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ORIGINAL ARTICLE

Impact of Gluma Desensitizer on the tensile strength of zirconia crowns bonded to dentin: an in vitro study

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Abstract This study tested the impact of Gluma Desensitizer on the tensile strength of zirconia crowns bonded to dentin. Human teeth were prepared and randomly divided into six groups (N=144, n=24 per group). For each tooth, a zirconia crown was manufactured. The zirconia crowns were cemented with: (1) Panavia21 (PAN), (2) Panavia21 combined with Gluma Desensitizer (PAN-G), (3) RelyX Unicem (RXU), (4) RelyX Unicem combined with Gluma Desensitizer (RXU-G), (5) G-Cem (GCM) and (6) G-Cem combined with Gluma Desensitizer (GCM-G). The initial tensile strength was measured in half (n=12) of each group and the other half (n=12) subjected to a chewing machine (1.2 Mio, 49 N, 5°C/50°C). The cemented crowns were pulled in a Universal Testing Machine (1 mm/min, Zwick Z010) until failure occurred and tensile strength was calculated. Data were analyzed with one-way and two-way ANOVA followed by a post hoc Scheffé test, t test and Kaplan–Meier analysis with a Breslow–Gehan analysis test (α=0.05). After the chewing simulation, the self-adhesive resin cements combined with Gluma Desensitizer showed significantly higher tensile strength (RXU-G, 12.8±4.3 MPa; GCM-G, 13.4±6.2 MPa) than PAN (7.3±1.7 MPa) and PAN-G (0.9±0.6). Within the groups, PAN, PAN-G and RXU resulted in significantly lower values when compared to the initial tensile strength; the values of all other test groups were stable. In this study, self-adhesive resin cements combined with Gluma Desensitizer reached better long-term stability compared to PAN and PAN-G after chewing simulation.

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Introduction

The utilisation of all-ceramic reconstructions is increasing due to their favourable mechanical stability [1–3], high esthetic properties and, most importantly, high biocompatibility [4]. Two different types of ceramic are currently in use: glass–ceramic and oxide–ceramics.

Glass–ceramic reconstructions must be adhesively cemented, and by using resin cements, the stability and the clinical long-term success are improved [5–8]. Resin cements chemically bond to both the ceramic substrate as well as the tooth substance and thereby reinforce the tooth reconstruction complex [5, 6, 9–13]. Furthermore, their high translucency and tooth-resembling colour improve the esthetic result [13]. From oxide ceramics, zirconia can be cemented traditionally (e.g. glass ionomer cement) or with resin cements. The main advantage of adhesive cemented reconstructions is a reduced marginal microleakage [14, 15]. Significantly higher bond strength values were obtained when zirconia was bonded with resin cement containing a phosphate monomer compared to those having bis-GMA monomers [16, 17].

The application of resin cements, because of their sensitivity to moisture [5, 14, 18], which implies the application of a dental dam, complicates its clinical utilisation. Self-adhesive resin cements are simple in use and more efficient in handling [14]. These partially hydrophilic resin cements do not require any pretreatment of tooth substance [5, 14, 18]. The difference of self-adhesive resin cements from those of other cements lies in the chemical composition: the addition of phosphor monomers combined with, e.g. phosphoric acid ester, carboxylic acid or amino acid derivate. These acidic monomers react with the tooth surface and generate a slight retentive pattern.

Self-adhesive resin cements do not require separate conditioning of dentin since their adhesion mechanism is based on the partial retention on the smear layer. The applied procedures are intended to provide sufficient acidity to penetrate the dentin through the smear layer and allow infiltration of the monomers inside the demineralised collagen network [19]. Due to this effect, priming and bonding can be eliminated.

When the enamel has been removed, millions of dentinal tubules are exposed [20], and dentin exposure means a potential increased risk of pulpal injuries [21]. The sensitivity of a prepared tooth can be reduced by pretreatment with a desensitizer. It has been reported that the sealing of dentin also decreases the sensitivity of a prepared tooth, resulting in less postoperative pain [22–25].

Gluma Desensitizer (5% glutaraldehyde) reduces dentin permeability, reduces dentin sensitivity and disinfects dentin [26, 27]. The diffusion of monomers into dentin is likely to be accelerated by 2-hydroxyethyl-methacrylate (HEMA) [28]. As soon as the dentin tubules are closed, the hydrodynamics of dentin liquidity is reduced and the sensitivity decreases. The dentin adhesives build a hybrid layer and seal the dentin surface in one application. For desensitisation, the obliteration of the dentin is relevant. Panavia21 with dentin pretreatment (ED Primer) seals the dentin surface and reduces sensitivity. Both Gluma Desensitizer and ED Primer contain HEMA, which is characterised by a good penetration into the dentin tubules. These substances produce a resin-reinforced layer of dentin, which in turn is assumed to be responsible for the improvements in shear bond strength, as previously noted [29, 30].

Self-adhesive resin cements have a positive effect on bond strength values on dentin. Higher bond strength with self-adhesive resin cements combined with Gluma Desensitizer was achieved than with conventional resin cements (Panavia21) combined with Gluma Desensitizer [31]. The conventional resin cement, Panavia21, shows excellent bond strength to dentin [8, 32]. It has been shown that when Panavia21 with self-adhesive ED Primer was combined with Gluma Desensitizer, a significant reduction of the shear bond strength values occurred [31, 33, 34]. It is assumed that in soluble desensitizers, the ED Primer reacts directly with dentin, but the desensitizer containing resin blocks the reaction with dentin [33].

The aim of this study was to investigate the long-term effect of Gluma Desensitizer in combination with one conventional resin cement and two self-adhesive resin cements on the bond strength of zirconia crowns bonded to dentin. The primary hypothesis tests whether the initial tensile strength of self-adhesive resin cements combined with Gluma Desensitizer compared to conventional resin cement is similar or not. The secondary hypothesis tests whether the tensile strength of self-adhesive resin cements combined with Gluma Desensitizer shows better long-term stability compared to conventional resin after 1.2 million chewing cycles or not.

