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ORIGINAL ARTICLE

The impact of cement mixing and storage errors on the risk of failure of glass–ceramic crowns

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Abstract This study evaluated the impact of different mixing ratios and wrongly stored blends of dual-curing composite cements on Empress2 glass–ceramic crowns by means of a flexural strength test and a fracture resistance test. Thermally damaged blends and fresh blends were mixed using different mixing ratios of dual-curing Panavia F and Variolink II composite cement (2:1; 1:1; 1:1.5; 1:1.75; base/catalyst). Sixteen groups of rectangular beams of both cements (two blends, four ratios, chemical-curing, light-curing) were constructed. Their flexural strength was determined in a three-point bending test. Furthermore, 64 Empress2 all-ceramic crowns were luted onto human molars, again using fresh and thermally damaged blends as well as different mixing ratios of the luting agents. After aging, fracture resistance was investigated. The flexural strength of dual-curing composite cements was influenced to a statistically significant extent by mixing ratios and storage conditions. In particular, the chemical curing mode of these cements was affected by the thermal damage of the blends. However, this study could not demonstrate a significant impact on the fracture resistance of Empress2 glass-ceramic crowns when different mixing ratios or wrongly stored cements were used. Dual-curing composite luting agents seem to tolerate a wide range of mixing errors, but their chemical curing mode may be affected by storage errors.

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Introduction

Luting agents transfer masticatory stress from the fixed prosthetic restoration to the tooth substance [8, 9, 14, 16]. Dentists rely on the reliable setting of luting agents as long as suitable mixing ratios are being used and environmental conditions in the oral cavity such as moisture are under control. However, luting agents may fail due to wrong storage conditions, even if shelf life has not been exceeded.

According to the authors' experience, investigations into flexural strength values of dual-curing composite cements have been influenced by wrong storage conditions of cements. Although all light-cured samples showed the expected strength values, no value could be found for exclusively chemical-cured specimens. It could be demonstrated that the poor chemical-curing reaction was not caused by a LOT failure but by thermal damage due to wrongly stored cements. The chemical-curing capacity of dual-curing composite cements is driven by benzoyl-peroxide starter molecules [4, 5]. Benzoyl-peroxides are known to be unstable at high temperatures, whereas ketones or camphor–chinons, which initiate light-curing reactions, prove to be more stable.

Laboratory tests have demonstrated that cement properties such as flexural strength are influenced by mixing ratios and undamaged setting reactions. The question arises as to whether the use of those damaged cements has a direct measurable effect on the survival rate of fixed partial dentures. The stress transfer capacity of those cements was assumed to be significantly reduced. Therefore, we hypothesized that, after simulation of clinical aging, the fracture resistance of Empress2 glass–ceramic molar crowns might be significantly lower, if cements with wrong mixing ratios or damaging setting procedures are used.

Materials and methods

Flexural strength of cement beams

The dual composite cements Panavia F and Variolink II were investigated using both their chemical- and light-curing capacities. A blend with thermal damage due to wrong storage conditions of the cement (unknown circumstances) and a fresh blend were used (Fig. 1).

Rectangular beams measuring 25 mm in length, 2 mm in height, and 2 mm in width were constructed in silicone moulds. The light-cured groups were polymerized for 40 s using Elipar Trilight from 3M ESPE (Seefeld, Germany). The chemical-cured groups were polymerized under room temperature and under the exclusion of any visible light. Figure 1 shows the mixing ratios used (catalyst/base): (a) regular 1:1; (b) 1:1.5; (c) 1:1.75; (d) 2:1. Eight specimens each were constructed per group.

The cement surplus was removed, the mould covered with a polyethylene foil, and then closed with a glass slab. Care was taken that the beams did not show any blisters or surface defects and fit to a size of 25×2×2 mm.

