Universität Zürich
Zentrum für Zahn-, Mund- und Kieferheilkunde
Vorsteher: Prof. Dr. med. dent. C.H.F Hämmerle

Klinik für Kronen- und Brückenprothetik,
Teilprothetik und zahnärztliche Materialkunde
Direktor: Prof. Dr. med. dent. C.H.F. Hämmerle

Arbeit unter der Leitung von Dipl. Ing. B. Stawarczyk, MSc

Impact of Gluma Desensitizer on the Tensile Strength of Zirconia Crowns bonded to Dentin: An in-vitro Study

INAUGURAL-DISSERTATION
zur Erlangung der Doktorwürde der Zahnmedizin
der Medizinischen Fakultät
der Universität Zürich

vorgelegt von
Leonie Hartmann
von Luzern (LU)

Genehmigt auf Antrag von Prof. Dr. med. dent. C.H.F. Hämmerle
Zürich 2010

Table of Contents

1. Abstract

Objectives: This study tested the impact of Gluma Desensitizer on the tensile strength of zirconia crowns bonded to dentin. **Methods:** Human teeth were prepared and randomly divided into 5 groups (N=120, n=24 per groups). For each tooth a zirconia crown was manufactured. The zirconia crowns were cemented with: i) Panavia21 (control group), ii) RelyX Unicem, iii) RelyX Unicem combined with Gluma Desensitizer, iv) G-Cem, v) G-Cem combined with Gluma Desensitizer. The initial tensile strength was measured in half (n=12) of each group and the other half (n=12) was subjected to a chewing machine (1.2 Mio, 49N, 5°C/50°C). The cemented crowns were pulled in a Universal Testing Machine (1 mm/min, Zwick Z010) until failure occured and the 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 (alpha = 0.05). **Results:** After chewing simulation, the self-adhesive resin cements combined with Gluma Desensitizer showed significantly higher tensile strength (RelyX Unicem: 12.8 ±4.3 MPa, G-Cem: 13.4 ±6.2 MPa) than the control group Panavia21 (7.3 ±1.7 MPa). Within the groups, Panavia21 and RelyX Unicem resulted in significantly lower values when compared to the initial tensile strength; the values of all other test groups were stable. **Conclusion:** In this study self-adhesive resin cements combined with Gluma Desensitizer reached better long-term stability compared to control group after chewing simulation.

2. Introduction

The utilization of all-ceramic reconstructions is increasing, based on adequate mechanical stability [1-3], high aesthetic properties, and most importantly high biocompatibility [4]. Two different types of ceramic are currently in use: glass-ceramic and oxide-ceramics, e.g. zirconia.

Glass-ceramic reconstructions are adhesive cemented to the tooth. By using a resin cement, the stability and the clinical long-term success are improved [5-8]. Resin cements chemically bond to both - the ceramic crown and the tooth substance, and thereby reinforce the tooth-reconstruction [5,6,9-13]. Furthermore, the high translucency and a tooth-resembling colour improve the aesthetic result [13]. Zirconia can be cemented traditionally (e.g. glass ionomer cement) or with resin cements. The main advantage of adhesive cemented reconstructions is a reduced margin microleakage [14,15]. Significant higher bond strength values were obtained when zirconia was bonded with resin cement containing an adhesive phosphate monomer compared to resin cement based on Bis-GMA monomers [16,17].

The sensitivity to moisture [5,14,18] of resin cements which requires the application of a dental dam, complicates the clinical utilization. Self-adhesive resin cements are simple to use and more efficient in handling [14]. These partly hydrophilic resin cements do not require any pretreatment of the tooth [5,14,18]. The key difference between a self-adhesive resin cement and a conventional resin cement 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 the dentin, since their adhesion mechanism is based on the partial retention of 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 a pretreatment of desensitizer. It has been reported that the sealing of dentin also decreases the sensitivity of a prepared tooth, resulting in less post-operative pain [22-25].

Gluma Desensitizer (5% Glutaralaldehyd) reduces dentin permeability resulting in a reduction of dentin sensitivity and a disinfection of the dentin [26,27]. The diffusion of monomers into dentin is likely to be accelerated by HEMA [28]. As soon as the dentin tubules are closed, the hydrodynamic 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 desensitization the obliteration of the dentin is relevant. Panavia21 with the dentin pre-treatment (ED Primer) seals the dentin surface and reduces the sensitivity. Both systems Gluma Desensitizer and ED Primer contain HEMA, which is characterized by a good penetration into the dentin tubules resulting in a resin reinforced layer of dentin. This, in turn, is assumed to be responsible for the previously noted improvements in shear bond strength [29,30].

Self-adhesive resin cements have a positive effect on bond strength values to dentin. Higher bond strength has been achieved with self-adhesive resin cements combined with Gluma Desensitizer 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. However, desensitizers containing resin block the reaction with dentin [33].

The aim of this study was to investigate the long-term effect of Gluma Desensitizer in combination with self-adhesive resin cements on the bond strength of zirconia crowns bonded to dentin. The primary hypothesis which was tested is that the initial tensile strength of self-adhesive resin cements combined with Gluma Desensitizer is similar to that from a conventional resin cement. The secondary hypothesis which was tested is that the tensile strength of self-adhesive resin cements combined with Gluma Desensitizer shows better long-term stability compared to conventional resin cements after 1.2 million chewing cycles.

