Flexural strength and modulus of a novel ceramic restorative cement intended for posterior restorations as determined by a three-point bending test

Karin Sunnegårdh-Grönberg, Anne Peutzfeldt and Jan W. V. van Dijken

Department of Odontology, University of Umeå, Umeå, Sweden; Department of Dental Materials, School of Dentistry, University of Copenhagen, Denmark

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The aim of this study was to compare a new restorative cement intended for posterior restorations, Doxadent, with other types of tooth-colored materials as regards flexural strength and flexural modulus. The new restorative material consists mainly of calcium aluminate. Four hybrid resin composites, one polyacid-modified resin composite, one resin-modified glass ionomer cement, one conventional glass ionomer cement, one zinc phosphate cement, and an experimental version as well as the marketed version of Doxadent were investigated. Flexural strength and flexural modulus were tested according to ISO standard 4049 and determined after 1 d, 1 week, and 2 weeks. Together with the zinc phosphate cement, Doxadent had the lowest flexural strengths (13–22 MPa). The strongest materials were the resin composites and the polyacid-modified resin composite (83–136 MPa). The highest flexural modulus was found for Doxadent (17–19 GPa). The flexural strength of Doxadent decreased significantly from 1 week to 2 weeks, while flexural modulus remained unchanged. The other materials reacted in different ways to prolonged water storage. It can be concluded that the restorative cement Doxadent had significantly lower flexural strength and significantly higher flexural modulus than today's materials used for direct posterior restorations. □ *Composite; glass ionomer cement; mechanical properties; posterior restorations; zinc phosphate cement*

Karin Sunnegårdh-Grönberg, Department of Odontology, University of Umeå, SE-901 85 Umeå, Sweden. Fax.+46 9 0770 580, e-mail. Karin.Sunnegardh@odont.umu.se

Recently, a calcium aluminate cement, Doxadent, has been marketed in Scandinavia for Class I and II direct restorations. Reportedly, Doxadent will also be launched in the rest of Europe and in the USA. The material is being marketed as a 'bioceramic' alternative to amalgam and resin composite with claims of uniqueness and biocompatibility. Ceramics may be defined as materials that are inorganic and non-metallic (1) or as materials that have in common that they are compounds of metals and non-metals (2). Thus, according to the composition and setting reactions described below, Doxadent may be classified as a ceramic material (just as is the case with a typical zinc phosphate cement). But in contrast to ceramic materials which are heat-treated and therefore used indirectly, e.g. feldspathic porcelains, Doxadent sets directly in the cavity as a consequence of an acid-base reaction.

The restorative cement is available in the form of small tablets based on calcium aluminate ($3CaO{\cdot}Al_2O_3$). The tablets also contain ZrO_2 and SiO_2. When the calcium aluminate tablet is brought into contact with the supplied liquid, which contains water and a small amount of Li^+ions, an acid-base reaction is initiated during which water acts as an acid and calcium aluminate dissolves to form Ca^{2+}, $Al(OH)_4{}^-$ and OH^-. The solutes precipitate to form a gel. Gradually, the amorphous gel changes into a crystalline phase of mainly katoite $[(CaO)_3(Al_2O_3)(H_2O)_6]$, and hardening of the material is initiated when the crystals come into contact with each other. According to the manufacturer, hardening begins within 2 min and is fully completed after 2 weeks. The material is claimed to be sufficiently hard for chewing after 60 min.

Being situated on occlusal surfaces of posterior teeth, Class I and II restorations are exposed to greater masticatory forces than are restorations in anterior teeth and must therefore be made of stronger materials. Consequently, restoring posterior teeth not only places greater demands on the strength of the restorative material but also on the handling characteristics. When forces are acting on a Class I or Class II restoration, the stress pattern formed depends on the shape of the restoration. In the centre of a Class I restoration the stress is mainly compressive. However, if the cavity is beveled, tensile stress will form at the occlusal restoration margins. In contrast to a Class I restoration, tensile stress will be present in a Class II restoration at the isthmus area owing to the two-surfaced design of the restoration (3). This difference between Class I and Class II restorations is also reflected in in vivo longevity studies where Class II amalgam restorations show lower longevity than Class I restorations, the main reasons for failure being fracture and recurrent caries (4, 5). These problems are not only associated with amalgams; bulk fracture is also a major reason for clinical failure of resin composite restorations (6–8). A drawback of the early resin composite versions was their lack of wear resistance. In a previous study, in