After 24 h in distilled water at 37°C, the beams were loaded to failure using a three-point bending test (Zwick, Ulm, Germany). Rounded supports were placed at a distance of 20 mm. The cross-head speed was v=1 mm/min. The flexural strength σs [MPa] of the beams was calculated using the formula [6]:

$$\sigma s = \frac{3 \times F \times 1}{2 \times b \times h2} \qquad \begin{array}{l} F = \text{force}, 1 = \text{length} \\ h = \text{height}, b = \text{width} \end{array}$$

Fracture resistance test of Empress2 glass–ceramic crowns

Sixty-four human molars (stored in 0.5% chloramine solvent) were used. Their roots were coated with a thin (750 µm±100) layer of polyether (Impregum, 3M-Espe, Seefeld, Germany) and axially inserted in polymethylmethacrylate (acrylic) resin (Palapress Vario; Heraeus-Kulzer, Wehrheim, Germany). The polyether layer should simulate the tooth movement capacity provided by the periodontium.

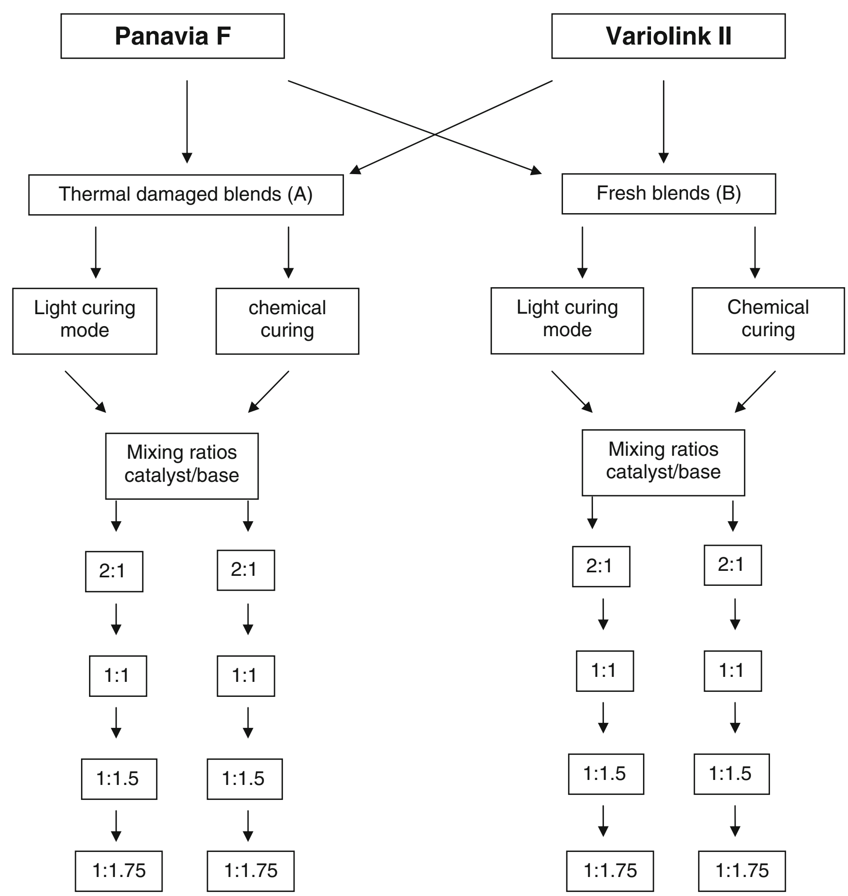

Fig. 1 Materials and mixing ratios used in the flexural strength test

The inserted teeth were randomly assigned in subgroups of eight teeth each. A 0.8-mm wide chamfer finish line was prepared in dentin using diamond burs. Impressions were taken from the prepared teeth (Permadyne-Penta, 3M-Espe, Seefeld, Germany), and gypsum cast were made. On the casts, Empress2 all-ceramic molar crowns were manufactured by a dental technician. The occlusal three-point contact areas were constructed to correspondent human molar antagonists that were later used to apply the occlusal load onto the crowns during the simulation of oral stress.

Before setting, the inner surfaces of the Empress2 all-ceramic crowns were etched for 20 s with <5% hydrofluoric-acid (IPS Ceramic etching gel, Ivoclar-Vivadent, Schaan, FL, USA), and a silane coupling agent was applied (Monobond S, Ivoclar-Vivadent, Schaan, FL, USA). The Empress2 all-ceramic crowns were adhesively cemented by using corresponding dentin adhesive systems and different mixed luting agents. Both are depicted in Table 1.

