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Comparison of Flexural Strength of Resin Cements After Storing in Different Media and Bleaching Agents

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Authors

Farideh Geramipanah *
Susan Mir Mohammad Rezaei #
Maryam Jafary #
Leyla Sadighpour #

Address for Correspondence

Dr. Leyla Sadighpour #
Email: sedighle@tums.ac.ir
lsedigh@yahoo.com
Phone: +98 21 8805860
Fax: +98 21 8805800

* *Department of Prosthodontics, School of Dentistry, Dental Implant Research Center, Tehran University of Medical Sciences, Kargar Shomali, Hakim HWY, 143995591 Tehran, Iran*

\# *Department of Prosthodontics, School of Dentistry, Tehran University of Medical Sciences, Kargar Shomali, Hakim HWY, 143995591 Tehran, Iran*

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ABSTRACT

The aim of this study was to determine the effects of different storage media and bleaching treatments on the flexural strength of two resin cements (Panavia and BisCem). One hundred rectangular-shaped specimens were prepared with two resin cements and were stored in five media types (n=10): distilled water (DW), lactic acid (LA), sodium hydroxide (NH) , in-office bleaching (OB) and home bleaching (HB). There was significant interaction between the solutions and cements ($p<0.05$). The lowest three-point flexural strength was found in sodium hydroxide for both cements ($p<0.05$). Both cements exhibited significant increase in flexural strength following home and in-office bleaching (except Panavia in OB) ($p<0.05$) compared with immersion in distilled water. Panavia recorded significantly higher flexural strength compared with BisCem in all media ($p< 0.002$), with the exception of in-office bleaching.

INTRODUCTION

Resin cements have become popular in dental clinics because of their ability to bond to a wide range of materials and substrates, including tooth structure and restorative materials.[1] In spite of the superior mechanical properties of resin cements, it has been demonstrated that their microstructure can be deteriorated by exposure to oral fluid which makes them vulnerable to surface loss, degradation and fracture.[2,3]

It is evident that the degradation mechanisms are induced by a wet environment by hydrolysis, in which water penetrates into the polymer structure and breaks down the cross-linked matrix and/or unprotected carbonic double bonds. As a result, oligomers and monomers are formed along with unbounded monomers, which subsequently leach out from the matrix causing softening and weakness.[3-5] Accordingly, water may attack the filler/matrix bond, de-bonding the filler/matrix interface and forming cleavage. The produced flaws could trigger crack propagation under various stress conditions applied in the oral environment.[4,6] Dissolving of the cement layer also promotes marginal microleakage and recurrent caries as well as marginal staining.[7] Because hydrolysis involves the interaction of H + and OH – ions with the polymeric structure, it was proposed that pH variations may influence the process and therefore the resin structure and material properties.[8,9] The pH of an oral environment can be affected by the types of food and beverages ingested. In addition, organic acids

such as lactic acid, propionic acid and acetic acid, which are produced by oral bacteria, are considered an important cause of decreasing pH in the mouth.[10-12]

Certain dental treatments, such as tooth whitening, have also been regarded as a source of pH alteration in an oral cavity. Bleaching agents have received considerable attention recently as the popularity of teeth whitening treatments has increased. The treatment regarded conservative and accessible to the public through over-the-counter products, as well as in-office procedures.[13,14]

Different commercial bleaching products contain varied concentrations of acidic or basic components, in which a greater peroxide concentration results in a more acidic pH.[15] Chemically, bleaching ingredients such as carbamide peroxide (CP) and hydrogen peroxide (HP) are unstable and break down to produce free radical molecules. These molecules easily penetrate through deep parts of teeth and restorations and may affect the polymeric network of resin materials.[16]

Following evidence revealing that bleaching agents may have modified dental hard tissue, some concerns have arisen on the influence of these solutions on restorations and restorative materials.[14,17] Previous studies have focused on the effect of bleaching agents on the superficial characteristics and certain mechanical properties of a variety of resin composites.[18-20] However, the effect of bleaching materials on the mechanical properties of resin cements has been less considered,[5,21] even though the strength of a cement plays a crucial role in its resistance to mechanical loading and the dislodgement of restorations.[22]

The aim of this study was to determine the effect of different storage media and bleaching treatments on the flexural strength of two self-adhesive resin cements. The null hypothesis was that the flexural strength would not be affected by different storage media and bleaching treatments.