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Table 1. Materials investigated

Material	Type	Batch no.	Manufacturer
Doxadent	Calcium aluminate hydrate	SC002 + SB002	Doxa Certex, Uppsala, Sweden
Experimental Doxadent	Calcium aluminate hydrate	–	Doxa Certex, Uppsala, Sweden
Definite	Resin composite	230 A3	Degussa, Hanau, Germany
Filtek Z250	Resin composite	20000315 A3	3M, St Paul, Minn., USA
Solitaire 2	Resin composite	221 A3	Heraeus Kulzer, Wehrheim, Germany
Tetric Ceram	Resin composite	B 30639 A4	Vivadent, Schaan, Liechtenstein
Dyract AP	Polyacid-modified resin composite	9903000100 A3	Dentsply/De Trey, Konstanz, Germany
ChemFil Superior	Conventional glass ionomer cement	9912001043 LYG 3	Dentsply/De Trey, Konstanz, Germany
Vitremer	Resin-modified glass ionomer cement	20001214 B2 20001004	3M, St Paul, Minn., USA
De Trey Zinc	Zinc phosphate cement	9912001048 9912000323	Dentsply/De Trey, Konstanz, Germany

vitro wear of Doxadent was determined to be inferior to that of resin composites and polyacid-modified resin composite (9).

Flexural strength is a fracture-related mechanical property since it is a measure of the resistance of restorations to withstand tensile forces. Materials with high flexural strength provide restorations with less susceptibility to fractures of the margins or the bulk. The value obtained in a laboratory determination of flexural strength of a material is related to the inherent strength combined with the effect of defects in the material. The presence of defects is a key parameter, since defects give rise to stress concentration, in particular in brittle materials. Thus, in laboratory determinations of flexural strength the specimens should be prepared in a manner similar to that used in the clinic in order that the flaws in the material may be comparable in the two situations. The stiffness or modulus of elasticity of a material is a measure of the resistance to deformation under load. An occlusal restoration with a high modulus of elasticity will undergo only a small deformation when subjected to forces from the opposing tooth during mastication. This implies that tensile forces generated at other parts of the restoration may be small, thus reducing the risk of fracture. Bulk, chip, and marginal fractures have been observed to be more frequent with microfilled resin composites than with hybrid and macrofilled composites (10–12). This may be an indication of the smaller modulus of elasticity of the microfilled materials. Another explanation that cannot be ruled out is that the fracture tendency is related to the poor bond that is formed between prepolymerized composite particles and the curable composite matrix. The poor quality of this bond is reflected in the relatively small flexural strength of these materials.

The purpose of the present study was to compare the new ceramic restorative cement with other direct restorative materials and with another ceramic cement, zinc phosphate cement, with respect to flexural strength and flexural modulus. The other direct restorative materials were resin composite, polyacid-modified resin composite, glass ionomer cement, and resin-modified glass ionomer cement. It was hypothesized that the restorative cement Doxadent would have a relatively low flexural strength and a relatively high modulus of elasticity. These hypotheses were based on 1) the relatively high in vitro wear previously found for Doxadent (9) and the significant, negative correlation between flexural strength and in vivo wear shown by Peutzfeldt & Asmussen (13), and 2) on the high modulus of elasticity previously reported for zinc phosphate cement compared with other types of cement and resinous materials (14).

Two versions of Doxadent were included in the study: an experimental version as well as the marketed version. According to the manufacturer, the two versions differ with respect to the process of tablet pressing, the process having been standardized and optimized to provide the marketed version with homogeneous and improved handling characteristics. Both versions of Doxadent are currently undergoing long-term clinical evaluation. Therefore both versions were included in this in vitro study to allow for analysis of possible correlations between the clinical and the laboratory performance.

Materials and methods

The investigated materials and their manufacturers are listed in Table 1. All materials were handled in accordance with the instructions of the manufacturers.