Materials and method

Two self-adhesive resin cements, RelyX Unicem (RXU) and G-Cem (GCM), and the conventional resin cement Panavia 21 (PAN) were tested in this study (Table 1). Pull-off test was used to measure tensile strength. Zirconia crowns (n=144) were milled. The zirconia surface was pretreated according to the manufacturer's instruction on the coresponding adhesive cement. Gluma Desensitizer was used for desensitising the dentin according to the manufacturer's instruction (Table 2). The resin cements were tested in combination with (PAN-G, RXU-G, GCM-G) and without (PAN, RXU, GCM) Gluma Desensitizer pretreatment before and after the chewing simultation (Fig. 1).

Specimen preparation

For this in vitro study, 144 extracted caries-free molars were collected in our clinic. The collected teeth were cleaned from periodontal tissue residues with a scaler, stored in 0.5% Chloramin T at room temperature for a maximum of 7 days and then stored in distilled water at 5°C for a maximum of 6 months [35].

Table 1 Summary of products used

Cement systems	Short name	Company	Lot no.
Panavia21	PAN	Kuraray Dental Co Ltd., Osaka, Japan	00406C UNI TC/00647C CAT
Clearfil Porcelain Bond Activator			00208B
Clearfil SE Bond Primer			00769A
RelyX Unicem Aplicap	RXU	3M ESPE , Seefeld, Germany	352388
RelyX Ceramic Primer			5WM
G-CEM Capsule	GCM	GC, Leuven, Belgium	803061
GC Ceramic Primer A			0901272
GC Ceramic Primer B			0901232
Gluma Desensitizer	G	Haereus Kulzer, Hanau, Germany	20088

All teeth were embedded with acrylic resin (Scandiquick, SCAN DIA, Hagen, Germany) parallel to the tooth axis in a special holding device with a cylindrical form presenting a hole in the middle to embed the tooth. The teeth were prepared for zirconia crowns with a motorised parallelometer (PFG 100, Cendres Métaux, Biel-Bienne, Switzerland), conicity of 10° and shoulder preparation with a 40-µm diamond dental bur (FG 305L/6, Intensiv SA, Grancia, Switzerland). To get a standardised coronal height of 3 mm, the holding device was positioned in a cutoff grinding machine (Accutom-50, Struers GmbH, Ballerup, Denmark). The edges of the coronal were rounded with a polishing disc (Sof-Lex 1982C/1982M, 3M ESPE). At the end of the preparation, every tooth had a height of 3 mm, a flat surface, a conicity of 10° and a shoulder preparation.

In order to calculate tensile strength, the prepared abutments were scanned with a Cerec 3D camera (Sirona, Bensheim, Germany) and the dentin surface area was calculated with the Cerec 3 Volume Program (Cerec Software 2.80 R2400 Volume Difference, Sirona; Fig. 2). Crowns with a thickness of 1.5 mm designed by the Cerec 3 InLab Program (3D Program version 3.10, Sirona) were produced. The zirconia crowns were milled (InLab MC XL milling machine, Sirona) in white state (Vita In-Ceram YZ-20/19; LOT30030, Vita Zahnfabrik, Bad Säckingen, Germany). In order to get more retention space for the acrylic resin, a groove of 1-mm depth was drilled (steel bur, Densply, Konstanz, Germany) into the zirconia crowns before sintering (LHT 02/16, Nabertherm GmbH, Lilienthal/Bremen, Germany) according to the manufacturer's instructions.

Then, the prepared teeth (N=144) were randomly divided into the 12 groups (n=12) corresponding to cements, pretreatment and ageing procedures (Fig. 1).

Bonding procedure

The zirconia crowns were cemented with PAN, RXU and GCM (Table 2). The zirconia surface was primed according to the manufacturer's instruction (Table 2). Within the the three cement groups, the teeth were divided into two subgroups and one per cement was additionally pretreated with Gluma Desensitizer (Fig. 1). The Gluma Desensitizer

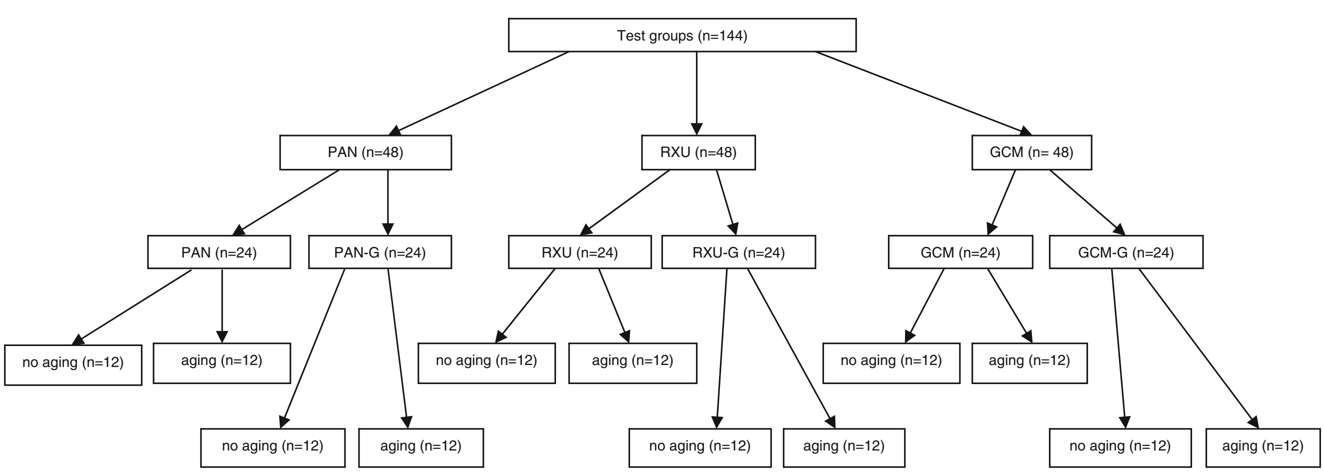

Fig. 1 Study design. Involved cements, their pretreatment and ageing

Fig. 2 Calculation of the dentin surface using the Cerec 3 Volume Program

was applied onto the dentin for 60 s before cementation and dried with air (Table 2). During the setting time of the cements, the specimens were stored in an incubator for 10 min at 37°C and loaded in the special device with 100 N. After the bonding procedure was completed, the inital tensile strength was tested in half of the specimens in the six groups (PAN, PAN-G, RXU, RXU-G GCM, GCM-G) and the other half subjected to simulated ageing (Fig. 1).

