

# Dual-cure resin cements: in vitro wear and effect of quantity of remaining double bonds, filler volume, and light curing

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The present study measured in vitro occlusal wear, quantity of remaining double bonds, and volumetric filler content of eight dual-cure resin cements. Furthermore, the effect of light curing on wear and quantity of remaining double bonds was evaluated. The filler content varied between 31 vol% and 66 vol%. In vitro wear varied between 30  $\mu\text{m}$  and 65  $\mu\text{m}$ , and quantity of remaining double bonds between 19% and 38% when resin cements had been both chemically cured and light cured. When light curing had been omitted, wear varied between 36  $\mu\text{m}$  and 74  $\mu\text{m}$ , and quantity of remaining double bonds between 25% and 56%. One resin cement did not harden when not cured by light. Light curing improved the wear resistance of three resin cements by up to 44% and reduced the quantity of remaining double bonds of six cements by up to 36%. A three-dimensional regression analysis found wear to decrease with decreasing quantity of remaining double bonds and increasing volumetric filler content. A negative correlation was shown between compressive strength and wear. □ *Conversion; dental materials; luting composite; wear resistance*

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The success of tooth-colored inlays depends to a great extent on the resin cement, which must ensure an effective bond between inlay and tooth structure and guarantee the final marginal adaptation. Recently, it has been found that resin cements wear more than the inlay materials themselves, leading to marginal ditching in vivo (1, 2). Together with the mechanical and other physical properties, the wear resistance of a resin cement is determined mainly by the filler type, the particle-size distribution, and the filler content (3–5). Thus, most of the resin cements marketed today are characterized by a high filler content. When highly loaded cements are used, adequate wetting of inlay and tooth and proper seating of inlays are ensured by, for example, ultrasonic insertion (6). The wear resistance, like many other properties, is also, to some extent, influenced by the degree of conversion of the resin cement polymer (7, 8).

By far, most resin cements today are dual-curing. This type of material was developed in an attempt to combine the most desirable properties of the chemical-cure and the light-cure materials and thereby providing adequate curing in deeper areas, reduced inhibition zone, and shorter setting time. Light polymerization of all parts of the cement film is not always possible because of, for instance, substantial thickness of the inlay or blocking of the light by tooth substance. To attain proper polymerization in such situations, minimizing pulpal response and maximizing strength and adhesion of the cement, the chemically activating part of the dual catalyst system should be effective.

It was the purpose of the present study to determine

in vitro wear, quantity of remaining double bonds (RDB), and volumetric filler content of eight proprietary dual-cure resin cements and to evaluate the effect of light curing on wear and quantity of RDB.

## Materials and methods

The eight dual-cure resin cements tested in this investigation are listed in Table 1.

### *In vitro wear*

The wear experiments were carried out by use of a wear machine similar to the one described in detail by de Gee et al. (9). This three-body wear-simulating machine has been found to mimic clinical occlusal wear to a high extent (10). In short, the wear machine consists of two motor-driven cylindrical wheels rotating against each other in a bowl containing a third medium, which is a mixture of white rice and shells of millet seeds in water (11). One wheel accommodates the test specimens. The other wheel, functioning as antagonist, is pressed by a spring force of approximately 15 N against the sample wheel. To simulate the sliding action of antagonist teeth, the surface speeds of the two wheels differ, in this case by 20%.

One by one the resin cements were mixed in accordance with the manufacturers' instructions, placed in a specimen wheel, covered with a matrix, and cured in a Dentacolor XS light-curing oven (Kulzer, Wehrheim,