3. Materials and methods

Two self-adhesive resin cements; RelyX Unicem (RXU) and G-Cem (GCM), and the conventional resin cement Panavia 21 (PAN) as control were tested in this study. Pull-out tests were used to measure the tensile strength of the bond. Zirconia crowns (n=120) were milled. The zirconia surface of the crown was pretreated according to the manufacturer’s instruction of the corresponding adhesive cement. Gluma Desensitizer was used for desensitizing the dentin according to the manufacturer’s instruction (Table 1). Both self-adhesive cements were tested in combination with (RXU-G, GCM-G) and without (RXU, GCM) Gluma Desensitizer pretreatment both before and after the chewing simulation (Fig. 1).

3.1 Specimen preparation

For this in-vitro study, 120 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 preserved in distilled water at 5°C for a maximum of six months [35].

All teeth were embedded in an 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 motorized parallelometer (PFG 100, Cendres Métaux, Biel-Bienne, Switzerland); conicity of 10° and shoulder preparation with a 40 µm diamonded dental bur (FG 305L/6, Intensiv SA, Grancia, Switzerland). To get a standardized coronal height of 3 mm, the holding device was positioned in a cut-off 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 the tensile strength, the prepared abutments were scanned with a Cerec 3D camera (Sirona, Bensheim, Germany) and the dentin surface area 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=120) were randomly divided into the ten groups (n=12) corresponding to cements, pretreatment, and aging procedures (Fig. 1).

3.2 Bonding procedure

The zirconia crowns were cemented with PAN, RXU and GCM (Table 2). The zirconia surface was primed according to the manufacturer’s instructions (Table 2). Within each of the RXU and GCM group, the teeth were divided into two sub-groups. One sub-group per self-adhesive cements was additionally pretreated with Gluma Desensitizer (Fig. 1). The Gluma Desensitizer 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 initial tensile strength was tested in half of the specimens in the five groups (control, RXU and GCM with and without Gluma) whilst the other half was first subjected to simulated aging (Fig. 1).

3.3 Chewing simulation

The aging 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 a frequency of 1.7 Hz. Simultaneously, the sample was subjected to thermocycling by changing the surrounding water temperature between 5°C and 55°C in the sample chamber every 120 s. In total, the temperature changed 6.000 times during the occlusal loading [37]. A special holder was screwed into the holding devices to position the specimens in the chambers. Palatinal cusp from nearly identical upper human molars fixed in amalgam acted as the antagonist.

3.4 Tensile strength measurement

To embed the crowns in the upper holding devices and to position the lower holding devices parallel, but 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 polymerization of the acrylic resin was carried out in a polymerisation pressure pot (30min, 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 cross head speed of 1 mm/min until the bonding broke, and the two holding devices became disconnected (Fig. 3). The measurement was stopped when the tensile load decreased by 10% from the maximum load (Fmax). The load at debonding was recorded and the tensile strength was calculated with the following formula: tensile load = failure load (N) / bond area (mm^2) (units of N / mm^2 = MPa).

3.5 Failure types

Four failure types were observed (Fig. 4): i) failure in the interface of dentin and cement, ii) mixed failure, iii) failure in the interface of zirconia crown and cement, and iv) failure in the coronel or root. The failure types were observed by one operator under an optical microscope (M3M, Wild, Heerbrugg, Switzerland) (x25) 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.

3.6 Statistical analysis

The Statistical Package of the Social Science Version 15 (SPSS INC, Chicago, IL, USA) was used to calculate descriptive statistics (mean and standard deviations (SD)) and 95% confidence intervals (95% CI) for the tensile strength. Two-way ANOVA for tensile strength with respect to aging (initial/aging) 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 “aging”. The influence of aging 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^2 test was applied to investigate if the failure type 4 rates (failure in the coronel or in the root) were different between the test groups with and without aging. 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 the survival and the cumulative distribution function for failure together with the Breslow-

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

4. Results

4.1 Tensile strength

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

No statstically significant difference in the initial mean tensile strength ranging from 10.7 to 14.1 MPa between the five groups was observed (Fig. 6). After chewing simulation and aging, 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).

Considering the impact of aging within each test group, the control group PAN (initial: 14.1 ± 3.5 MPa ; aging: 7.3 ± 1.7 MPa; $p<0.001$) and the test group RXU (initial: 12.8 ± 2.9 MPa; aging: 9.1 ± 3.0 MPa; $p= 0.006$) showed a significantly lower tensile strength after aging (Table 3). GCM followed this trend (initial: 10.7 ± 2.9 MPa; aging: 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 aging.

4.2 Failure types

The frequency of the failure type 1 to 4 with 95% CI is presented in Table 4. Failure type 2 (mix failure) was the most frequently observed type. 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 flexure strength of the teeth. In total, failure type 4 occurred 9 times initially: 2 times within in the control group PAN and within self-adhesive resin cements combined with Gluma Desensitizer RXU-G once and GCM-G six times. After aging, a total of 8 failures type 4 were found only with self-adhesive resin cements combined with Gluma Desensitizer (RXU-G: 3; GCM-G: 5). Examples of the occuring failure types are shown in Figure 5.

4.3 Kaplan-Meier Survival Analysis

Significant differences were found in the frequency of failure type 4 between the test groups (initial: p=0.03; aging: p=0.04) using the Chi^2 test. The median failure tensile strength given by Kaplan-Meier survival analysis of the initial and aging values are shown in Table 5.

According to the Breslow-Gehan test significant differences were found in the initial groups (p=0.013) (Fig. 7). Within the GCM group - in comparison to the remaining groups - the lowest failure tensile strength (until the debonding of the ZrO_2 crown) occurred. The median initial tensile strength for GCM (9.9 MPa) is the smallest one and is statistically different from PAN (14.5 MPa) and GCM combined with Gluma Desensitizer (15.0 MPa).