Chewing simulation

The ageing was performed with a chewing machine (custom-made device at the University of Zurich). The specimens were mechanically loaded with 49 N for 1.2 million times by the antagonist at the frequency of 1.7 Hz. Simultaneous thermocycling was achieved by changing the surrounding water temperature in the sample chamber every 120 s from 5°C to 50°C. In total, the temperature changed 6,000 times during the occlusal loading [36]. A special holder was screwed into the holding devices to position the specimens in the chambers. Palatinal cusps from nearly identical upper human molars fixed in amalgam acted as antagonists.

Tensile strength measurement

To embed the crowns in the upper holding devices and to position the lower holding devices parallel and with a space of 1.5 mm between each other, the space between the lower holding devices was filled with Lab Putty (Coltène/Whaledent AG, Altstätten, Switzerland). In addition, acryl resin was inserted through the screw hole at the bottom of the holding device. The polymerisation of the acrylic resin was carried out in the polymerisation pressure pot (30 min, 45°C, 2.5 bar, Ivomat, Ivoclar Vivadent, Schaan, Liechtenstein).

The specimens were fixed with a screw at the upper and lower holding device in the Universal Testing Machine (Zwick/Roell Z010, Zwick, Ulm, Germany) and were pulled with a crosshead speed of 1 mm/min until the two holding devices disconnected (Fig. 3). The measurement was stopped as soon as the tensile load decreased by 10% of the maximum load (F_{max}). The load at debonding was recorded and the tensile strength was calculated with the following formula: failure load (N)/bond area (mm^2)=MPa.

Failure types

Four failure types were observed (Fig. 4): (1) failure in the interface of dentin and cement, (2) mixed failure, (3) failure in the interface of zirconia crown and cement and (4) failure in the coronel or root. The failure types were observed by one operator under a optical microscope (M3M, Wild, Heerbrugg, Switzerland, ×25) and photos were made (SEM, Tescan Vega TS 5136 XM, Elektronen-Optik-Service GmbH, Dortmund, Germany) to collect more detailed information on the observed failure types.

Statistical analysis

Statistical Package for the Social Sciences version 15 (SPSS Inc., Chicago, IL, USA) was used to calculate descriptive statistics (mean and SD) and 95% confidence intervals (95% CI) for means of tensile strength. Two-way analysis of variance (ANOVA) for tensile strength with respect to ageing (initial/ageing) and to the test groups was conducted. To observe significant interaction ($p<0.05$) between the test groups, one-way ANOVA for tensile strength followed by a Scheffé post hoc test was applied for each group separately for the subgroups “initial” and “ageing”. The influence of ageing within the groups was compared with a two-sample Student’s t test.

Failure types after debonding were presented in a contingency table with 95% CI for relative frequency. A chi-square test was applied to investigate if failure type 4 rates (failure in the coronal or in the root) were different

Table 2 Composition and application steps of the bonding agents and cements

Bonding agent and cement	Composition	Application steps as recommended by the manufacturer
Composition of the bonding agents and cements		
Pretreatment of the dentin		
Panavia21, ED Primer A	MDP, HEMA, water, MASA, accelator, water	1. Mix one drop of ED Primer A with one drop of ED Primer B for 5 s
Panavia21, ED Primer B	MASA, Na-benzene sulfonate, accelator,water	2. Apply on dried dentin, leave 60 s and blow the remnants away, leaving the surface shiny
Panavia 21, cement catalyst	Hydrophobic aromatic dimethacrylate, hydrophibic alipathic dimethacrylate, MDP, fillers, BPO	1. Dispense equal amounts of Panavia21 Catalyst and Universal pastes 2. Slowly turn the dispenser knob one complete turn to the right until it clicks
Panavia 21, cement base	Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, fillers, DEPT, sodium aromatic sulfonate	3. Mix the paste for 20–30 s until a smooth, uniform paste results 4. Oxyguard II to all margins for 3 min remove by rinsing with water
Pretreatment of zirconia		
Clearfil Porcelain Bond Activator	3-Trimethoxysilylpropyl methacrylate, hydrophobic aromatic dimethacrylate	1. Mix one drop of Clearfil Porcelain Bond Activator with one drop of Clearfil SE Bond Primer
Clearfil SE Bond Primer	2-HEMA, 10-MDP, hydrophilic alipathic dimethacrylate, dl-Campherquinone, water, accelerators, dyes and others	2. Apply on enamel and dentin by means of a microbrush 3. Leave 20 s and airbrush gently
RelyX Unicem Aplicap	Powder: glass fillers, silica, calcium hydroxide, self-cure initiators, pigments, lightcure initiators	1. Insert capsule into Activator, press handle and hold for 2–4 s 2. Mix 10 s with RotoMix Capsule Mixing Unit
	Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilisers, self-cure initiators	3. Insert capsule into applier
Pretreatment of zirconia		
RelyX Ceramic Primer	Ethanol, water, methacrylacid-3-trimethoxysilylpropylester	1. Apply a thin layer to the bonding surface of the ceramic and dry with air
G-CEM Capsule	4-META, UDMA, alumino-silicate glass, pigments, dimethacrylates, water, phosphoric ester monomer, initiators, campherquinone	1. Shake the capsule and push the plunger until it flush with the body 2. Place the capsule into an Applier and click the lever once 3. Mix for 10 s 4. Insert capsule into Applier
Pretreatment of zirconia		
GC Ceramic Primer A	Ethanol	1. Mix one drop of GC Ceramic Primer A with one drop of GC Ceramic Primer B for 5 s
GC Ceramic Primer B	Methyl methacrylate, Ethanol, 2-HEMA	2. Apply a thin layer to the bonding surface of the ceramic and dry with an air syringe
Gluma Desensitizer	HEMA, glutaraldehyde, distilled water	1. Apply on dried dentin and leave for 30–60 s 2. Dry and spray with air