Table 1. Resin cements tested

| Resin cement          | Code | Manufacturer                               | Batch no.                              |
|-----------------------|------|--|--|
| Dicor L.A. Cement     | A    | Dentsply,<br>York, USA                     | Base: 920115<br>Catalyst: 920429       |
| Dual Cement           | B    | Vivadent,<br>Schaan, Liechtenstein         | Base: 460479<br>Catalyst: 560050       |
| Coltène Duo Cement    | C    | Coltène,<br>Altstätten, Switzerland        | Base: 9212848<br>Catalyst: 9212848     |
| Porcelite Dual Cure   | D    | Kerr,<br>Romulus, USA                      | Base: 750700<br>Catalyst: 751150       |
| Sono-Cem              | E    | ESPE<br>Seefeld, Germany                   | Base: 0006 X091<br>Catalyst: 0008 X091 |
| 3M Luting Material    | F    | 3M,<br>St. Paul, Minn., USA                | Base: 2018B02<br>Catalyst: 2297B01     |
| Twinlook Cement       | G    | Kulzer,<br>Wehrheim, Germany               | Base: 033<br>Catalyst: 033             |
| Vita Cerec Duo Cement | H    | Vita Zahnfabrik,<br>Bad Säckingen, Germany | Base: 9107-463<br>Catalyst: 9107-463   |

Germany) for 90 sec. Ten samples were needed to fill each wheel. Besides one specimen of each of the eight resin cements, each wheel accommodated specimens of two resin composites not included in the present investigation. After individual manufacturing of the 10 specimens, they were all glued to the wheel and wet-ground to obtain a perfect cylindrical outer surface. After storage in water at 37°C for 1 week, the wheel was then mounted in the wear machine together with an 'antagonist' wheel. Before recording of base-line values, a wear run of 20,000 revolutions was performed. The wear experiment itself involved 200,000 revolutions of the specimen wheel. The wear was expressed in micrometers of material loss, recorded with a dial gauge by use of a mounting that ensured measurement at identical points before and after the wear experiment. The antagonist wheel, being narrower than the specimen wheel, resulted in an unworn reference plane of material at each side of the specimen. On each specimen, three rows of recordings were made, each consisting of three recordings across the worn area and one control measurement on each of the two reference planes. The mean difference in wear before (base line) and after the wear experiment was calculated for each of the 10 specimens on the wheel. A total of three wheels were produced and run in the wear machine.

#### Degree of conversion

The quantity of RDB was determined by a method based on the one developed by Ruyter & Györösi (12). The method is applicable to resins with aromatic moieties (such as, for example, bisphenol-A-glycidyl dimethacrylate (BISGMA)) and involves the recording of a transmission infrared spectrum of a resin before and after polymerization. The quantity of RDB was obtained as follows: from the spectrum of the monomer the absorbance  $^{MA}(=)$  of the C = C band at 1640 cm<sup>-1</sup>

and the absorbance  $^{MA}(...)$  of the aromatic C...C band at 1610 cm<sup>-1</sup> were determined by use of the base-line method (13). Likewise, from the spectrum of the resulting polymer the absorbances  $^{PA}(=)$  and  $^{PA}(...)$  at the same frequencies were determined. The ratio between  $^{PA}(=)/^{PA}(...)$  and  $^{MA}(=)/^{MA}(...)$  was then taken as the fraction of unreacted double bonds in the polymer.

To calculate the quantity of RDB of the resin cements not containing BISGMA but rather urethane species, another reference band whose absorbance does not change during polymerization had to be used. The N-H peak at 3380 cm<sup>-1</sup> was chosen, as this absorbance peak has earlier been deemed serviceable as internal standard (14, 15).

The infrared spectra were recorded by means of a Beckman IR spectrophotometer, Acculab 9 (Beckman Instruments, Inc., Fullerton, Calif., USA). The spectra of base and catalyst (monomer) and of the cured resin cement (polymer) were recorded. The spectra of the monomer were obtained immediately after approximately 10 mg of either base or catalyst had been pressed into a thin film between two KBr discs. The spectra of the polymer were obtained in the following manner: equal amounts of base and catalyst were mixed in accordance with the manufacturer's instructions. Approximately 10 mg of the mixed resin cement was compressed between two glass plates. Half of the cement film specimens were then irradiated for 80 sec on each side (Visilux 2, 3M, St. Paul, Minn., USA). Thirty minutes after start of the mix the assembly was put in water at 37°C. After 1 day the resin cement film was separated from the glass plates, reduced in diameter to fit the holder of the spectrophotometer, and returned to the waterbath. After another 6 days a spectrum of the resin cement was recorded. All experiments were carried out in triplicate, and the mean value and standard