Twenty-four hours after cementation, the Empress2 all-ceramic crowns were thermally and mechanically loaded: 6,000×5/55°C, every 2 min changing; 1.2×106× 50 N, 1.66 Hz [2, 17, 19]. These parameters should represent a 5-year period of oral stress [17]. The entire simulation process took place 8.3 days. The load of 50 N was applied by the same third human antagonists, which were used to form the occlusal contact area of the crowns. Thus, it was assured that the load was axially inserted with a three-point support into the Empress2 all-ceramic crown, which should be stressed.

After aging, all undamaged crowns were mechanically loaded to fracture using the universal testing machine Zwick 1446 (Zwick, Ulm, Germany). The force was axially applied to the center of the crowns (three-point contact area) with a 12.5-mm diameter steel ball using a cross-head speed of 1 mm/min. A tin foil with a thickness of 0.4 mm was inserted between the steel ball and the crown to avoid local force peaks (Fig. 2).

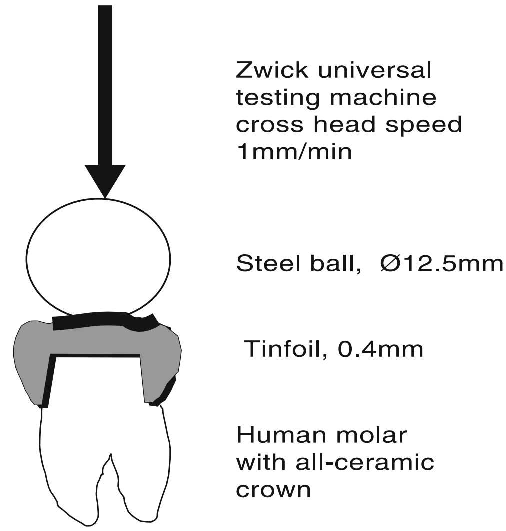

Fig. 2 Set-up of the fracture test of the Empress2 all-ceramic crowns

Statistics

Statistical analysis was performed using the program SPSS 12 (SPSS, Chicago, IL, USA). Means and standard errors of means were calculated. Statistical differences were determined using ANOVA and Tukey post hoc test. The level of significance was set at α=0.05.

Results

Flexural strength of cement beams

Chemical curing mode

Panavia F specimens made of thermally damaged blends achieved 35.9 MPa in the regular mixing ratio, which was

Table 1 Materials and mixing ratios used to lute Empress2 all-ceramic crowns

Material; curing mode	Mixing ratios
Panavia F (Kuraray, Osaka, J)	1:1 paste A/B
Total etch, A/B Primer	1:1.5
	1:1 cement annealed for 24 h in 60°C water bath before mixing
Chemical-curing mode (control):	1:1 paste A/B
Panavia 21 (Kuraray, Osaka, J)	1: 1.5
Total etch, A/B Primer	
Variolink II low viscosity	1:1 base/catalyst
(Ivoclar-Vivadent, Schaan, FL)	1:1.5
Total etch, Syntac classic	1:1 cement annealed for 24 h in 60°C water bath before mixing

Table 2 Flexural strength in MPa of chemical-cured Panavia F cement beams

Material; mixing ratios,	Mean (MPa)	SE
Group A blends with thermal damage		
Panavia F 2:1 chemical curing	27.1	2.3
Panavia F 1:1 chemical curing	35.9	1.3
Panavia F 1:1.5 chemical curing	36.9	2.7
Panavia F 1:1.75 chemical curing	30.9	2.0
Group B fresh blends		
Panavia F 2:1 chemical curing	70.1	2.3
Panavia F 1:1 chemical curing	82.2	10.6
Panavia F 1:1.5 chemical curing	56.1	3.6
Panavia F 1:1.75 chemical curing	48.5	6.7

Means, SE of means

Table 3 *P* values of post hoc test of flexural strength means of Panavia F, chemical cured, fresh, and thermally damaged blends

Parameter	Panavia 1:1	Panavia 1:1.5	Panavia 1:1.75	Panavia 2:1fresh	Panavia 1:1fresh	Panavia 1:1.5 fresh	Panavia 1:1.75 fresh
Panavia 2:1	0.967	0.943	1.0	0.000	0.000	0.020	0.218
Panavia 1:1		1.0	0.999	0.002	0.000	0.252	0.823
Panavia 1:1.5			0.996	0.002	0.000	0.309	0.875
Panavia 1:1.75				0.000	0.000	0.067	0.454
Panavia 2:1fresh					0.914	0.874	0.486
Panavia 1:1fresh						0.200	0.059
Panavia 1:1.5 fresh							0.999

Tukey-HSD, $\alpha \leq 0.05$, $n=8$

less than 50% of the flexural strength measured for specimens using fresh blends (82.2 MPa; Table 2). The difference in flexural strength values was statistically significant for the 2:1 and the 1:1 ratio (Table 3).