Within the chewing simulated groups, significant differences, according to Breslow-Gehan test (p<0.001) (Fig. 8), were determined. The median failure tensile strength for 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 strength of 14.2 MPa and 10.6 MPa, respectively. The pretreatment of Gluma Desensitizer resulted into a significantly higher median failure tensile strength in the RXU and GCM group. The median failure tensile strength for GCM combined with Gluma Desensitizer was the highest one (14.2 MPa).

5. Discussion

5.1 Tensile strength

The tested 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 other studies which show 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 significantly impact the initial tensile strength and was similar to that of conventional resin cement (Panavia21). 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 desensitization of dentin with Gluma Desensitizer had a positive effect on long-term tensile strength of the self-adhesive resin cements and, therefore, the secondary hypothesis was accepted.

The present study tested the impact of the application of Gluma Desensitizer on two self-adhesive resin cements and compared the results to those of the control group Panavia21. Several studies reported that the desensitization of dentin had no impact on the bond strength of conventional resin cements to human and/or bovine dentin [37-39]. Three other studies reported a 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 appplication of desensitizers. It is hypothesized 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 explained by a condensation reaction between HEMA and phosphate through the elimination of water.

5.2 Failure types

The frequency of failure within the dentin (type 4) within the self-adhesive resin cements combined with Gluma Desensitizer in both initial and aged groups was unexpected. Self-adhesive resin cements applied without Gluma Desensitizer showed no type 4 failures. Within the control group Panavia21, only two failures within the dentin occurred without aging; the reduced lower tensile strength after aging resulted in a different occurrence of failure types. The frequency of failure within the dentin could be the result of a higher tensile strength compared to the internal strength of the tooth.

In the literature, one study tested the tensile strength in pull-out 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 found failure within the dentin after tensile strength measurement, whereby all results were included in the statistic analysis of variance (ANOVA) [41].

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. This has been documented elsewhere [41-43].

5.3 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. The failure types 1 to 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 GCM was statistically significantly lower than in the control group (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 speciments with failure type 4; PAN: 2x, RXU-G: 1x, and GCM-G: 6x. The pretreatment of Gluma Desensitizer resulted with both statistical analysis techniques in higher long-term tensile strength of self-adhesive resin cements.

5.4 Evaluation of the test method

This research used the pull-out 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.

The advantage of this study, using a pull-out-test, is the integration of the surface bond area calculation, where the prepared abutments were scanned with Cerec 3D camera and their areas were calculated with the Cerec 3 Volume Program. It can be assumed that the applied method presents more precise results than previous published data. Ernst et al determined the bond area by wrapping 0.1 mm of tinfoil around the preparation determining the weight of the foil [40,42]. Yim et al and Palacios et al used standardized 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 [34,41].

In our study, the specimens were subjected to chewing simulation, where the stress for all specimens was standardized and reproducible. The use of specially developed loading machines with additional artificial aging through thermocycling is a well-proven and established method to simulate the clinical situation [39,44,45]. It is claimed that our 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-months data of composite inlays [46,47]. This correlation was only used for the measurements of abrasion stability. In summary, more longitudinal clinical aging data are still needed. At the current time, only trends and indications of the true impact of aging 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 reaction with dentin fluid protein.

5.5 Clinical relevance

Gluma Desensitizer is normally recommended for the use under restorations to reduce postoperative sensitivity, after the dentinal smear layer has been removed and before cementation procedures. But 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 tensile bond strength of self-adhesive resin cements combined with Gluma Desensitizer showed better results than conventional resin cement.

6. References

1. Sturzenegger B, Fehér A, Lüthy H, Schärer P, Gauckler LJ (2001) Reliability and strength of all-ceramic dental restorations fabricated by direct ceramic machining (DCM). Int J Comp Dent 4:89-106.
2. Lüthy H, Filser F, Loeffel O, Schumacher M, Gauckler LJ, Hämmerle CHF (2005) Strength and reliability of four unit all-ceramic posterior bridges. Dent Mater 21:930-937.
3. Fischer J, Stawarczyk B (2007) Compatibility of machined Ce-TZP/Al2O3 nanocomposite and a veneering ceramic. Dent Mater 23:1500-1505
4. Piconi C, Maccauro G (1999) Zirconia as a ceramic biomaterial. Biomaterials 20: 1-25.
5. Abo-Hamar SE, Hiller KA, Jung H, Federlin M, Friedl KH, Schmalz G (2005) Bond strength of a new universal self-adhesive resin luting cement to dentin and enamel. Clin Oral Invest 9:161-167.
6. Bindl A, Richter B, Mörmann WH (2005) Survival of ceramic computer-aided design/manufacturing crowns bonded to preparations with reduced macro-retention geometry. Int J Prosthodont 18:219-244.
7. Burke FJT, Fleming GJP, Nathanson D, Marquis P (2002) Are adhesive technologies needed to support ceramics? An assessment of the current evidence. J Adhes Dent 4:7-22.
8. Stewart GP, Jain P, Hodges J (2002) Shear bond strength of resin cements to both ceramic and dentin. J Prosthet Dent 88:277-284.
9. Burke FJT, Qualthrough AJE, Hale RW (1998) Dentin-bonded all-ceramic crowns: current status. J Am Dent Assoc 129:455-460.