Table 2. Mean values and standard deviations of in vitro wear ( $\mu\text{m}$ ), quantity of remaining double bonds (RDB) (%), and filler content (vol%) of resin cements. Values with same lettering within each of the five series and values connected by horizontal lines were not significantly different

| Resin cement | Wear ( $\mu\text{m}$ )     |                            | RDB (%)                   |                            | Filler content (vol%)     |
|--------------|----------------------------|----------------------------|---------------------------|----------------------------|---------------------------|
|              | +Light                     | -Light                     | +Light                    | -Light                     |                           |
| A            | <sup>c</sup> 65 $\pm$ 1.4  | <sup>c</sup> 74 $\pm$ 4.8  | <sup>a</sup> 19 $\pm$ 2.8 | <sup>b</sup> 28 $\pm$ 0.4  | <sup>a</sup> 31 $\pm$ 0.2 |
| B            | <sup>a</sup> 30 $\pm$ 2.7  | <sup>a</sup> 36 $\pm$ 6.8  | <sup>a</sup> 20 $\pm$ 0.5 | <sup>a</sup> 25 $\pm$ 2.2  | <sup>b</sup> 37 $\pm$ 1.3 |
| C            | <sup>c</sup> 45 $\pm$ 3.9  | <sup>ab</sup> 51 $\pm$ 2.9 | <sup>b</sup> 27 $\pm$ 1.2 | <sup>b</sup> 29 $\pm$ 0.7  | <sup>d</sup> 49 $\pm$ 2.0 |
| D            | <sup>b</sup> 38 $\pm$ 3.3  | <sup>a</sup> 44 $\pm$ 5.5  | <sup>d</sup> 35 $\pm$ 0.6 | <sup>d</sup> 37 $\pm$ 0.1  | <sup>c</sup> 44 $\pm$ 0.2 |
| E            | <sup>ab</sup> 34 $\pm$ 2.0 | <sup>b</sup> 61 $\pm$ 8.1  | <sup>d</sup> 38 $\pm$ 0.5 | <sup>c</sup> 44 $\pm$ 0.5  | <sup>e</sup> 56 $\pm$ 0.9 |
| F            | <sup>ab</sup> 34 $\pm$ 0.8 | <sup>ab</sup> 49 $\pm$ 8.3 | <sup>c</sup> 31 $\pm$ 1.7 | <sup>cd</sup> 35 $\pm$ 0.8 | <sup>e</sup> 66 $\pm$ 0.0 |
| G            | <sup>b</sup> 39 $\pm$ 4.7  | <sup>a</sup> 46 $\pm$ 3.1  | <sup>b</sup> 28 $\pm$ 1.0 | <sup>c</sup> 34 $\pm$ 1.5  | <sup>f</sup> 59 $\pm$ 1.7 |
| H            | <sup>d</sup> 56 $\pm$ 1.5  | —                          | <sup>d</sup> 36 $\pm$ 1.1 | <sup>f</sup> 56 $\pm$ 1.0  | <sup>c</sup> 45 $\pm$ 0.4 |

deviation of the three results computed. The values of absorbance of the monomer was found as the mean value of the base and catalyst values.

*Filler volume*

The content of filler by weight was determined after burning of the resin cement at 600°C for 30 min. The content by volume of inorganic fillers in the resin cements was determined by a pycnometric method. The density of the polymerized resin cement and of the inorganic filler was determined by measuring the weight of a volume-calibrated pycnometer filled with water in

which a known quantity of cured resin cement or filler was immersed. The sample volume equals the pycnometer volume minus the undispersed volume of water of known density. For each resin cement three density determinations were made.