Measurement of chemical-cured Variolink specimens made of thermally damaged blends failed 24 h after curing at 37°C because of incomplete polymerization. The Variolink beams could not be removed from the silicon moulds (Table 4). Specimens constructed from fresh blends showed flexural strength values that were comparable with those of Panavia fresh blends (1:1:85.3 MPa).

Light-curing mode

Light-cured specimens made of thermally damaged blends did not seem to be influenced by storage conditions. Variolink light-cured specimens showed significantly higher flexural strength values than those of Panavia F (Table 5). However, the flexural strength of Panvia F specimens did not seem to be influenced to a statistically significant extent by different mixing ratios, whereas Variolink II specimens showed significantly different strength values depending on the mixing ratio.

Fracture resistance of aged Empress2 all-ceramic crowns

Figure 3 indicates that the fracture resistance values of Empress2 all-ceramic crowns are widely distributed. Therefore, ANOVA did not show any statistically significant differences between the groups ($p=0.383$).

Empress2 all-ceramic crowns luted with regular Panavia mixing ratios had a mean fracture resistance of about 1,750 N. No statistical difference could be found between Panavia F and chemical-cured Panavia 21 (control). The means of fracture resistance were reduced if the mixing ratios of both cements were changed to 1:1.5 of pastes A/B. However, the difference was not statically significant. Cementation with annealed luting agents did not influence fracture resistance values.

Empress2 all-ceramic crowns cemented with Variolink showed nearly identical fracture resistance means independent of wrong mixing ratios or annealed luting agents. Values ranged between 1,180 and 1,250 N. Value distribution was higher for the 1:1.5 mixing ratio, but the mean was different from the recommended 1:1 ratio. Cement annealed at 60°C 24 h in advance tended to show a lower but not a statistically significant fracture

Table 4 Flexural strength in MPa of chemical-cured Variolink II cement beams

Material, mixing ratio	Mean (MPa)	SE
Group A blends with thermal damage		
Variolink 2:1 chemical curing	–[a]	–
Variolink 1:1 chemical curing	–[a]	–
Variolink 1:1.5 chemical curing	–[a]	–
Variolink 1:1.75 chemical curing	–[a]	–
Group B fresh blends		
Variolink 2:1 chemical curing	54.23	2.1
Variolink 1:1 chemical curing	85.3	11.6
Variolink 1:1.5 chemical curing	80.3	3.2
Variolink 1:1.75 chemical curing	73.2	2.9

Means, SE of means

[a] Incomplete setting reaction, specimens could not be constructed

Table 5 Flexural strength in MPa of light-cured Panvia F and Variolink II beams made of thermally damaged blends

Material; mixing ratio, curing mode	Mean (MPa)	SE
Group A blends with thermal damage		
Variolink 2:1 light-curing	108.2	2.2
Variolink 1:1 light-curing	123.1	5.0
Variolink 1:1.5 light-curing	127.3	3.2
Variolink 1:1.75 light-curing	121.1	4.4
Group A blends with thermal damage		
Panavia F 2:1 light-curing	78.4	4.5
Panavia F 1:1 light-curing	66,6	3.0
Panavia F 1:1.5 light-curing	66.8	1.3
Panavia F 1:1.75 light-curing	66.1	4.3