BPO benzoylperoxid, *HEMA* 2-hydroxyethyl-methacrylate, *MASA* *N*-methacrylolyl-5-aminosalicylic acid, *MDP* 10-methacrylate oxydecyl dihydrogen phosphate, *4-META* 4-methacryloyloxyethyl-trimellitat-anhydrid, *UDMA* urethane-dimethacrylate

between the test groups with and without ageing. All failures within the tooth (type 4) were categorised as censored measurements. The failure types 1 to 3 were analysed in one group and called non-censored data because a real bond fracture occurred. The Kaplan–Meier estimates of survival and the cumulative distribution function for failure together with the Breslow–Gehan test were computed.

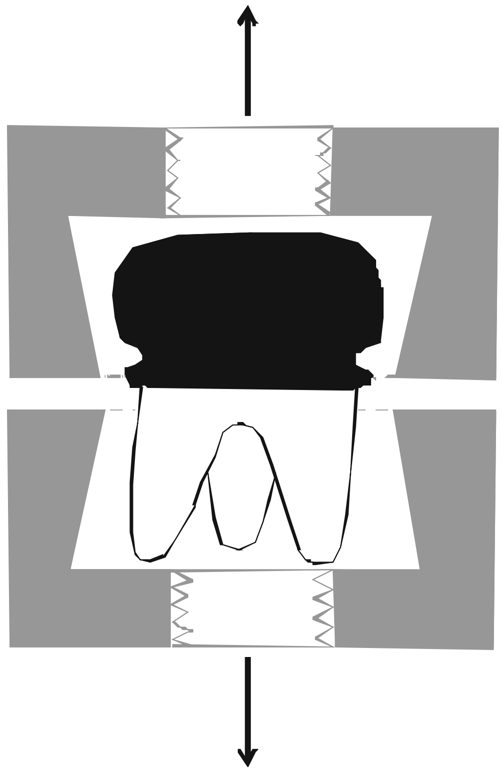

Fig. 3 Pull-out test design

Results of the statistic analyses with p value smaller than 5% were interpreted as statistically significant.

Results

Tensile strength

Table 3 provides descriptive statistic (mean, SD, 95% CI) of the tensile strength for each group.

PAN-G (2.6±1.4 MPa) showed the lowest initial tensile strength. No difference was found between the initial mean tensile strength ranging from 10.7 to 14.1 MPa in the remaining five groups (Fig. 5). After the chewing simulation, a significantly higher mean tensile strength was observed for both self-adhesive resin cements when combined with Gluma Desensitizer (RXU-G, 12.8± 4.3 MPa; GCM-G, 13.4±6.2 MPa) compared to the conventional resin cement Panavia21 (7.3±1.7 MPa). PAN-G showed significantly lower tensile strength (0.9±0.6 MPa).

Considering the impact of ageing within each test group, PAN (initial, 14.1±3.5 MPa; ageing, 7.3±1.7 MPa; $p<0.001$), PAN-G (initial, 2.6±1.4 MPa; ageing, 0.9± 0.6 MPa; $p=0.001$) and RXU (initial, 12.8±2.9 MPa; ageing, 9.1±3.0 MPa; $p=0.006$) showed significantly lower mean tensile strength after ageing (Table 3). GCM followed this trend (initial, 10.7±2.9 MPa; ageing, 8.6±2.2 MPa; $p=0.06$). The two self-adhesive resin cements when combined with Gluma Desensitizer showed similar tensile strength independent of ageing.

Failure types

Table 4 describes the observed frequencies of the different types of failures which were observed after debonding. Uncensored observations correspond to failure modes 1, 2 and 3, where debonding of the crown was observed. Failure mode 4 (fracture of the tooth) is considered to be a censored observation as no debonding of the crown was observed for the whole range of tensile strength applied.

PAN-G groups showed only fracture type 1. Failure type 2 (mix failure) was most frequent within the initial and aged self-adhesive resin cements. No type 3 failure (failure in the interface zirconia crown and cement) was observed.

Failure type 4 occurred in the coronal or in the root when the bond strength of the crowns on dentin was higher than the initial flexural strength of the teeth. In total, failure type 4 occurred nine times initially: two times in the control group PAN, once within self-adhesive resin cements combined with Gluma Desensitizer RXU-G once and GCM-G six times. After ageing, a total of eight type 4 were observed only in self-adhesive resin cements combined with Gluma Desensitizer (RXU-G, 3; GCM-G, 5). Examples of the failure types are shown in Fig. 6.

Kaplan–Meier survival analysis

Significant differences were found in the frequency of failure type 4 between the test groups (initial: $p=0.03$; ageing: $p=0.04$, chi-square test). Table 5 reports the median failure tensile strength given by Kaplan–Meier survival observed in different test groups. It is the tensile strength up to which 50% of the teeth in one particular test group have experienced debonding of the zirconia crown and 50% of the teeth have not experienced debonding of the crown. These estimates are adjusted for censoring. They correspond to the tensile strength for which the estimate of the cumulative failure function crosses the probability of 50% in Figs. 7 and 8.