*Statistical methods*

The mean values of each of the five comparable series were first analyzed by analysis of variance (16). Statistically significant differences between the mean values of any series were then determined with the Newman-Keuls multiple range tests (17). To detect any

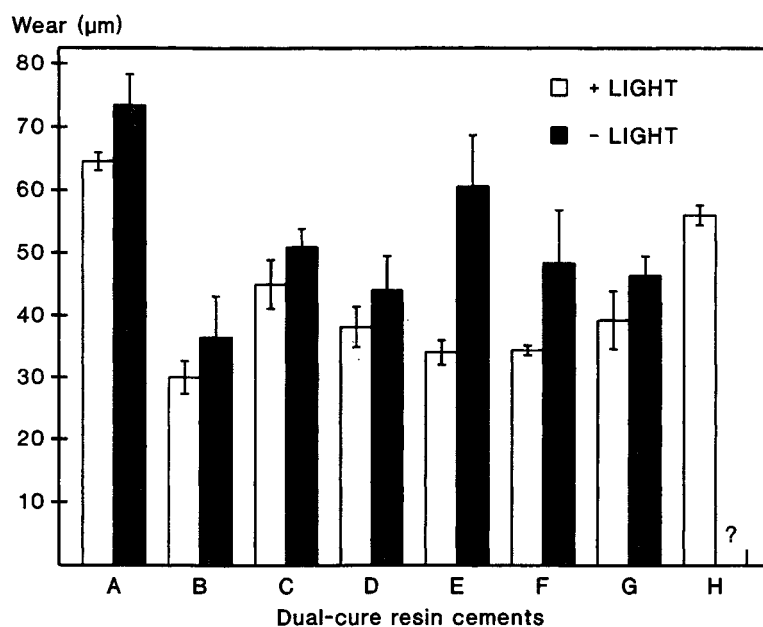


Fig. 1. Mean values and standard deviations of in vitro wear ( $\mu\text{m}$ ) of light-cured and non-light-cured resin cements A-H (Table 1).

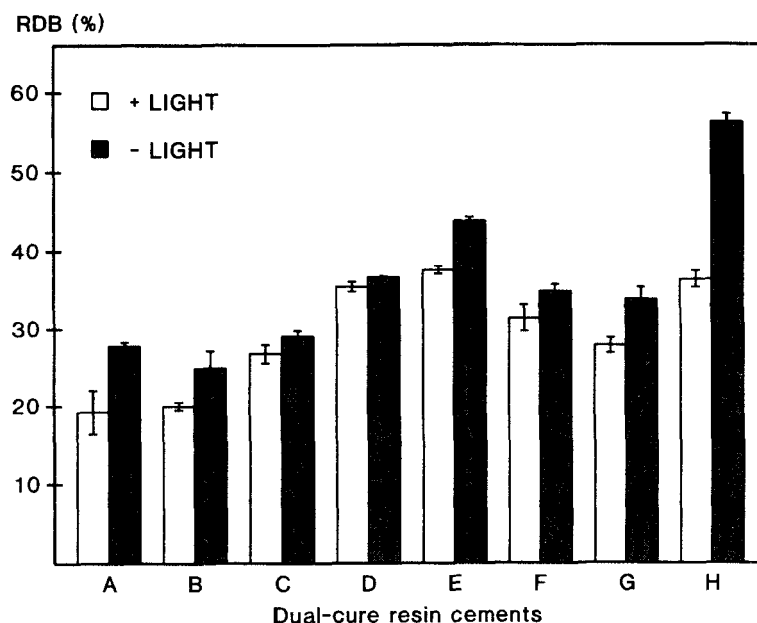


Fig. 2. Mean values and standard deviations of quantity of remaining double bonds (RDB) (%) of light-cured and non-light-cured resin cements A-H (Table 1).

general effect of light curing on wear and/or quantity of RDB, *t* tests of matched pairs were carried out (18). By use of a pooled standard deviation for wear ( $SD = 4.6$ ) and for RDB ( $SD = 1.2$ ), *t* tests were used to analyze the effect of light curing on each of the eight resin cements (16). In search of possible correlations between any or all of the three variables in vitro wear, quantity of RDB, and filler volume, two-dimensional linear regression analyses and a three-dimensional regression analysis were performed (16).