Means, SE of means

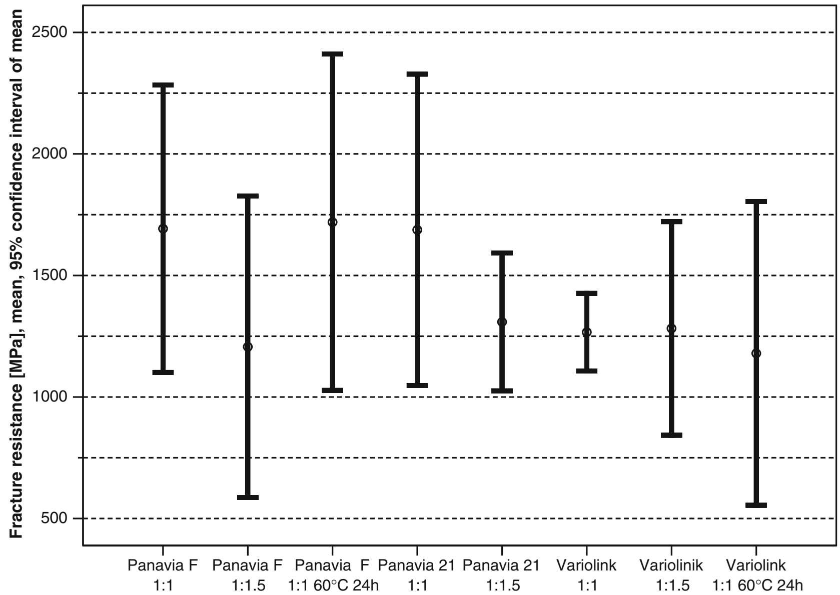

Fig. 3 Fracture resistance of Empress2 all-ceramic crowns after thermal cycling and mechanical loading (p=0.384)

resistance. All but two crowns tested exceeded a final fracture load of 500 N. The two exceptions were Variolink-cemented Empress2 all-ceramic crowns (in the 1:1.5 and the 1:1 annealing group) reaching 343 and 370 N, respectively (Fig. 3).

Discussion

Although the luting agent is an important connector between tooth substance and restoration, many clinicians disregard the cementation procedure. Clinicians are frequently unaware of the influence of wrong storage conditions or cement mixing errors on long-term results. Most publications focus on adhesive properties or the indication of the type of cement used [1, 12, 14, 15]. Literature reports on dual-curing composite cements emphasize that the adequate polymerization of a resin luting agent is a crucial factor for ensuring optimal bond strength at the ceramic–resin luting agent–dentin interface as well as for optimal physical properties [13, 20]. The intensity of radiation and the translucency of restoration determine the adequate polymerization of resin cement [11]. However, the fact is often neglected that a high impact will also have variations of the paste-to-paste ratio or of the inactivity of the polymerization starter molecules caused by wrong storage.

Fleming reported a frequent occurrence of mixing errors [10]. Investigating the mixing methods of 40 dental nurses in their department, his team found a wide range of mixing ratios for cement. As a consequence, compressive strength values ranged between 40 and 110 MPa, and about 60% of the samples did not reach the official strength value of 80 MPa [10].

Although the consequences of mixing ratio variations of glass ionomer cements are described in the literature [3, 21], we detected only a few reports dealing with varying paste-to-paste ratios of resin cements [7]. De Gee and coworkers found paste-to-paste variations to have no significant influence on diametric tensile strength, but a more sensitive behavior on simulated occlusal wear tests [7]. This investigation showed no significant difference in the flexural strength values of varying mixing ratios. Therefore, both cements seem to have a wide “therapeutic range” and tolerate a considerable extent of mixing errors. Clinical simulation in an artificial mouth showed a tendency to lower fracture resistance in Panavia F and Panavia 21 but not in Variolink specimens. The decreasing means were not statistically significant. The reason for the different fracture resistance means between Panavia and Variolink may be caused by different wetting power or different compressive strength values.

A wide distribution of fracture resistance means has been considered to be typical for all-ceramics [18]. Thus, the decision is difficult whether fracture resistance was influenced by variations of mixing ratios or by the artificial thermal damage of cements, as the chosen test-design (artificial mouth and all-ceramic crowns) may just not have been sensitive enough to detect the luting agent influence. On the other hand, despite the frequent occurrence of mixing errors, clinical reports are obviously unobtrusive. The wide “therapeutic range” of resin cements disguises failures. Alternatively, other types of failure of all-ceramic crowns may be more important than failures caused by wrong luting procedures.

Conclusion

Dual-curing composite luting agents seem to tolerate a wide range of mixing errors. However, storage under too high temperature may reduce the chemical polymerization capacity of those cements.

