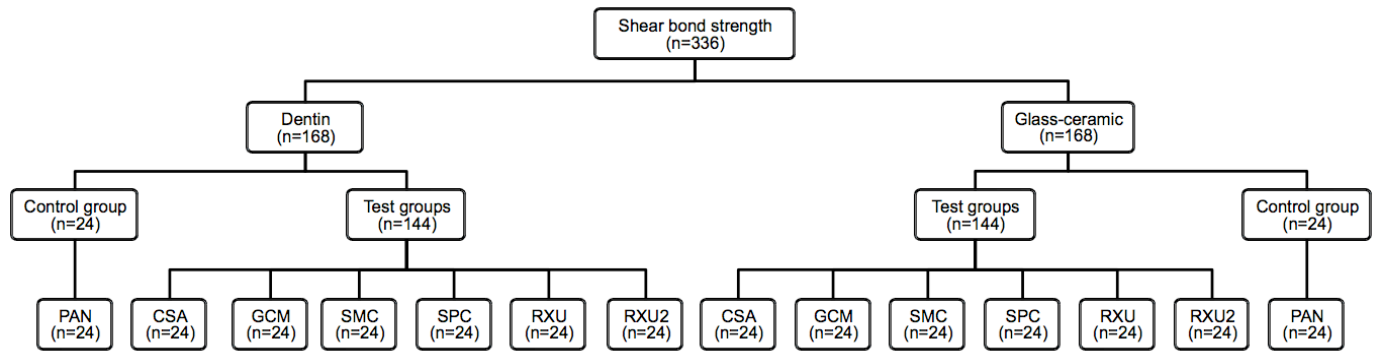


Fig. 2