## Results

The results are shown in Table 2 and Figs. 1–3. The eight (seven) mean values of wear and RDB with or without light curing, respectively, and of filler content were found to differ with statistical significance ( $P < 0.005$ ). Within each of the five groups of results in Table 2, identical lettering designates mean values with no statistically significant difference. Thus, after having been light-cured, B, E, and F wore the least and A the most. A and B showed the least quantity of RDB, and D, E, and H the highest. In the non-light-cured series B, C, D, F, and G wore the least and A the most. H did not polymerize to such an extent that the resin became hard and could be used as wear specimens. B showed the least quantity of RDB, and H the highest. The filler content varied between 31 and 66 vol%. A had the lowest filler volume, F the highest.

Generally, light curing decreased wear ( $P < 0.01$ )

and quantity of RDB ( $P < 0.02$ ). With regard to the effect of light curing on each of the eight resin cements, horizontal lines in Table 2 connect values that did not differ; that is, light curing had no effect. Thus, it was found that light curing decreased the wear of A ( $P < 0.05$ ), E ( $P < 0.005$ ), and F ( $P < 0.01$ ) and decreased the quantity of RDB of A ( $P < 0.001$ ), B ( $P < 0.005$ ), E ( $P < 0.005$ ), F ( $P < 0.025$ ), G ( $P < 0.005$ ), and H ( $P < 0.0005$ ).

A significant negative correlation was found between filler volume and wear ( $P < 0.01$ ), whereas no significant correlation was found between quantity of RDB and wear. The following relationship was determined among wear ( $Y$ ), quantity of RDB ( $x_1$ ), and filler volume ( $x_2$ ):  $Y = 55 + 0.65x_1 - 0.58x_2$ . Both coefficients of regression differed from zero ( $b_1, P < 0.05$ ;  $b_2, P < 0.01$ ). Thus, wear decreased with decreasing quantity of RDB and increasing filler volume.

## Discussion

The eight resin cements varied significantly with regard to all three investigated variables: wear (+light), 30–65  $\mu\text{m}$ ; quantity of RDB (+light), 19–38%; and filler content, 31–66 vol%. The effect of light polymerization also varied considerably. Reductions in quantity of RDB of up to 36% and in wear of up to 44% were seen as a consequence of light curing. The quantity of RDB measured reflects not only the effect of the two catalytic mechanisms in a resin cement but also characteristics

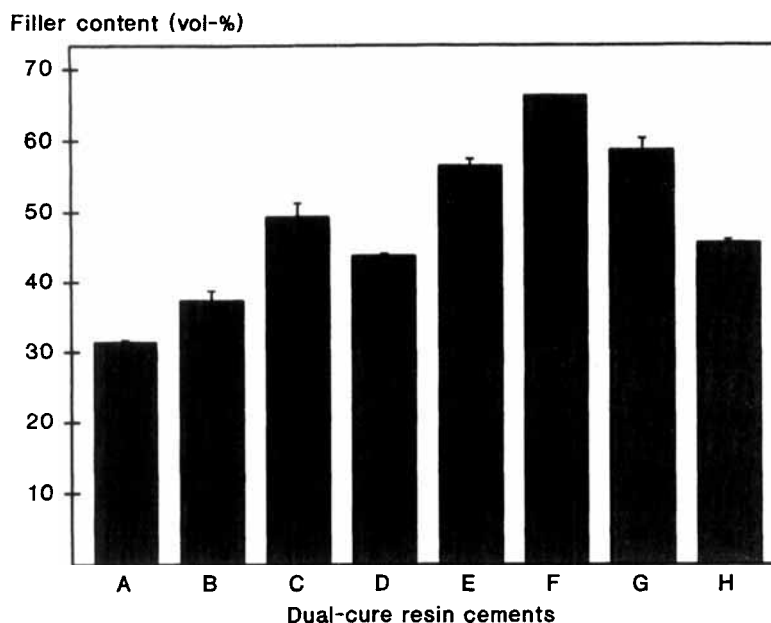


Fig. 3. Mean values and standard deviations of filler content (volume %) of resin cements A-H (Table 1).