10. Knobloch LA, Kerby RE, Seghi R, Berlin JS, Lee JS (2000) Failure toughness of resin-based luting cements. J Prosthet Dent 83:204-209.
11. Mitchell CA, Douglas WH, Cheng YS (1999) Failure toughness of conventional, resin-modified glass-ionomer and composite luting cements. Dent Mater 15:7-13.
12. Piwowarczyk A, Lauer HC, Sorensen JA (2005) Microleakage of various cementing agents for full cast crowns. Dent Mater 21:445-453.
13. Yang B, Ludwig K, Adelung R, Kern M (2006) Micro-tensile bond strength of three luting resins to human regional dentin. Dent Mater 22:45-56.
14. Behr M, Rosentritt M, Regnet T, Lang R, Handel G (2004) Marginal adaptation in dentin of a self-adhesive universal resin cement compared with well-tried systems. Dent Mater 20:191-197.
15. Rosentritt M, Behr M, Lang R, Handel G (2004) Influence of cement type on the marginal adaption of all-ceramic MOD inlays. Dent Mater 20:463-469.
16. Blatz MB, Sadan A, Martin J, Lang B (2004) In vitro evaluation of shear bond strength of resin to densely-sintered high-purity zirconium oxide after long-term storage and thermal cycling. J Prosthet Dent 91:356-362.
17. Kern M, Wegner SM (1998) Bonding to zirconia ceramic: adhesive methods and their durability. Dent Mater 14:64-71.
18. De Munck J, Vargas M, Van Landuyt K, Hikita K, Lambrechts P, Van Meerbeek B (2004) Bonding of an auto-adhesive luting material to enamel and dentin. Dent Mater 20:963-971.
19. Bayle MA, Gregoire G, Sharrock P (2007) The role of acrylophosphonic acid monomers in the formation of hybrid layers based on self-etch adhesive. J Dent 35:302-308.
20. Richardson D, Tao L, Pashley DH (1991) Dentin permeability: effect of crown preparation. Int J Prosthodont 4:219-225.

21. Pashley DH, Pashley EL (1991) Dentin permeability and restorative dentistry: a status report for the American Journal of Dentistry. Am J Dent 4:5-9.

22. Watanabe T, Sano M, Itoh K, Wakumoto S (1991) The effect of primers on the sensitivity of dentin. Dent Mater 7:148-150.

23. Schüpbach P, Lutz F, Finger WJ (1997) Closing of dentinal tubules by Gluma desensitizer. Eur J Oral Sci 105:414-421.

24. Felton DA, Bergenholtz G, Kanoy B (1991) Evaluation of the desensitizing effect of Gluma Dentin Bond on teeth prepared for complete-coverage restorations. Int J Prosthodont 4:292-298.

25. Dondi dall'Orologio G, Malferrari S (1993) Desensitizing effects of Gluma Primer and Gluma 2000 on hypersensitive dentin. Am J Dent 6:283-286.

26. Meiers JC, Griffo TE, Miller GA (1992) Antimicrobial activity of dentin bonding systems and glass ionomers. J Dent Res Special Issue 71:310 (Abstract.-Nr. 1639).

27. Schmidlin PR, Zehnder M, Gohring TN, Waltimo TM (2004) Glutaraldehyde in bonding systems disinfects dentin in vitro. J Adhes Dent 6:61-64.

28. Nakabayashi N, Watanabe A, Gendusa NJ (1992) Dentin adhesion of modified 4-META/MMA-TBB resin: function of HEMA. Dent Mater 8:259-264.

29. Edler TL, Krikorinon E, Thompson VP (1991) FITR surface analysis of dentin and dentin bonding agents. J Dent Res 70:1534.

30. Erickson RL (1989) Mechanism and clinical implications of bond formation for two dentin bonding agents. Am J Dent 2:117-123.

31. Sailer I, Tettamanti S, Stawarczyk B, Fischer J, Hämmerle CHF (2010) In-vitro study of the influence of dentin desensitizing and sealing on the shear bond strength of two universal resin cements. J Adhes in press.

32. Lüthy H, Loeffel O, Hämmerle CHF (2006) Effect of thermocycling on bond strength of luting cements to zirconia ceramic. Dent Mater 22:195-200.
33. Huh JB, Kim JH, Chung MK, Lee HY, Choi YG, Shim JS (2008) The effect of several dentin desensitizers on shear bond strength of adhesive resin luting cement using self-etching primer. J Dent 36:1025-1032.
34. Yim NH, Rueggeberg FA, Caughman WF, Gardner FM, Pashley DH (2000) Effect of dentin desensitizers and cementing agents on retention of full crowns using standardized crown preparation. J Prostet Dent 83:459-465.
35. ISO/TS 11405: 2003. Dental materials – Testing of adhesion to tooth structure
36. Krejci I, Reich T, Lutz F, Albertoni M (1990) In-Vitro-Testverfahren zur Evaluation Dentaler Restaurationssysteme. 1. Computergesteuerter Kausimulator. Schweiz Monatsschr Zahnmed 100:953-960.
37. Aranha AC, Siqueira Junior CADS, Cavalcante LMA, Pimenta LAF, Marchi GM (2006) Microtensile bond strengths of composite to dentin treated with desensitizer products. J Adhes Dent 8:85-90.
38. Cobb DS, Reinhardt JW, Vargas MA (1997) Effect of HEMA-containing dentin desensitizers on shear bond strength of a resin cement. Am J Dent 10:62-65.
39. Soeno K, Taira Y, Matsumura H, Atsuta M (2001) Effect of desensitizers on bond strength of adhesive luting agents to dentin. J Oral Rehabil 28:1122-1128.
40. Ernst CP, Wenzl N, Stender E, Willershausen B (1998) Retentive strengths of cast gold crowns using glass ionomer, compomer, or resin cement. J Prosthet Dent 79:472-476.
41. Palacios RP, Johnson GH, Phillips KM, Raigrodski AJ (2006) Retention of zirconium oxide ceramic crowns with three types of cement. J Prosthet Dent 96:104-114.

