치의학석사 학위 논문

Comparison of dental resin cements used for zirconia restorations by diametral tensile strength and fracture toughness

지르코니아 수복물에 사용하는 치과용 레진 시멘트간의 간접인장강도와 파괴 인성의 비교

2012년 08월

서울대학교 대학원

치의과학과 치과보철학 전공

J.I. Shaginyan

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이 논문을 치의학석사 학위논문으로 제출함

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J.I.Shaginyan의 석사학위논문을 인준함

2012년 8월

위 원 장 _______________ **(인)**

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위 원 ____________________ **(인)**

학위논문 원문제공 서비스에 대한 동의서

본인의 학위논문에 대하여 서울대학교가 아래와 같이 학위논문 저작물을 제공하는 것에 동의합니다.

1. 동의사항

①본인의 논문을 보존이나 인터넷 등을 통한 온라인 서비스 목적으로 복제할 경우 저작물의 내용을 변경하지 않는 범위 내에서의 복제를 허용합니다.

②본인의 논문을 디지털화하여 인터넷 등 정보통신망을 통한 논문의 일부 또는 전부의 복제.배포 및 전송 시 무료로 제공하는 것에 동의합니다.

2. 개인(저작자)의 의무

본 논문의 저작권을 타인에게 양도하거나 또는 출판을 허락하는 등 동의 내용을 변경하고자 할 때는 소속대학(원)에 공개의 유보 또는 해지를 즉시 통보하겠습니다.

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②서울대학교는 본 논문에 대한 공개의 유보나 해지 신청 시 즉시 처리해야 합니다.

논문제목

Comparison of dental resin cements used for zirconia restorations by diametral tensile strength and fracture toughness

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-Abstract-

Comparison of dental resin cements used for zirconia restorations by diametral tensile strength and fracture toughness

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Purpose

The purpose of this study was to compare diametral tensile strength and fracture toughness of four dental resin cements used for zirconia restorations.

Materials and Methods

Four resin cements (Panavia F2.0, Zirconite, Clearfill SA luting, Rely-X Unicem) were investigated. Specimens of the materials were prepared in a cylindrical stainless steel mold (ø 4 × 6 mm) for diametral tensile strength test and in a mold (20 × 6 × 4 mm) with a single-edge notch in the middle for fracture toughness test (n = 10). All specimens were stored in air at 37^{o}C for 24 hours. Both tests were conducted using an Instron universal testing machine at a crosshead speed of 0.5 mm/min and the maximum failure load was recorded at 23 ± 1^{o}C. The data were statistically analyzed using one-way ANOVA and the multiple comparison Scheffé test using SPSS (α = 0.05).

Result

The diametral tensile strength found to be the highest in Panavia F2.0 (37.77 ± 2.72 MPa) followed by Rely-X Unicem (31.98 ± 2.92 MPa) and Zirconite (30.43 ± 2.06 MPa). However, diametral tensile strength between Zirconite and Clearfill SA luting (26.37 ± 6.75 MPa) was not found to be significantly different. Panavia F2.0 (1.52 ± 0.16 $MNm^{-1.5}$) had the highest fracture toughness, while fracture toughness of Zirconite (1.18 ± 0.16 $MNm^{-1.5}$), Clearfill SA luting (1.18 ± 0.1 $MNm^{-1.5}$) and Rely-X Unicem (1.18 ± 0.06 $MNm^{-1.5}$) was found similar, but significantly lower than one of Panavia F2.0.

Conclusion

Among investigated cements the highest diametral tensile strength and fracture toughness were found for Panavia F2.0. In cementation of zirconia restorations Panavia F2.0 is believed to withstand load forces better, than other investigated cements, demonstrating high resistance to fracture.

Keywords

Diametral tensile strength; Fracture toughness; Resin cements; Yttria-stabilized tetragonal zirconia.

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Contents

I. INTRODUCTION

Recent growth of interest in esthetics among patients resulted in rapid increase of CAD/CAM yttria-stabilized tetragonal zirconia restorations. For such successful esthetic restoration usage of cements with bonding abilities is strongly recommended. Resin cement might be a perfect settlement for treatment with high esthetic demands. However, using of CAD/CAM system accentuates accuracy of prosthesis. In general, gap widths would be increased from the crown margin to the occlusal surface, where the widest gaps are evident [1]. CAD/CAM techniques involve scanning, software and machining procedures, what impair the fit of CAD/CAM zirconia restoration [2]. Increased gap would be filled with the resin cement, and the film thickness would increase, compare to the film under conventional metal-ceramic restoration. This causes increased stress concentration in cement material between the restoration and its abutment. Especially this concentration of stress is evident at the sites of increased occlusal load, such as working cusps of molars and premolars or incisal part of upper teeth [3]. In addition, bonding resin cement plays important role in durability of the restoration. Compare to full crown restorations, what encircles all abutment, stress concentrations occur impressively in cements beneath CAD/CAM ceramic onlays or inlays restorations. The supporting cement should be able to withstand such loads, what gives longer life for prosthesis and, as following result, increases patient's satisfaction with dental treatment.