of the overall composition of the resin cement—for example, the types of monomer used and the ratio between the monomers. Differences between the cements with regard to the composition probably account for the difference in the overall level of conversion between the resin cements. Differences in efficiency of the chemical-cure part and the light-cure part of the catalyst system then account for the differences in the effect of light curing. Hasegawa et al. (19) found Dual Cement (B) and Dicor L.A. Cement (A) to behave mainly as light-cured materials, whereas Coltène Duo Cement (C) had a strong chemical-cure part in the catalyst system. These findings conform rather well with the present findings. Thus, wear and quantity of RDB of Coltène Duo Cement (C) had reached a minimum already without light curing, whereas light curing resulted in a significant decrease in wear and/or quantity of RDB of Dicor L.A. Cement (A) and Dual Cement (B). With regard to Vita Cerec Duo Cement (H), the chemical-cure part of the catalyst system seemed to be non-existing, as the resin cement would not harden when light polymerization was omitted. Consequently, this cement cannot be recommended in situations in which effective light curing cannot be guaranteed.

The wear process in vivo is very complex, and the resistance of a certain material to wear reflects many interplaying factors of the process and of the properties of the material. Filler content and degree of conversion were found to be among these factors. All else being equal, the higher the filler content and the degree of

conversion, the higher the wear resistance. Information about the filler content of a specific composite is readily accessible, whereas information about degree of conversion obtainable is very difficult to get. Consequently, to use filler content as a criterion of selection for resin cements seems practicable and recommendable. It should be borne in mind that highly filled resin cements often are very viscous. Some mode of vibration must therefore be applied during cementation of the restoration to ensure proper wetting and seating.

As new materials have to be screened and approved in the laboratory before being released on the market, a test able to predict in vivo wear performance reliably has been needed (20). Many methods with different levels of sophistication have been designed, with various success in ranking the materials in the same order in which they were ranked clinically. As mentioned in the introduction, the wear-simulating machine used in the present study has proved to be one of the more qualified in this respect. Only a few studies and a few resin cements have been compared clinically with regard to wear performance. However scarce, the existing data lend support to the findings of the present study and to the justification of the wear machine used. In comparison with cements not included in this study, 3M Luting Material (F) and Dual Cement (B) were, for example, reported to have good wear resistance and Dicor Cement (A) to have poor wear resistance (21, 22).

Apart from highly sophisticated methods, simple mechanical properties have been correlated to clinical wear behavior, generally without much success (20).

On the basis of the present wear experiments, linear regression analysis was carried out between compressive strength and the mean values of wear (+light). The regression analysis was based on the following values of compressive strength: A, 266 MPa; B, 370 MPa; C, 269 MPa; D, 330 MPa; E, 410 MPa; F, 390 MPa; G, 320 MPa; and H, 274 MPa. The values were provided by the manufacturers on request, and compressive strength was the only mechanical property on which all eight manufacturers reported. The analysis found a negative correlation of statistical significance between compressive strength and wear ( $r = -0.83$ ,  $P < 0.01$ ). The finding of this correlation suggests that measurement of compressive strength could be of relevance when the clinical performance of a resin cement has to be predicted.

In conclusion, the present study showed substantial differences in in vitro wear, quantity of RDB, volumetric filler content, and effect of light curing of the eight dual-cure resin cements investigated. Linear correlations were found between wear, on the one hand, and quantity of RDB, filler content, or compressive strength, on the other hand.

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