METHOD AND MATERIALS

EXPERIMENTAL DESIGN

A total of 100 rectangular-shaped specimens were prepared with two adhesive cements using a stainless steel mold (L×W×H: 25×2×2 mm).[23] The material specifications are summarized in Table 1. A self-adhesive cement (BisCem) and a self-etch dual-cured cement (Panavia F2) were examined. The cements were mixed according to the manufacturer's instruction, and the molds were filled with the paste in a single increment. Excess cement was removed by pressing the two sides of the mold against glass slabs and polyester strips. The cements were light polymerized from the top and bottom using

Table 1. The specifications of the materials evaluated

Product name	Manufaturer	Material type	Composition
Panavia F2	Kuraray Medical INC Okayama Japan	Dual-cured Adhesive Resin cement	Paste A: 10-MDP, Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Hydrophilic aliphatic dimethacrylate, Silanated silica filler, silanated colloidal silica, dl-camphorquinone, catalyst, initiator Paste B: Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Hydrophilic aliphatic dimethacrylate, Silanated barium glass filler, sodium flouride, catalyst, accelerator, pigment
BisCem	Bisco Schaumburg USA	Dual-cured Self-Adhesive Resin cement	Paste A:BisGMA, unreacted dimethacrylate monomer, glass filler Paste b: Phosphate acidic monomer, glass filler
Xtra Power WHITEsmile Home bleaching	Birkenau, Germany	Home bleaching gel	22% Carbamide peroxide
Xtra Power WHITEsmile In-office bleaching	Birkenau, Germany	In- office bleaching gel	38% hydrogen Peroxide gel

an LED light cure unit (Bluephase 16i) for 20 s at a distance of one millimeter and a light intensity of 1600 mW/cm2, overlapping the previously irradiated section. After removing them from the mold, specimens were stored in an incubator at 37°C and 100% humidity for 24 h in order to reach to their ultimate strength. Before immersion in media, specimens were finished with silicon carbide papers (600, 1200 and 2400 grit) on both sides. Specimens were randomly assigned to three storage media and two bleaching agents (n=10). The storage media included distilled water (DW), lactic acid (LA), sodium hydroxide (NH), Home bleaching (HB) and office bleaching (OB). Each solution pH was measured using a digital pH-meter (PH 960, Precisa Instrument AG, Dietikon, Switzerland) with accuracy of 0.01. The device was initially calibrated using buffer solutions provided by the manufacturer at pH=4.00 and pH=7.00. Potentiometric and reference electrodes were connected. Sample solutions were then placed in the magnetic stirrer and pH was recorded (Table 2). A temperature sensor was also mounted to the stirrer to monitor sample temperature. 50cm^3 of each solution was placed in lightproof sealed containers. The storage time was one month. Solutions were refreshed daily to ensure pH consistency.

The home-bleaching (HB) treatment was performed using a flexible-plastic, vacuum-formed material (Sof-Tray). The trays were filled with a 22% CP gel (Xtra Power) for eight hours, after which specimens were rinsed under running water, cleaned and kept in their containers in DW. The procedure was repeated for five days. A longer exposure scenario was simulated by applying the in-office bleaching (OB) procedure followed by the HB procedure.[24] The OB treatment consisted of four consecutive applications of a 38% HP gel (Xtra Power) for fifteen minutes, at eight-hour intervals. The specimens were rinsed gently under running water and stored in DW for twenty-four hours, followed by the HB protocol as described earlier.

Table 2. Corresponding pH of the tested media

Sample Media	Composition	pH
DW	Distilled water	7.1
NH	Sodium hydroxide (0.1 N)	13.0
LA	Lactic acid (0.01 M)	2.7
OB	38% hydrogen peroxide	6.1-6.4
HB	22% carbamide peroxide	5.4-5.8

FLEXURAL TEST

The three-point bending test (3PBT) loads were applied using a universal testing machine (Zwick Roell Z050, Ulm, Germany) with a crosshead speed of 0.5 mm/min. The flexural strength (σ) was obtained in megapascals (MPa) using the following formula:

$$\sigma = \frac{3FL}{2bh^2}$$

(Eq. 1)

where F is the maximum load exerted on the specimen (N), L is the distance between the supports (20mm), b is the width and h is the height of the specimen.

STATISTICAL ANALYSIS

Data obtained from the flexural tests were submitted to a two-way analysis of variance (ANOVA) to reveal any interactions between the cements and the storing media (SPSS version 13). A one-way ANOVA was used to evaluate the effect of media on the cements, followed by a Tamhane *post hoc* analysis. The mean difference of flexural strength of the two cements was also analyzed using a t-test. The significance level for all tests was set to α=0.05.