42. Ernst CP, Cohnen U, Stender E, Willershausen B (2005) In vitro retentive strength of zirconia oxide ceramic crowns using different luting agents. J Prosthet Dent 93:551-558.

43. Heintze SD (2010) Review: Crown pull-off test (crown retention test) to evaluate the bonding effectiveness of luting agents. Dent Mater 26:193-206.

44. Göhring TN, Schönenberger KA, Lutz F (2003) Potential of restorative systems with simplified adhesives: quantitative analysis of wear and marginal adaptation in vitro. Am J Dent 16:275-282.

45. Manhart J, Schmidt M, Chen HY, Kunzelmann KH, Hickel R (2001) Marginal quality of tooth-colored restorations in class II cavities after artificial aging. Oper Dent 26:357-366.

46. Krejci I, Lutz F (1990) In-Vitro-Testverfahren zur Evaluation Dentaler Restaurationssysteme. 3. Korrelation mit In-vivo-Resultaten. Schweiz Monatsschr Zahnmed 100:1445-1449.

47. Lutz F, Krejci I (1994) Mesio-occlusodistal amalgam restorations: quantitative in vivo data up to 4 years. A data base for the development of amalgam substitutes. Quintessence Int 25:185-190.

48. Oilo G (1993) Bond strength testing – what does it mean. Int Dent J 43:492-498.

7. Tables and Figures

Table 1 Summary of products used.

Cement systems	Short name	Company	Lot-Nr.
Panavia 21 Clearfil Porcelain Bond Activator Clearfil SE Bond Primer	PAN	Kuraray Dental Co Ltd., Osaka, Japan	00406C UNI TC / 00647C CAT 00208B 00769A
RelyX Unicem RelyX Ceramic Primer	RXU	3M ESPE, Seefeld, Germany	352388 5WM
G-CEM Capsule GC Ceramic Primer A GC Ceramic Primer B	GCM	GC, Leuven, Belgium	803061 0901272 0901232
Gluma Desensitizer	G	Haereus Kulzer, Hanau, Germany	20088

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

Composition of the bonding agents and cements		
Bonding agent and cement	**composition**	**Application steps as recommended by the manufacturer**
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 dired 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. Dispence equal amounts of Panavia21 Catalyst and Universal pastes 2. Slowly turn the dispencer 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 air-brush gently
RelyX Unicem Aplicap	Powder: glass fillers, silica, calciumhydroxide, self-cure initiators, pigments, lightcure initiators Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-cure initiators	1. Insert capsule into Activator, press handle and hold for 2 – 4 s 2. Mix 10 s with RotoMix Capsule Mixing Unit 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

GC Ceramic Primer B	Methyl methacrylate, Ethanol, 2-HEMA	drop of GC Ceramic Primer B for 5 s 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		

Table 3 Mean, standard deviation (SD) and 95% confidence interval of mean tensile bond strength (MPa) and p-value of the two sample Student`s t-test between initial and aging groups.

	initial			aging	
group	**Mean (SD) MPa**	**95% CI MPa**	**p- value**	**Mean (SD) MPa**	**95% CI MPa**
PAN, control group	14.1 (3.5)[A]	(11.9,16.4)	**< 0.001**	7.3 (1.7)[a]	(6.1,8.4)
RXU	12.8 (2.9)[A]	(10.9,14.6)	**0.006**	9.1 (3.0)[a,b]	(7.2,11.0)
RXU-G	13.1 (2.9)[A]	(11.2,14.9)	0.874	12.8 (4.3)[b]	(10.1,15.6)
GCM	10.7 (2.9)[A]	(8.8,12.5)	0.06	8.6 (2.2)[a,b]	(7.2,10.0)
GCM-G	13.7 (4.2)[A]	(11.0,16.3)	0.92	13.4 (6.2)[b]	(9.5,17.4)

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

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

	Decementing of the crown				**Failure in the tooth End of measuring**	
Failure mode	**1 (freq.)**	**2 (freq.)**	**3 (freq.)**	1-3 (rel. freq. %) (95% CI)	**4 (frequency)**	4 (rel. freq. %) (95% CI)
Initial						
PAN, control group	1	9	0	83.3 (51.5,97.9)	2	16.7 (2.0,48.4)
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)
Aging						
PAN, control group	2	10	0	100 (73.5,100)	0	0 (0,26.5)
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)

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

Group	initial median (95%CI) (MPa)	aging median (95% CI) (MPa)
PAN, control group	14.5 (13.2,15.8)	6.7 (5.0,8.4)
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)

Figure 1 Study design. Involved cements, their pretreatment and aging.