Eighteen rectangular specimens ($25 \times 2 \times 2$ mm) of each material were produced ($n = 6$) in split brass molds. The specimens of the calcium-aluminate cements were produced as follows: a tablet was partially immersed in the supplied liquid allowing it to absorb the liquid for 5 s. Subsequently, the tablet was totally immersed in the liquid for another 5 s, then blot dried on a piece of absorbing tissue, distributed in the mold, and condensed with an amalgam stopper under maximum hand pressure. The procedure was repeated with new tablets until the mold was filled with excess. During the condensation, the mold and the material in the mold were shielded by a wet paper towel to minimize desiccation of the specimen. Finally, excess material was carved away. The other materials were applied into the mold in accordance with well-established

Table 2. Flexural strength and flexural modulus ($n = 6$) of 1-day-old, 1-week-old, and 2-week-old specimens. Mean values ± standard deviations

Material	Flexural strength (MPa)			Flexural modulus (GPa)		
	1 day	1 week	2 weeks	1 day	1 week	2 weeks
Doxadent	18 ± 4	22 ± 4	17 ± 2	17.5 ± 3.7	16.5 ± 3.6	17.7 ± 4.1
Experimental Doxadent	20 ± 2	19 ± 8	12 ± 2	17.2 ± 2.2	19.0 ± 5.0	19.2 ± 5.5
De Trey Zinc	13 ± 3	15 ± 3	13 ± 2	15.0 ± 2.7	13.2 ± 1.5	12.9 ± 1.6
ChemFil Superior	32 ± 8	40 ± 10	46 ± 4	9.8 ± 2.3	12.1 ± 1.0	14.4 ± 0.7
Vitremer	78 ± 10	68 ± 13	68 ± 5	8.5 ± 0.5	6.7 ± 1.0	7.5 ± 1.0
Dyract AP	111 ± 16	114 ± 17	112 ± 18	6.9 ± 0.4	7.3 ± 0.5	8.3 ± 0.7
Tetric Ceram	110 ± 4	102 ± 4	99 ± 11	7.4 ± 0.3	6.5 ± 0.4	6.0 ± 0.5
Filtek Z250	136 ± 18	129 ± 15	125 ± 17	8.6 ± 0.7	8.2 ± 0.9	7.8 ± 0.6
Solitaire 2	104 ± 8	88 ± 10	83 ± 8	6.2 ± 0.3	5.2 ± 0.9	4.9 ± 0.4
Definite	107 ± 6	97 ± 8	96 ± 7	6.7 ± 0.7	7.0 ± 0.6	7.5 ± 1.2

procedures. After application and/or condensation of a material into the mold, excess material was removed by pressing a transparent matrix against the top surface of the specimen. All auto-curing materials were left to cure at room temperature and 100% humidity for 1 h. During the last 45 min, the matrices and mold were removed from the specimens. The light-curing materials were irradiated by an overlapping procedure for 100 s on the top and bottom sides of the specimen with an XL 3000 (3M, St Paul, Min., USA) light-curing unit at an intensity of 450 mW/cm^2. For each material, the intensity of the light-curing unit was verified with a radiometer (Optilux Radiometer, SDS Kerr, Danbury, Ct., USA) prior to sample preparation. After light-curing, the specimens were kept at room temperature and 100% humidity for 1 h, the last 45 min without the matrices and mold. After 1 h, all the specimens were transferred to a bath containing demineralized water at 37°C for 1 d, 1 week or 2 weeks.

The specimens were then ground under running water on #1000 silicon carbide paper, and the dimensions of each specimen were recorded with a micrometer. Flexural strength and flexural modulus were determined according to ISO standard 4049 for polymer-based materials with a 3-point bending test at a cross-head speed of 0.75 mm/min and with 20 mm between the two supports. The test was performed by means of an Instron Universal Testing Machine (model 5566, Instron Ltd., High Wycombe, UK) with the specimens submerged in a water bath maintained at 37°C. A mean value and standard deviation were calculated for each material and storage time.

Statistical analysis

The results were analyzed by Kruskal-Wallis H tests and Mann-Whitney U tests. For each material the influence of storage time in water was analyzed by one-way ANOVA and Newman-Keuls' multiple-range tests or in the case of lack of homogeneity of the standard deviations, by Kruskal-Wallis H tests (15). The level of significance used was $\alpha = 0.05$.