For each test group separately, the estimated proportion of teeth that experienced debonded crown before a given tensile strength when adjusted for censoring is shown. For each test group separately, the estimated cumulative function of the debonded crown given the tensile strength is presented. These estimates are adjusted for censoring. According to the Breslow–Gehan test, significant differences were found in the initial groups ($p<0.001$, Fig. 7). The lowest tensile strength occurred within the GCM and PAN-G groups in comparison to the remaining groups. The median initial

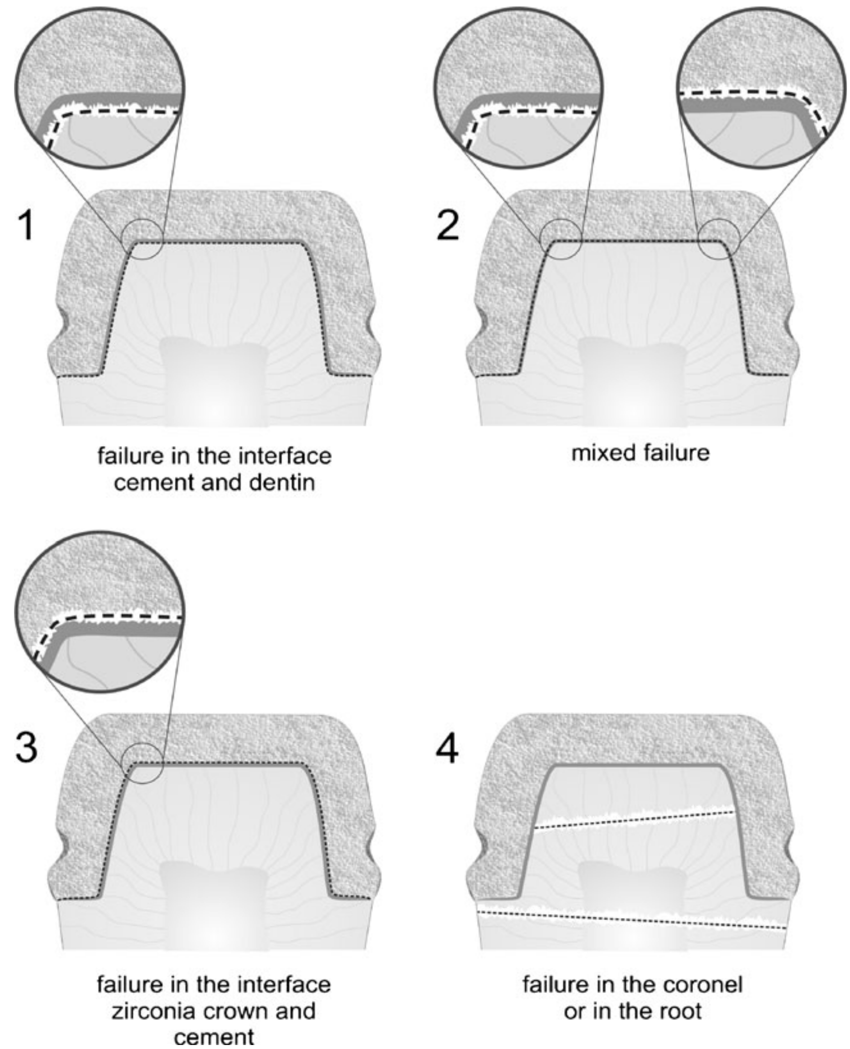

Fig. 4 Classification of failure types 1 to 4

tensile strength for PAN-G (2.1 MPa) was the lowest and statistically different from GCM (9.9 MPa) and PAN (14.5 MPa) and GCM combined with Gluma Desensitizer (15.0 MPa). PAN-G had the poorest survival as the estimated cumulative function of the debonded crown increases very quickly with increasing tensile strength.

Within the chewing simulated groups, significant differences were determined according to Breslow–Gehan test ($p<0.001$, Fig. 8). The median failure tensile strength for PAN-G (0.8 MPa) and PAN (6.7 MPa) was significantly lower than for the self-adhesive resin cements combined with Gluma Desensitizer (RXU, 10.6 MPa; GCM, 14.2 MPa). When GCM and RXU were combined with Gluma Desensitizer, 50% of the specimens debonded at tensile strengths of 14.2 and 10.6 MPa, respectively. The pretreatment of Gluma Desensitizer resulted in

Table 3 Mean, SD and 95% CI of mean tensile bond strenght (MPa) and p value of the two-sample Student's t test between initial and ageing groups

Group	Initial		p value	Ageing	
	Mean (SD, MPa)	95% CI (MPa)		Mean (SD, MPa)	95% CI (MPa)
PAN	14.1 (3.5)B	11.9–16.4	<0.001	7.3 (1.7)b	6.1–8.4
PAN-G	2.6 (1.4)A	1.7–3.6	0.001	0.9 (0.6)a	0.5–1.3
RXU	12.8 (2.9)B	10.9–14.6	0.006	9.1 (3.0)b,c	7.2–11.0
RXU-G	13.1 (2.9)B	11.2–14.9	0.874	12.8 (4.3)c	10.1–15.6
GCM	10.7 (2.9)B	8.8–12.5	0.06	8.6 (2.2)b,c	7.2–10.0
GCM-G	13.7 (4.2)B	11.0–16.3	0.92	13.4 (6.2)c	9.5–17.4

The letters reflect the results from the one-way ANOVA within the same ageing level. Different letters represent a significant post hoc test between the levels of the test groups factor

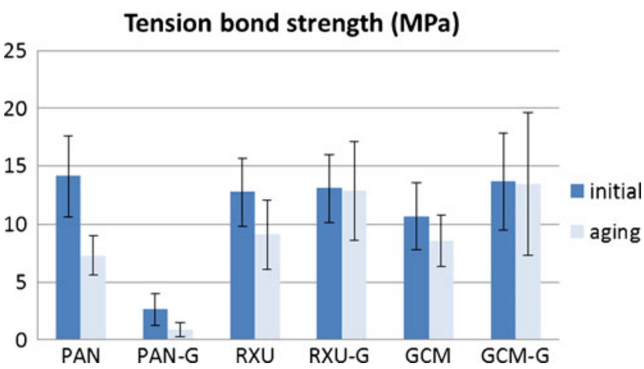

Fig. 5 Diagram of tensile strength (MPa) of initial and chewing simulated groups

significantly higher median failure tensile strength in the RXU and GCM groups. The median tensile strength for GCM combined with Gluma Desensitizer was the highest (14.2 MPa). In summary, PAN-G had the poorest survival as the estimated cumulative function of the debonded crown increases very quickly with increasing load. On the other hand, GMC-G showed the best survival as the estimated cumulative function of debonded crown increases slowly with increasing tensile strength.