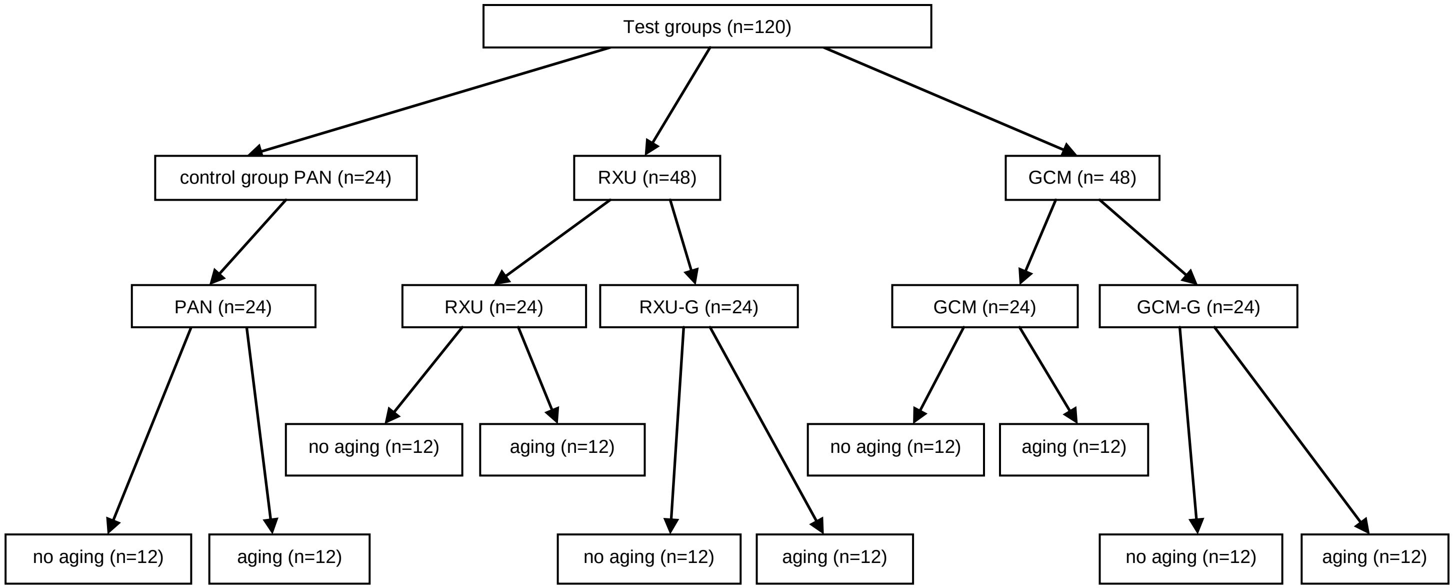


Figure 2 Calculation of the dentin surface using the Cerec 3 Volume Program.

Figure 3 Pull-out test design.

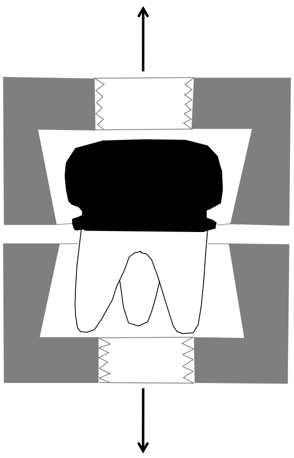


Figure 4 Classification of failure types 1 to 4.