RESULTS

The mean values of flexural strength for the two cements in different media are summarized in Table 3, Table 4 and Figure 1. The highest flexural values were found in the HB group for both cements (64.86 MPa for Panavia F2 and 51.88 MPa for BisCem). While, the lowest flexural values were observed in the NH group for both cements (9.76 MPa for Panavia F2 and 4.41 MPa for BisCem). The two-way ANOVA showed significant interactions between the solutions and cements ($p < 0.05$). It

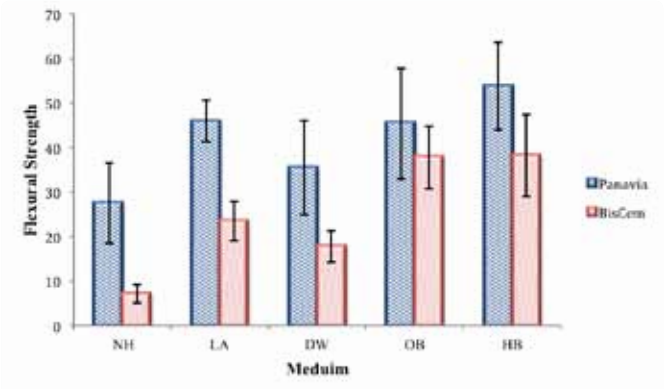

Figure 1. The mean flexural strength values (MPa) for two cements in five media. Vertical line on the top of each bar demonstrates standard deviation.

follows that the effect of media on each cement and the difference of each cement in each media should be compared separately.

The media affected the flexural strength of the cements significantly ($p<0.05$). Multiple comparisons between the effects of each media on the cements are shown in Table 3 and 4.

The lowest three-point flexural strength was found in NH for both cements ($p<0.05$). Both cements exhibited significant increase in flexural strength following HB and OB (except Panavia in OB) ($p<0.05$) compared with DW. Flexural strength of Panavia F2 was 22.25% lower in NH and increased 31.5 % after HB. While strength value of BicCem decreased 59.7% in NH and improved 53.1 % after HB.

The post-hoc analysis of the difference between the mean strength of the two cements revealed that Panavia F2 recorded significantly higher flexural strength compared with BisCem in each media ($p< 0.002$) except for OB.

DISCUSSION

In the present study, different storage media significantly affected the flexural strength of the two resin cements. Therefore the null hypothesis of the study was rejected.

In general, strength is an important property of a cement, enabling it to withstand the mechanical stress applied in an oral environment with variable chemical and temperature conditions.[22] Although the effect of storage media on the fracture toughness and shear punch test of several luting agents has been investigated in a number of studies,[5,21] flexural tests are common for assessing strength characteristics and recommended by the International Organization for Standardization (ISO).[23] The 3PBT was used in our study. However, the test fails to successfully predict the clinical behavior of tested materials.[25] Nevertheless, the test values are helpful when comparing and ranking the materials of choice.

To understand the influence of different solutions on the flexural strength of the cements, specimens were immersed in acidic and basic solutions. A number of studies suggested using a very high alkaline medium (pH=13.0) or a very low acidic

Table 3. Descriptive data of mean flexural strength of Panavia F2 and the multiple comparisons in each media

Storage medium	Minimum strength (MPa)	Maximum strength (MPa)	Mean (MPa)	Sig.
NaOH	9.76	40.59	27.64 (9.10)	a
Lactic acid	39.76	55.51	45.90 (4.71)	b
Distilled water	18.16	49.54	35.51 (10.56)	abc
In-office bleaching	11.73	54.79	45.53 (12.44)	bc
Home bleaching	38.92	64.86	53.78 (9.86)	bcd

Items in columns with different letters are significantly different at the 95% level.

Table 4. Descriptive data of mean flexural strength of BisCem and the multiple comparisons in each media.

Storage medium	Minimum strength (MPa)	Maximum strength (MPa)	Mean (MPa)	Sig.
NaOH	4.41	10.82	7.23 (2.08)	a
Lactic acid	16.59	29.94	23.59 (4.45)	b
Distilled water	14.19	25.70	17.89 (3.58)	c
In-office bleaching	25.62	45.91	37.82 (7.00)	cd
Home bleaching	18.46	51.88	38.22(9.25)	bd

Items in columns with different letters are significantly different at the 95% level.

medium (pH<2.0) for accelerated hydrolysis.[7] In the present study, acidic pH=2.7 and basic pH=13 solutions were examined, as they are close to extreme oral conditions. In addition, because hydrolysis by water is a slow process, this study used NH (0.1 N), which provides 106 times more OH- ions, to accelerate the hydrolysis process.[6] The results of the present study showed that the NH medium, followed by DW, had the strongest effect on decreasing the flexural strength of both cements. Although similar studies have not been conducted on resin cements, Medeiros *et al*.[9] also found that a universal resin composite stored in 100% ethanol obtained the lowest diametral strength when compared with water. On the other hand, Deepa *et al*.[26] found that the adverse effects of storage media on the mechanical properties of several resin composites was higher when the specimens were exposed to 75% ethanol compared with water and acidic solutions. It was believed that hydrolysis of polymer-filler content is accelerated when hydroxyl ion attacks the siloxane bond of the silane coupling agent and the ester covalent bond between the resin/filler interface. The result of both mechanisms is a weakened filler/resin bond, which ultimately leads to filler exfoliation and crack formation.[5,10] It has also been suggested that any crack or pore acts as a weak link that could propagate upon loading or any other form of stress, until catastrophic failure occurs.[7,25] On the other hand, in the present study, both cements showed superior strength when exposed to LA compared to DW and NH. Potential reasons for such a difference lies in the role of the H+ ion in the hydrolysis process. It is postulated that the result of water diffusion into the polymeric structure and the oxidation of reactive sites is methacrylic acid.[3] Polymer hydrolysis is a slow-rate diffusion-controlled reaction, and increased concentration of the H+ ion, such as in an mild acidic pH, could increase the rate of forming an equilibrate state. In this circumstance, the hydrolysis is reduced. Therefore, polymer hydrolysis is slower in acidic media than in water or basic media.[10]