Discussion

Tensile strength

The included self-adhesive resin cements, either combined with Gluma Desensitizer or not, exhibited similar initial tensile strength as a conventional resin cement with a dentin primer. This finding supports the fact that the self-adhesive resin cements without any preconditioning of enamel and/or dentin still obtain bond strength values similar to conventional resin cements [5, 14, 18]. The combination of the tested self-adhesive resin cements with Gluma Desensitizer did not impact the initial tensile strength and was similar to that of conventional resin cement. Hence, the first hypothesis of this study was accepted.

After the chewing simulation, both self-adhesive resin cements combined with Gluma Desensitizer exhibited better bonding performance than the conventional resin cement and better long-term stability compared to the self-adhesive resin cements without the pretreatment of Gluma Desensitizer. The tensile strength of the conventional resin cement and of the self-adhesive resin cements without the pretreatment of Gluma Desensitizer showed lower tensile strength after chewing simulation values. The findings of the present in vitro study showed that the desensitisation of dentin with Gluma Desensitizer had a positive effect on long-term tensile strength of the self-adhesive resin cements; therefore, the secondary hypothesis was accepted.

The present study tested the impact of the application of Gluma Desensitizer with two self-adhesive resin cements and compared the results obtained in the control group Panavia21. The conventional resin cement combined with Gluma Desensitizer showed very low results compared with the self-adhesive resin cements. Several studies reported that the desensitisation of dentin had no impact on the bond strength of conventinal resin cements to human and/or bovine dentin [37–39]. Three other studies reported a

Table 4 Relative frequencies with 95% confidence interval for relative frequency of failure types for all groups after debonding

Failure mode	Decementing of the crown				Failure in the tooth (end of measuring)	
	1 (freq.)	2 (freq.)	3 (freq.)	1–3 (rel. freq., %) (95% CI)	4 (frequency)	4 (rel. freq., %) (95% CI)
Initial						
PAN	1	9	0	83.3 (51.5–97.9)	2	16.7 (2.0–48.4)
PAN-G	12	0	0	100 (73.5–100)	0	0 (0.26–26.5)
RXU	0	12	0	100 (73.5–100)	0	0 (0–26.5)
RXU-G	1	10	0	91.7 (61.5–99.8)	1	8.3 (0.2–38.5)
GCM	1	11	0	100 (73.5–100)	0	0 (0–26.5)
GCM-G	0	6	0	50 (21.0–78.9)	6	50 (21.0–78.9)
Ageing						
PAN	2	10	0	100 (73.5–100)	0	0 (0–26.5)
PAN-G	12	0	0	100 (73.5–100)	0	0 (0.26–26.5)
RXU	0	12	0	100 (73.5–100)	0	0 (0–26.5)
RXU-G	0	9	0	75 (42.8–94.5)	3	25 (5.4–57.2)
GCM	0	12	0	100 (73.5–100)	0	0 (0–26.5)
GCM-G	0	7	0	58.3 (27.6–84.8)	5	41.7 (15.1–72.3)