Conflict of interest statement The authors declare that they have no conflict of interest.

References

1. Akungor G, Akkayan B, Gaucher H (2005) Influence of ceramic thickness and polymerization mode of a resin luting agent on early bond strength and durability with a lithium disilicate-based ceramic system. J Prosthet Dent 94:234–241
2. Attia A, Kern M (2004) Influence of cyclic loading and luting agents on the fracture load of two all-ceramic crown systems. J Prosthet Dent 92:551–556
3. Behr M, Rosentritt M, Loher H, Handel G (2006) Effect of variations from the recommended powder/liquid ratio on some properties of resin-modified cements. Acta Odontol Scand 64:214–220
4. Brauer GM (1981) Initiator-accelerator systems for acrylic resins and composites. Polymer science and technology, Volume 14, Biomedical and dental applications of Polymers. Plenum, pp 395–409
5. Darvell BW (2002) Materials science for dentistry. In: Darvell BW (ed) Resin restorative materials, 7th edn. BW Darvell, Hong Kong (ISBN 962-85391-5-9), pp 116–133
6. Darvell BW (2002) Materials science for dentistry. In: Darvell BW (ed) Mechanical testing, 7th edn. BW Darvell, Hong Kong (ISBN 962-85391-5-9), pp 20–21
7. deGee AJ, ten Harkel HC, Davidson CL (1989) The influence of mixing ratio on the working time, strength and wear of composites. Aust Dent J 34:466–469
8. Federlin M, Männer T, Hiller KA, Schmidt S, Schmalz G (2006) Two-year clinical performance of cast gold vs. ceramic partial crowns. Clin Oral Investig 10:126–133
9. Federlin M, Wagner J, Männer T, Hiller KA, Schmalz G (2007) Three-year clinical performance of cast gold vs. ceramic partial crowns. Clin Oral Investig 11:345–352
10. Fleming GJ, Marquis PM, Shortall AC (1999) The influence of clinically induced variability on the distribution of compressive fracture strength of a hand-mixed zinc phosphate dental cement. Dent Mater 15:87–97
11. Hofmann N, Pabsthart G, Hugo B, Klaiber B (2001) Comparison of photoactivation versus chemical or dual-curing of resin-based luting cements regarding flexural strength, modulus and surface hardness. J Oral Rehabil 28:1022–1028
12. Özcan M, Kerkdijk S, Valandro LF (2008) Comparison of resin cement adhesion to Y-TZP ceramic following manufacturers' instructions of the cements only. Clin Oral Invest 12:279–282
13. Pace LL, Hummel SK, Marker VA, Bolouri A (2007) Comparison of the flexural strength of five adhesive resin cements. J Prosthodont 16:18–24
14. Pospiech P (2002) All-ceramic crowns: bonding or cementing? Clin Oral Invest 6:189–197
15. Preuss A, Rosentritt M, Frankenberger R, Beuer F, Naumann M (2008) Influence of type of luting cement used with all-ceramic crowns on load capability of post-restored endodontically treated maxillary central incisors. Clin Oral Investig 12:151–156
16. Proos KA, Swain MV, Ironside J, Steven GP (2003) Influence of cement on a restored crown of a first premolar using finite element analysis. Int J Prosthodont 16:82–90
17. Rosentritt M, Behr M, Gebhard R, Handel G (2006) Influence of stress simulation parameters on the fracture strength of all-ceramic fixed-partial dentures. Dental Mater 22:176–182
18. Scherrer SS, de Rijk WG (1993) The fracture resistance of all-ceramic crowns on supporting structures with different elastic moduli. Int J Prosthodont 6:462–467
19. Stappert CF, Guess PC, Chitmongkolsuk S, Gerds T, Strub JR (2007) All-ceramic partial coverage restorations on natural molars. Masticatory fatigue loading and fracture resistance. Am J Dent 20:21–26
20. Walker MP, Spencer P, Eick JD (2003) Mechanical property characterisation of resin cement after aqueous aging with and without cyclic loading. Dent Mater 19:645–652
21. Wilder AD, Boghosioan AA, Bayne SC, Heymann HO, Sturdevant JR, Roberson TM (1998) effect of powder/liquid ratio on the clinical and laboratory performance of resin-modified glass-ionomers. Dent 26:369–377