An interesting observation in this study was the superior performance of two cements after the bleaching treatment. Carbamide Peroxide or Hydrogen Peroxide is used as active ingredient in bleaching agents, both of which decompose to free radicals. Free radicals are responsible for the oxidation of unprotected double bonds in the resin structure, either in the polymeric matrix or at the filler/matrix interface. For bleaching materials that were tested in the present study, the measured pH ranged from 5.9 to 7 for in-office and from 6.1 to 6.4 for home bleaching. The mild acidity of the bleaching products that was used in the current study, could be attributed to other ingredients that are added to the composition for enhancing certain properties of these materials. Carbopol (carboxypolymethylene), for instance, is an organic acid and a common thickening agent that prolongs the release of hydrogen peroxide.[27] Thus, the mild acidic pH of bleaching materials may illustrate the higher strength achieved for both cements in this study that has been discussed earlier. However, due to differences in the active and inactive ingredients of the bleaching products, method of usage and the resultant pH of the materials, different results may be expected.[15,19] In order to clarify its effects on the tested materials, specific details of the composition of each product would be required but are not available.

Panavia F2 showed higher flexural strength comparing to BisCem in all solutions but OB. Due to the lack of relevant studies, direct comparison with other investigations was not possible due to the lack of similar studies. However, available investigations revealed that the effects of bleaching on restorative materials were material-dependent.[16,17,20] The monomers and polymerized structure of Panavia F2 are more hydrophobic than BisCem. Hydrophilic monomer could be more susceptible than hydrolysis induced by water and/or acidic solutions. More hydrophobic monomers in Panavia may explain the overall higher strength of Panavia F2. In addition, the setting reaction in self-adhesive resin cements such as Biscem, involves two sets of partially overlapped reactions including free-radical polymerization of organic matrix and the acid-base reaction between the acidic monomer and the acid-soluble filler and calcium ion on the tooth. Therefore, it may be concluded that the solubility of filler is higher in BisCem compared with Panavia F2.[28]

Other factors contributing to the hydrolysis of resin cements include the polymer constituents, filler size and the type and quality of the filler/matrix interface.[12] For example, the ratio of triethyleneglycol-dimethacrylate (TEGDMA) to bisphenol A glycidyl methacrylate (Bis-GMA) in the resin matrix is shown to have influence on the hydrolysis of the matrix: with a higher TEGDMA percentage, higher degradation is expected.[29,30] Panavia F2 lacks TEGDMA, which may account for its better performance compared to BisCem. In addition, the existence of 10-MDP in Panavia F2 is responsible for a more hydrophobic matrix in this cement and more stability in hydrolytic media.[5]

In the present study the flexural strength of the two cements were not significantly different following immersion in the two bleaching agents compared with other solutions.

Hatanaka *et al*.[20] found that, although an HB product containing 10% CP significantly changed the Knoop hardness of composites, the influence on flexural strength was not significant. Due to the difference in materials and the different protocols and test methods that have been incorporated across the literature, a consistent conclusion is hard to make.

As a limitation of our study, the dimensions of the specimens used were not close to real-life size. Therefore, further investigation is suggested to examine the effects of different media and/or bleaching agents on the resin layer with relevant geometry and tested with the restoration in place.

CONCLUSION

Within the limitations of this study, bleaching treatments (in-office and home) increased the flexural strength of either Panavia F2 or BicCem in compared with other solutions. Furthermore, storage media with different pH levels, as tested in this study, showed diverse effects on the cements. Flexural strength deterioration was more influential when pH was increased.

MANUFACTURERS' DETAILS

- BisCem, Bisco Inc., Schaumburg, IL, USA
- Panavia F2.0, Kurary, Osaka, Japan
- Bluephase 16i, Ivoclar Vivadent Schaan, Liechtenstein
- Sof-Tray, Ultradent, South Jordan, UT, USA
- Xtra Power, WHITEsmile, Birkenau, Germany
- SPSS version 13, Chicago, IL, USA

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