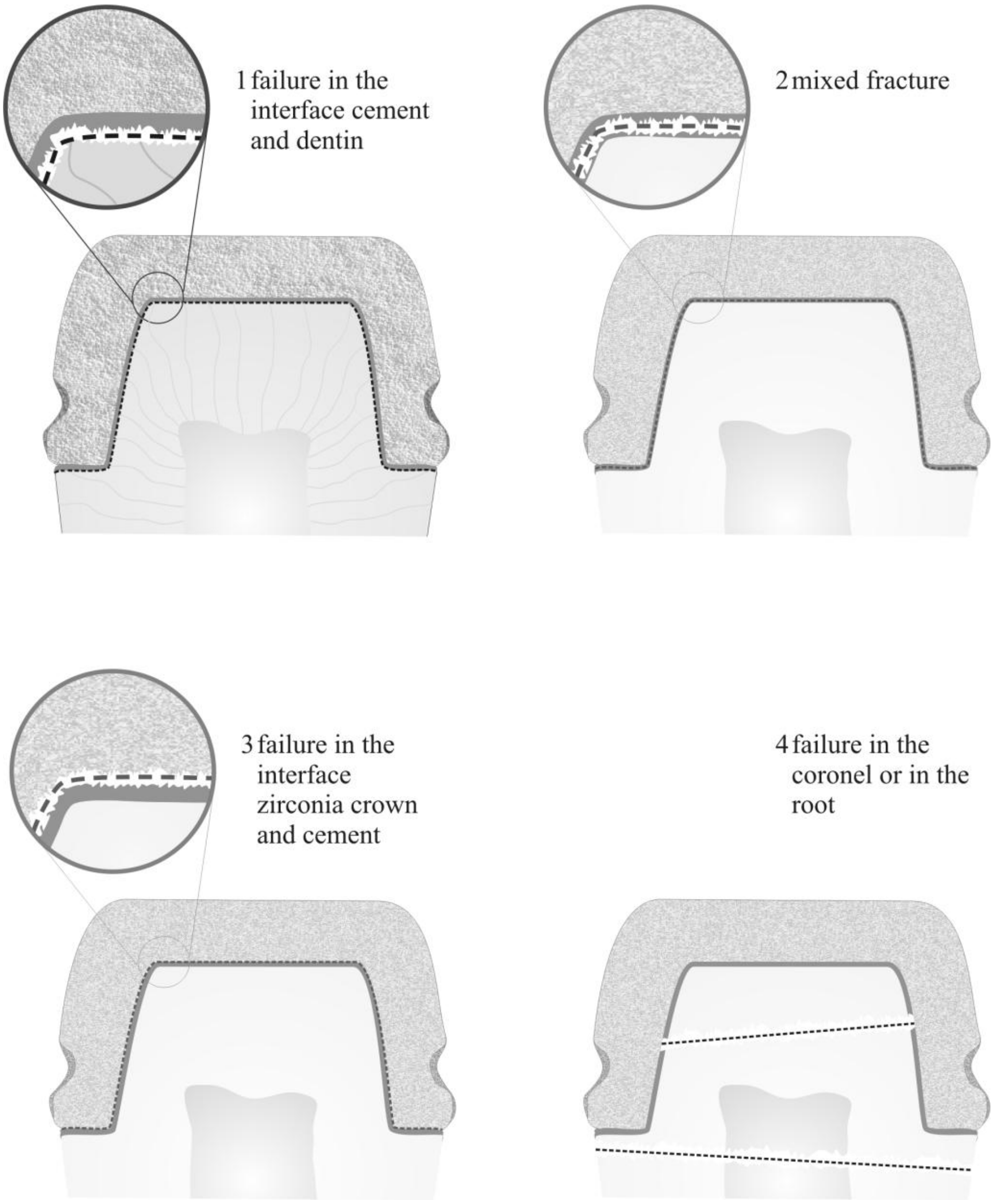
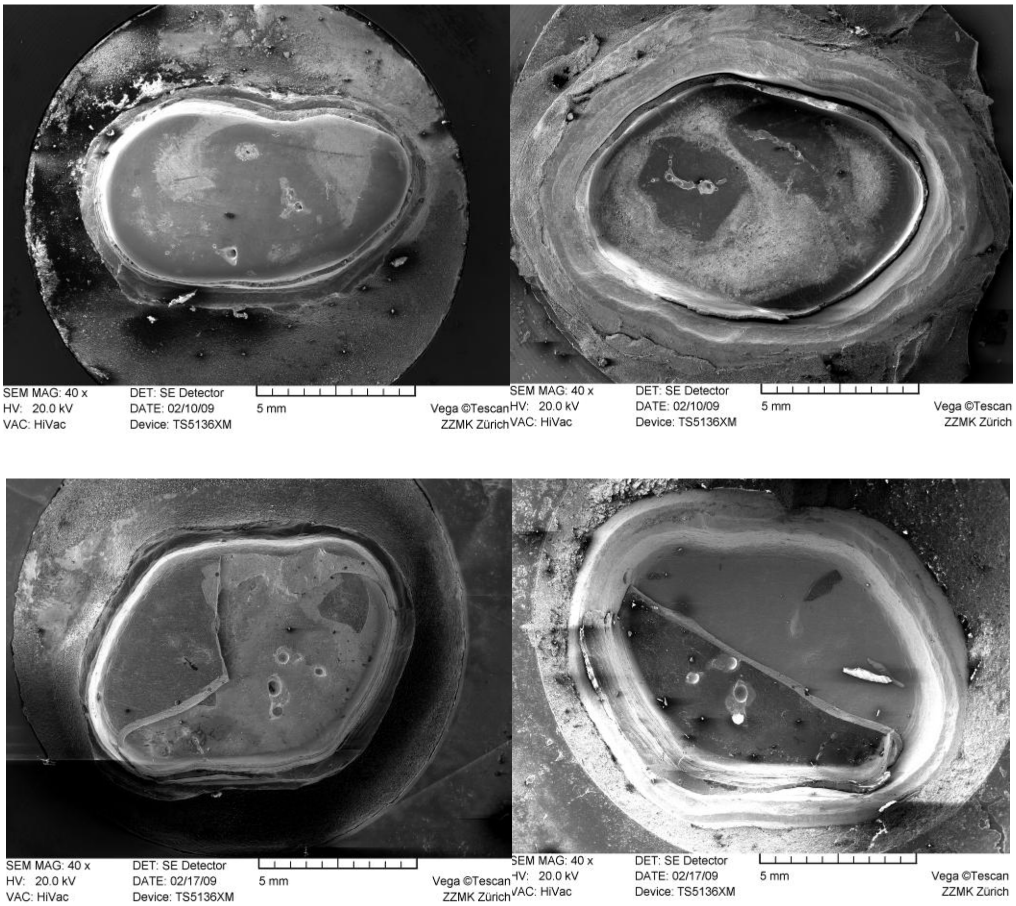


Figure 5 SEM-picture: failure type 1-failure in the interface dentin and cement, type 2 - mixed failure, and type 4 - failure in the coronel or root.



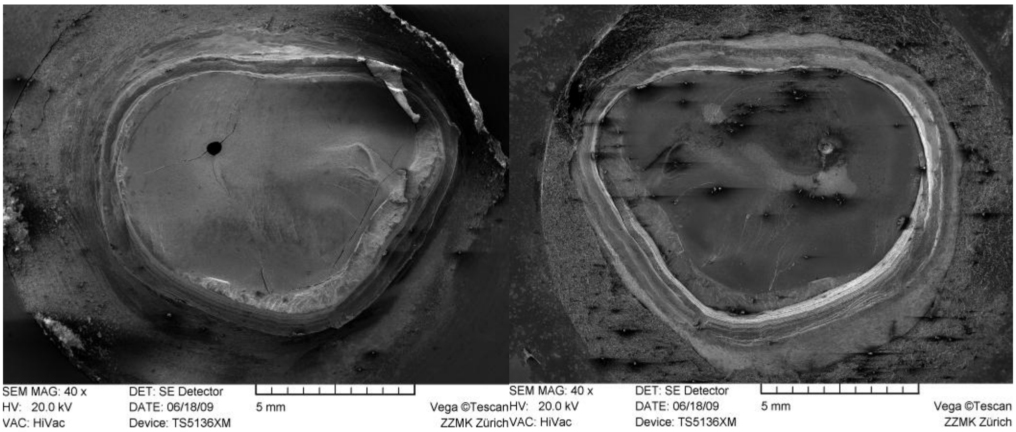


Figure 6 Diagram of tensile strength (MPa) of initial and chewing simulated groups.