Results

The results are given in Table 2 as mean values and standard deviations of flexural strength and flexural modulus for each of the 3 storage times. Irrespective of storage time, the following general ranking was found between the flexural strengths of the different types of materials with the strongest material mentioned first: resin composite = polyacid-modified resin composite > resin-modified glass ionomer cement > conventional glass ionomer cement > Doxadent ≥ experimental Doxadent ≥ zinc phosphate cement. With respect to flexural modulus, almost the opposite ranking was obtained: experimental Doxadent = Doxadent ≥ zinc phosphate cement ≥ conventional glass ionomer cement ≥ resin-modified glass ionomer cement = polyacid-modified resin composite = resin composite.

The tested materials responded differently to prolonged storage in water. Flexural strength remained unchanged from 1 d to 2 weeks for 5 materials (De Trey Zinc, Vitremer, Dyract AP, Tetric Ceram, and Filtek Z250). For the conventional glass ionomer cement (Chemfil Superior), flexural strength increased significantly over the storage time. For Solitaire 2 and Definite, flexural strength decreased over time in water, while for the two versions of Doxadent, flexural strength decreased significantly from 1 week to 2 weeks. With respect to flexural modulus, this remained unchanged throughout the experimental period for 5 materials (the two versions of Doxadent, De Trey Zinc, Filtek Z250, and Definite). For Chemfil Superior and Dyract AP, flexural modulus increased significantly over storage time. Tetric Ceram and Solitaire 2 showed a significant decrease in flexural modulus over time in water. Regarding Vitremer, flexural modulus decreased significantly between 1 d and 1 week, but increased during the second week.

Discussion

Different methods are available for testing flexural strength and flexural modulus. In this study, flexural strength was measured according to the ISO standard for polymer-based filling restoratives. Obviously, some of the materials tested were water-based cements and not polymers, and the pertinence of determining flexural strength for a water-based cement may be debated. The mechanical property included in the ISO standard (#9917) for dental water-based cements, which comprises glass ionomer cements and zinc phosphate cements, is that of compressive strength. However, this choice of property may be regarded as the result of tradition rather than a profound evaluation of which property is the clinically most relevant. Indeed, such an evaluation may not lead to a simple answer as the water-based cements are used for three different purposes: luting, lining, and restoration. Furthermore, the standard was formulated at a time when glass ionomer cements were not considered for use in Class I and Class II cavities. The Introduction pointed out the clinical relevance of flexural strength for stress-bearing restorations. On this background it seems appropriate to determine flexural strength also for materials that are not resinous but are marketed for Class I and II restorations. Since flexural strength can be measured in different ways, the results depend on the method used, and details about the method used should always be given. Therefore, the flexural strength of a material is useful only if references are present and the test method known. Otherwise, only relative comparisons are indicated.

Li & White showed flexural strength values of 5–7 MPa for zinc phosphate cement, making it the weakest material among a group of other restoratives including resin-modified and conventional glass ionomer cements and resin composites, which is in keeping with the results of the present study (14). Also in agreement with the present study, Li & White found the zinc phosphate cement to have a higher modulus of elasticity in comparison with the other materials tested and that the modulus of elasticity was stable over time. Piwowarczy et al. measured the flexural strength of a zinc phosphate cement under conditions similar to those of the present study but found a slightly higher value (20 MPa versus 13 MPa) (16). The results of the resin composites tested in this study correspond well with the findings of other studies regarding relative as well as absolute values (17, 18). Comparing our results with those of Iazetti et al. and Yap et al., agreement can be seen for the resin composite Tetric Ceram but not for the compomer Dyract AP for which we found a higher flexural strength (19, 20). The fact that resin-modified glass ionomer cement showed higher flexural strength than conventional glass ionomer cement was expected and is in agreement with other studies (21, 22).

The strength and stiffness of a material depend on numerous factors related to composition and structure, and it is only possible to give an incomplete outline of the relationships between structure and properties. Regarding the resin composites, degree of conversion, filler fraction, and silanization of the filler particles play a major role. The higher the degree of conversion, the higher is the strength (23), the higher the filler fraction the stiffer and stronger is the material (24), and a poor silanization leads to inferior mechanical properties (25). As for the glass ionomer cements, they are less strong than resin composites. An explanation may be that the degree of cross-linking of the linear polymer chains in a glass ionomer cement is smaller than is the case with the structure originating from the polymerization of the bifunctional methacrylate monomers of a resin composite. The higher strength of the resin-modified glass ionomer cement as compared to the conventional glass ionomer cement is probably also due to the higher degree of cross-linking in the former material. In a resin-modified glass ionomer cement the cross-linking is brought about by the polymerization of methacrylate side-groups on the polymer chains in addition to the formation of polysalt, characteristic of conventional glass ionomer cements. The ceramic materials had the smallest strength but the highest modulus. This is in disagreement with the general rule that indicates a positive correlation between these two properties (2). An explanation for the low strength is most likely related to defects in the ceramic specimens. Both surface and interior flaws will act as stress amplifiers, in particular in brittle materials such as ceramics, and lead to failure at a relatively low applied force.