The other important role of resin cements in zirconia dental restoration is increasing its longevity with protection of the margin from leakage and secondary caries [4]. Compare to acid-base reaction luting cements, dual-curing resin cements have low solubility, and high mechanical strength [5,6]. Values of the diametral tensile strength and fracture toughness (K_{IC}) can help identify resin cement with high strength among several kinds of resin cements available to clinical practitioners.

Tensile strength is one of the important mechanical properties of material, responsible for clinical failures of restorations [7]. One of the simple methods to measure the tensile strength of brittle material directly is diametral tensile strength test, adopted by the British Standards Institution [8]. The compressive force is applied to the cylindrical specimen across the diameter by compression plates. The maximum load peak is measured right before the catastrophic failure of the specimen occurs. Such stresses occur in the cement during mastication or parafunctional loads applied to the prosthesis.

As for the filling medium between the restoration and the abutment, the chipping of the cement at the margin can be reduced with improvement of the fracture toughness of the cement as a better fracture resistance assessment than other strength parameters [9-11]. Especially at the marginal region surface flaw and film thickness [4] of the cement are very important due to presence of notches at the region. For brittle materials, such as cements, fracture toughness is an important factor, as it means the amount of force or energy required to propagate a surface flaw or pre-existing crack through a material [12].

In case of cement failure at marginal region, the longevity of the restoration is questionable. Nowadays many kinds of resin cements are introduced to the clinical practice. However, there are not many recent studies about diametral tensile strength and fracture toughness of resin cements for zirconia restoration.

The purpose of this study was to compare diametral tensile strength and fracture toughness of four dental resin cements used for zirconia restorations.

II. MATERIALS AND METHODS

Four dual-cured resin cements were investigated in this study (Table 1). For the experiment of diametral tensile strength a special cylindrical stainless steel mold was used. The mold consisted of two parts, to avoid application of any force to the specimen while removal from the mold after the cement was set.

Zirconite was injected into the mold with the automixing gun applicator, Clearfill SA luting cement, Rely-X Unicem, and Panavia F2.0 were mixed with a clean plastic spatula for 30 seconds and placed immediately into the mold. To avoid adhering of the samples to the mold, the separating medium was used. After the material was injected in the mold, the two glasses were firmly clamped with hand pressure against each side of the mold to prevent inhibition of polymerization by oxygen and creation of the flat-end surfaces of the specimen [13,14]. After setting of the material, the mold was disassembled. The specimen was gently removed from the mold and additionally light-cured for 5 seconds at every side of the specimen with the Blue LED-curing light (LD-105, Monitex Industrial Co., Ltd. Taiwan). For each material ten cylindrical specimens (4.0 ± 0.1 mm in diameter and 6.0 ± 0.1 mm in height) were fabricated according to specification No.27 of ANSI/ADA [12,15-17]. All specimens were stored in air at 37^{o}C for 24 hours.

To measure diametral tensile strength of the specimens a diametral compression test (indirect tensile test) was used. In the test a specimen was compressed diametrically to failure. The diametral compression test was conducted using an Instron universal testing machine (Instron Corp., Ganton, MA, USA). The specimens were loaded at a crosshead speed of 0.5 mm/min and the failure load was recorded at 23 ± 1^{o}C [9,15-17]. The flats ends of the specimens were perpendicular to the platens of the testing machine so that load was applied to the diameter of the specimens. The maximum load applied until failure of the specimens was recorded.

The diametral tensile strength was calculated from the following equation.

$$\boldsymbol{\sigma = \frac{2P}{\pi DT}}$$

where σ is the diametral tensile strength (MPa)

P = the maximum fracture load (N)

D = the diameter (mm) of the specimen

T = the length (mm) of the specimen.

For fracture toughness (K_{IC}) test the specimens had a size of 20 × 6 × 4 mm [18]. As a special characteristic of the specimen was a single-edge notch sample design. The mold contained a razor blade in its middle what produced a sharp notch. The mixed cement was placed in a mold, made in Teflon which was coated with separating medium to avoid adhering to the mold.