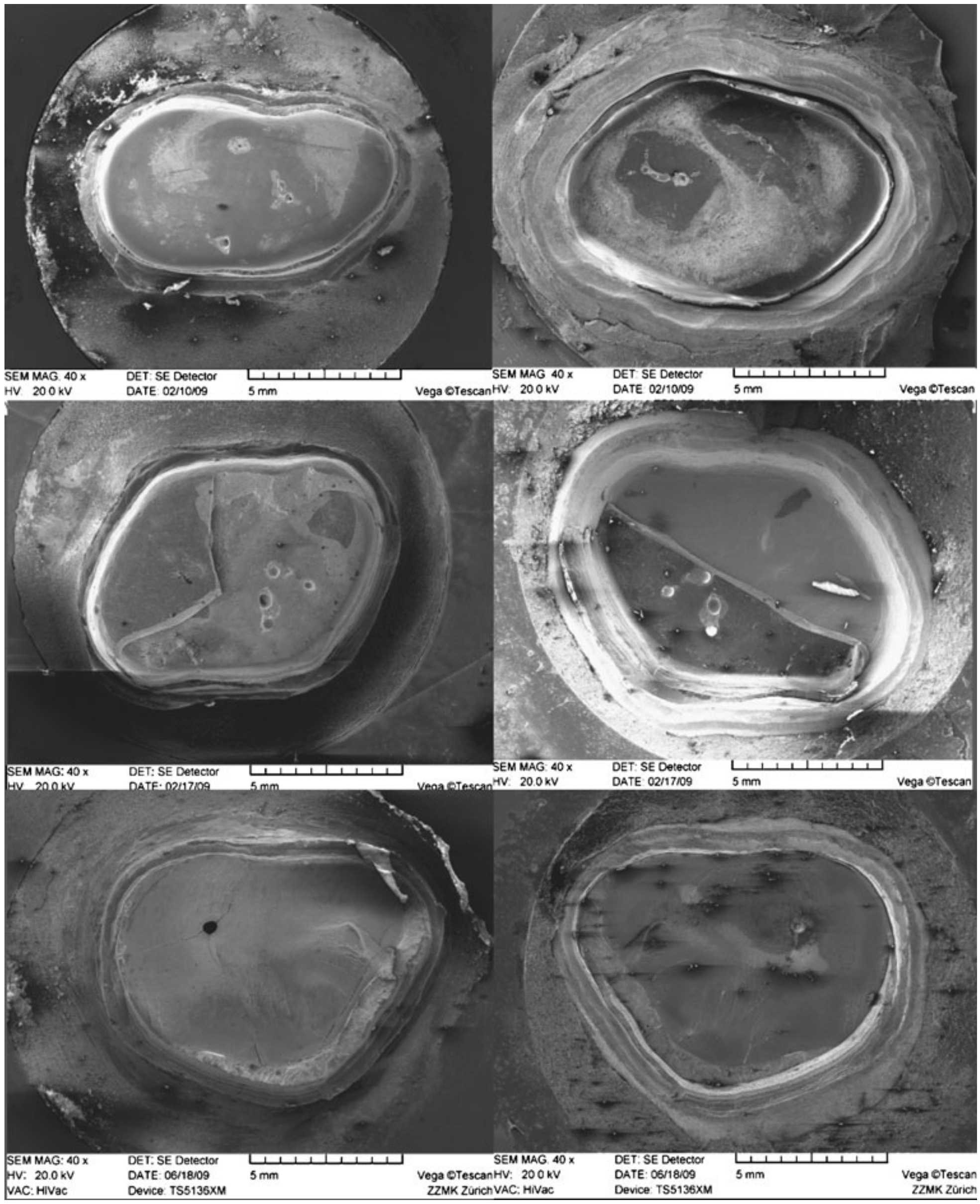

Fig. 6 SEM picture: failure type 1—failure in the interface dentin and cement; type 2—mixed failure; and type 4—failure in the coronel or root

negative effect of the desensitizer on the bond strength of the conventional resin cement Panavia21 [31, 33, 34]. It was stated that the resin cement was not able to polymerize with the dentin desensitizer [34].

The long-term tensile strength of self-adhesive resin cements tended to be positively influenced by the application of desensitizers. It is hypothesised that the bond strength of self-adhesive resin cements and the desensitizers, and between the desensitizers and dentin, exceeded the bond strength of self-adhesive resin cement and dentin itself [31]. This might be due to the fact that Gluma Desensitizer contains glutaraldehyde and HEMA which provides hydrophilic properties to improve the bonding to hydrophilic dentin. Self-adhesive resin cements contain phosphate groups to improve the bonding to dentin. The positive observations regarding Gluma Desensitizer in this study may be be explained by a condensation reaction between HEMA and phosphate through the elimination of water.

Failure types

The frequency of failure within the dentin (type 4) for the self-adhesive resin cements combined with Gluma Desensitizer in both initial and aged groups was not expected. Self-adhesive resin cements applied without Gluma Desensitizer showed no type 4 failures. Within the control group, Panavia21 showed only two failures within the dentin without ageing; the reduced lower tensile strength after ageing resulted in a different occurrence of failure types. The frequency of the failure within the dentin could be the result of a higher tensile strength compared to the internal strength of the tooth.

Table 5 Median survival tensile strength (MPa) and 95% confidence interval of survival in all test groups

Group	Initial median (95%CI, MPa)	Ageing median (95% CI, MPa)
PAN	14.5 (13.2–15.8)	6.7 (5.0–8.4)
PAN-G	2.1 (0.4–3.7)	0.8 (0.4–1.2)
RXU	12.2 (9.0–15.3)	7.8 (4.6–11.1)
RXU-G	13.9 (9.6–18.3)	10.6 (8.8–12.3)
GCM	9.9 (5.9–13.8)	8.8 (5.5–12.1)
GCM-G	15.0 (11.1–18.7)	14.2 (8.8–19.6)

In the literature, one study tested the tensile strength in a pull-off test and observed failure types in the tooth of a few specimens cemented with Panavia21 [34]. Another study reported that most of the remaining cements were found inside the gold alloy crowns (adhesive failure in the cement–dentin interface) [40]. Moreover, Palacios et al. [41] found failure within the dentin after tensile strength measurement, whereby all results were included in the statistic ANOVA.

The reason for the absence of failure type 3 (failure in the interface zirconia crown and cement) in the present study might be explained by the fact that the bond strength of self-adhesive cements with phosphate monomers and zirconia is adequate, which has been documented elsewhere [41–43].

Kaplan–Meier survival analysis

Failures in the tooth (failure type 4) were categorised as a censored event because including type 4 failures into the analysis underestimates the true tensile strength. Failure types 1–3 (decementing of the crown) were non-censored. By using the survival analysis, the Kaplan–Meier estimates of survival and the cumulative distribution function for failure, as well as the Breslow–Gehan test, were computed for the tension bond strength of non-censored and censored observations.

By using the Kaplan–Meier estimates of survival, the initial tensile strength of PAN-G and GCM was statistically significantly lower than in PAN and GCM combined with Gluma Desensitizer. However, analysing the complete data with ANOVA, no differences between the groups were observed. The reason for these different results is based on the censored data for specimens with failure type 4: PAN two times, RXU-G once and GCM-G six times. The pretreatment of Gluma Desensitizer resulted in both statistical analysis to high long-term tensile strength of self-adhesive resin cements.

Evaluation of the test method

This research used the pull-off test using prepared human teeth where zirconia crowns were bonded according to standard clinical procedures. However, the teeth were prepared manually and the water supply was not controlled with the handpiece as under clinical conditions.