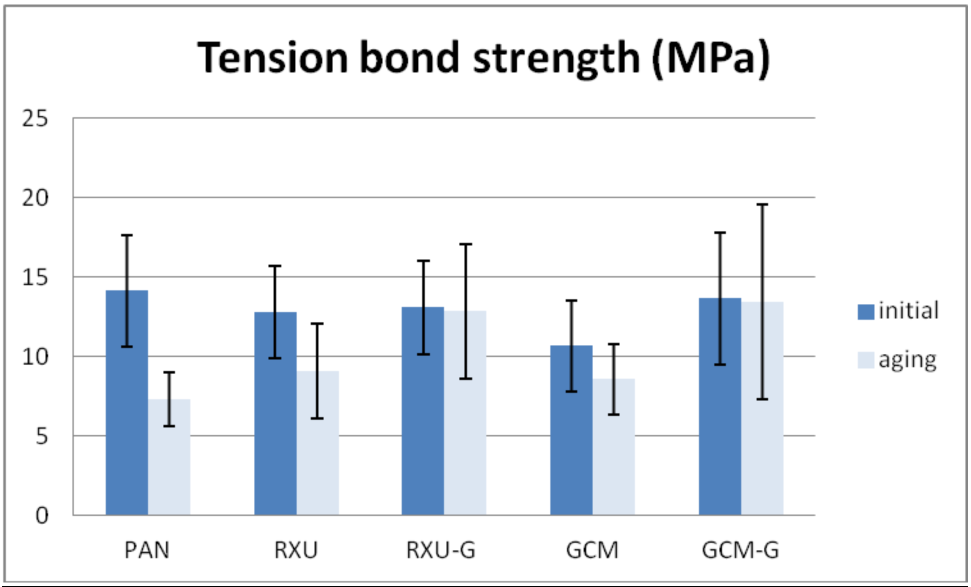


Figure 7 The cultimative distribution function for failure with respect to initial tensile strength (MPa) by Kaplan-Meier.

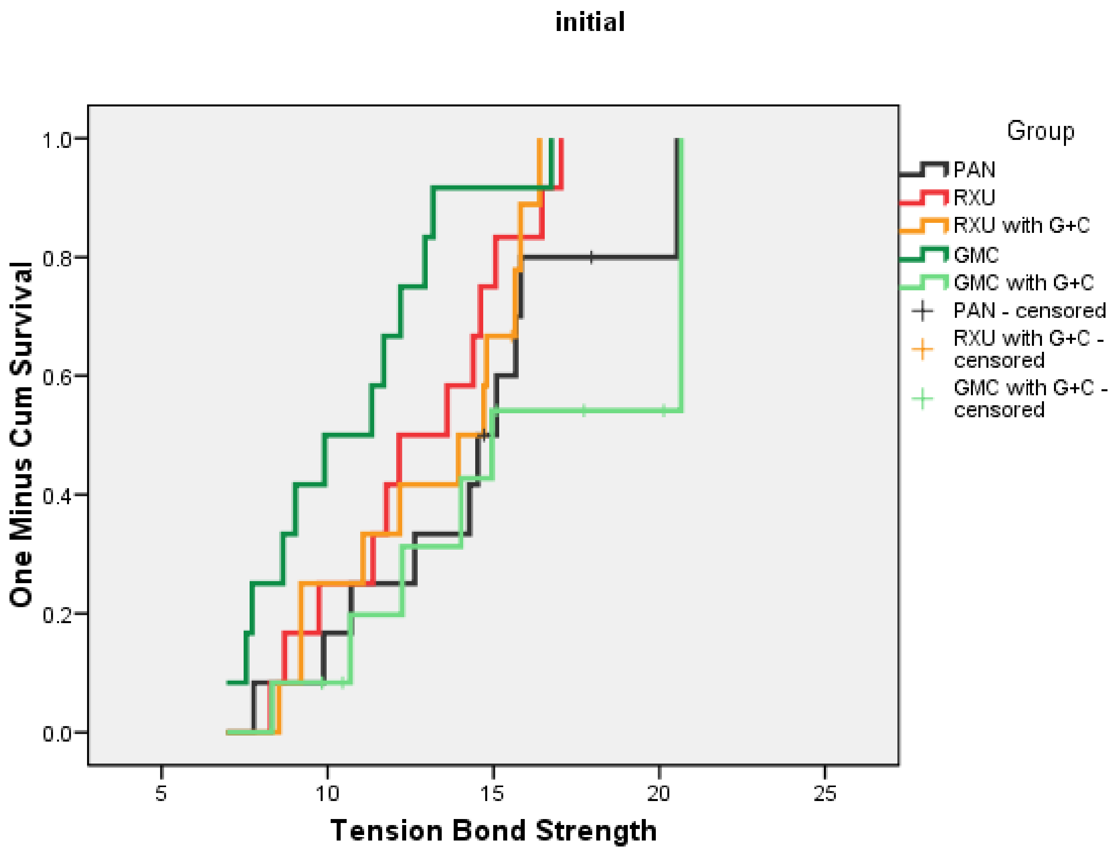


Figure 8 The cultimative distribution function for failure with respect to tensile strength (MPa) after chewing simulation by Kaplan-Meier.