The storage time in water affected the materials in different ways. The conventional glass ionomer cement increased both in flexural strength and in flexural modulus during the test period in agreement with previous findings (14, 26). This increase can be explained by an increase in degree of hydration as the cement ages in water. Hydration of the matrix salts is presumed to give increased strength. Furthermore, additional cross-linkage with replacement of carboxyl hydrogen ions by metal ions and increasing predominance of aluminium over calcium in the cross-links is also thought to have a strengthening effect (27). Uno et al. and Williams & Billington also found the relative insensitivity of the resin-modified glass ionomer cement concerning mechanical properties over time (28, 29). Two diametrically directed reactions may take place in the resin-modified glass ionomer cement when it comes into contact with water: a reinforcing reaction similar to the one described for conventional glass ionomer cement and a weakening reaction caused by water uptake of the polymer matrix present in the material. Apparently, the two opposing reactions outbalanced each other under the conditions of this study. The increase in flexural modulus of the polyacid-modified resin composite after 2 weeks water storage contrasts with the effect of time in water on diametral tensile strength reported by Uno et al. (28). They found a decrease in diametral tensile strength similar in magnitude to that of resin composite. The difference may lie in material differences in that the polyacid-modified resin composite used by Uno et al. was a predecessor to the one used in this study, or it may lie in the fact that two different mechanical properties were determined.

The prolonged water storage of 2 weeks for the 2 versions of Doxadent weakened the material. This was not expected, since the setting reaction proceeds for about 2 weeks according to the manufacturer. This weakening may be speculated to stem from water uptake and/or partial dissolution of the cement. If so, this contrasts with the marketed claims of chemical stability and biocompatibility given for Doxadent.

The supposed change in the manufacturing process between the experimental and the marketed version of Doxadent did not lead to any decisive improvements in mechanical properties. Both versions of the ceramic restorative cement showed significantly lower flexural strength and higher flexural modulus than any other restorative material used for posterior restorations tested in this study. Thus the hypotheses put forward in the introduction were confirmed. The flexural strength of Doxadent reached only about 20% of the strength of the polyacid-modified resin composite and the resin composites, materials that are commonly used for Class II restorations. A large clinical study deemed conventional glass ionomer cements unsuitable for permanent Class II restorations because of a high failure rate caused by insufficient mechanical properties (30). When restoring primary teeth with resin-modified glass ionomer cements and polyacid-modified resin composite, instead of conventional glass ionomer cement, Qvist et al. showed a decrease in failure rate (31). The significant correlation seen between in vivo failure rates and in vitro flexural strength values reported for the same materials indicates that the flexural strength of a restorative material influences the longevity of a restoration (32). In vitro studies show that marginal fracture is more common when using a composite with inferior physical properties (33, 34). Marginal breakdown of restoratives with low fracture-related properties has also been found in clinical studies (10, 35).

It can be concluded that as determined by a 3-point bending test, Doxadent had a significantly lower flexural strength and significantly higher flexural modulus than the resin composites, polyacid-modified resin composite, resin-modified glass ionomer cement, and conventional glass ionomer cement also studied. The flexural strength value that is needed clinically is not known, but as discussed above the strength of glass ionomer cements is probably not sufficient for stress-bearing restorations. Since the flexural properties determined in vitro may be dependent on how the specimens were prepared and on the method used, a certain measure of caution should be applied in interpretation of the clinical relevance of the results. However, it appears obvious that specimens prepared in the laboratory have fewer flaws than restorations made in the clinic. Therefore, and with the reservations expressed, it seems warranted to state that despite a possible beneficial effect of a high modulus, the dramatically lower flexural strength of Doxadent may be expected to manifest itself in a relatively high incidence of bulk and marginal fractures. As a consequence, the material cannot be recommended for use in posterior cavities until clinical studies show otherwise.

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