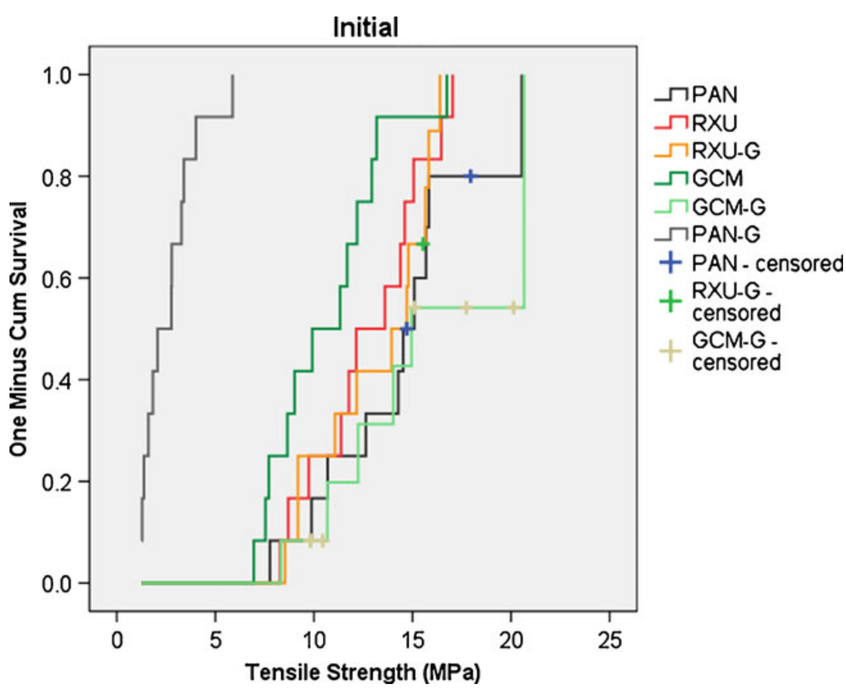

Fig. 7 Cultimative distribution function for failure with respect to initial tensile strength (MPa) by Kaplan–Meier

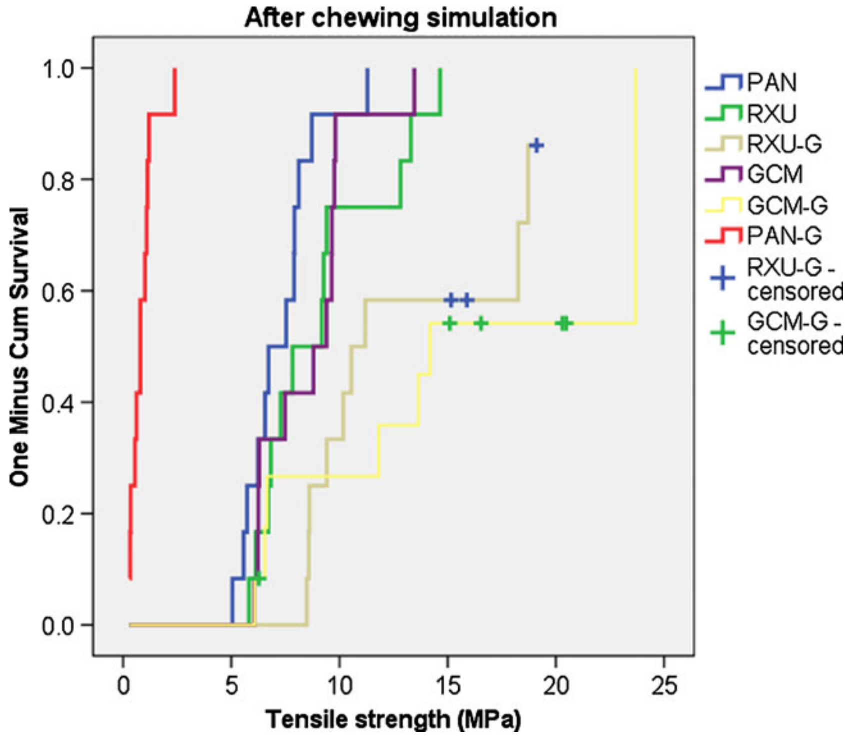

Fig. 8 Cultimative distribution function for failure with respect to tensile strength (MPa) after chewing simulation by Kaplan–Meier

The advantage of this study using pull-out-tests is the integration of the surface bond area calculation where the prepared abutments were scanned with Cerec 3D camera and their areas calculated with the Cerec 3 Volume Program. It can be assumed that the applied method presents precise results than previously published data. Ernst et al. [40, 42] determined the bond area by wrapping 0.1 mm of tinfoil around the preparation, determining the weight of the foil. Yim et al. and Palacios et al. [34, 41] used standardised crown preparations and the specimens bond area was calculated using the formula for a truncated cone to which the area of the flat occlusal surface was added.

In our study, the specimens were subjected to a chewing simulation where the stress for all specimens was standardised and reproducible. The use of especially developed loading machines with additional artificial ageing through thermocycling is a well-proven and established method to simulate the clinical situation [39, 44, 45]. It is claimed that the chewing simulation of 1.2 Mio cycles corresponds to 5 years in vivo [46, 47]. However, this assumption has not yet been systematically verified with different materials and is only based on the extrapolation of 4-year clinical wear data on amalgam fillings and 6-month data of composite inlays [46, 47]. This correlation was only used for the measurements of abrasion stability. In summary, more longitudinal clinical ageing data are still needed. At the time, only trends and indications as to the true extent of ageing can be obtained.

One possible reason for the observed variations of the bond strength values could be the quality of the human teeth. It has been demonstrated that the bond strength of resin cements is dependent on the micromorphology of the dentin that is used for the bond strength test [48]. Another limitation of this study was the use of extracted teeth which probably caused some loss of dentin fluid protein, and such an environment could have prevented Gluma Desensitizer from reacting with dentin fluid protein.

Clinical relevance

Gluma Desensitizer is normally recommended to be used under restorations to reduce postoperative sensitivity, after the dentinal smear layer removal and before cementation procedures. So far, it has not been found to affect bond strength values of self-adhesive resin cements [31, 38, 39]. The long-term stability of the tensile strength of self-adhesive resin cements combined with Gluma Desensitizer showed better results than conventional resin cement without and with Gluma Desensitizer.

Conclusions

Within the limitations of this in vitro study, it can be concluded that:

1. The tested self-adhesive resin cements reached the intial tension bond stregth of conventional resin cement.

2. The tensile strength of self-adhesive resin cements combined with Gluma Desensitizer showed better long-term stability compared to conventional resin cement after 1.2 million chewing circles.
3. Panavia21 with Gluma Desensitizer resulted in higher number of adhesive failures between the dentin and the cement before and after ageing.
4. G-Cem with Gluma Desensitizer resulted in the highest number of cohesive fracture in the teeth before and after ageing followed by RelyX Unicem combined with Gluma Desensitizer.

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Conflict of interest The authors declare no conflicts of interest.

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