After the placement in the mold the material was light cured for 30 seconds on every end side and in the middle of the specimen. When the material had set in the mold, the sample was carefully removed and additionally light-cured for 5 seconds at every side of the specimen with the Blue LED-curing light (LD-105, Monitex Industrial Co., Ltd. Taiwan).

All specimens were stored in air at 37^{o}C for 24 hours. The fracture toughness (K_{IC}) test was conducted using an Instron universal testing machine (Instron Corp., Ganton, MA, USA). The specimens were loaded at a crosshead speed of 0.5 mm/min and the failure load was recorded at 23 ± 1^{o}C [15-18, 21].

The fracture toughness was determined from the ASTM standard equation (American Society for Testing and Materials, 1983):

$$K_{IC} = (3PL/BW^{3/2})Y$$

where P = peak load at fracture;

L = length;

B = width;

W= height;

and Y= calibration functions for given geometry,

$(1.93[a/W]^{1/2} – 3.07[a/W]^{3/2} + 14.53[a/W]^{5/2} –25.11[a/W]^{7/2} + 25.80[a/W]^{9/2})$

After the fracture toughness test was completed, the surfaces of specimen of each kind of cement lying right over the sharp notch and below the crosshead of testing machine were analyzed with scanning electron microscope.

For all the data obtained in the this study, the mean values and standard deviations were computed. Statistical analysis of the data was performed with SPSS (PASW Statistics 17.0 IBM Acquires SPSS Inc., Chicago, IL, USA) using one-way ANOVA and multiple comparison Scheffé test at the significance level of 0.05.

III. RESULTS

Among investigated cements the diametral tensile strength found to be the highest in Panavia F2.0 (37.77 ± 2.72 MPa) followed by Rely-X Unicem (31.98 ± 2.92 MPa) and Zirconite (30.43 ± 2.06 MPa) (Fig. 1). However, diametral tensile strength was not found to be significantly different between Zirconite and Clearfill SA luting (26.37 ± 6.75 MPa).

The mean fracture toughness data and standard deviations for investigated materials are presented in Fig. 2 with statistical analysis described as superscripts. Panavia F2.0 (1.52 ± 0.16 $MNm^{-1.5}$) had the highest fracture toughness, when for Zirconite (1.18 ± 0.16 $MNm^{-1.5}$), Clearfill SA luting (1.18 ± 0.1 $MNm^{-1.5}$), and Rely-X Unicem (1.18 ± 0.06 $MNm^{-1.5}$) fracture toughness was found similar, but significantly lower than one for Panavia F2.0.

IV. DISCUSSION

Nowadays increased interest in esthetic results of dental restorations provoked heightened usage of CAD/CAM zirconia restorations by clinicians. However, the overall accuracy of CAD/CAM restoration is worse than that of conventional metal-ceramic restoration [1]. Multiple techniques, such as scanning, software, and machining are involved for production of CAD/CAM zirconia restoration [2]. Thus, increased gap between the abutment and restoration should be filled with resin cement. Accordingly, the thickness of the cement film will increase, resulting in heightened stress escalation in the cement compare to materials beneath conventional restorations produced by wax lost technique. In the aim to withstand these stresses, occurred from masticational loads or parafunctional habits, the resin cements should have improved mechanical characteristics than conventional luting cements. These aims can be achieved by using dual-cured bonding cements. Such cements provide equitable stress distribution, less probability of tensile or compressive failure, and greater clinical success [14].

Loads that stretch or elongate the specimen cause tensile stresses [19]. The determinant factors for the tensile strength are the filler type and content with monomer type [20]. However, until now there are not many studies to evaluate important physical properties of resin cements used for zirconia restorations. Hence, the main purpose of this study was to compare diametral tensile strength and fracture toughness (K_{IC}) of four dental resin cements used for zirconia restorations.

For the dual-cured bonding cements the diametral tensile strength reaches its maximal value at 24 hours without significant difference between 24 hours and 7 days test measurements [5]. For this reason, all the specimens were stored for only 24 hours.

The uptake of water or solvent by material may cause expansion of the specimen [21]. Also, the storage of cured resin material in the water at 37^{o}C has a tendency to lower the strength of the specimens [16]. On the other hand, after seating of the restoration on the abutment, saliva or other fluids are not in direct contact with the cement, except marginal part of restoration. According to above mentioned, in this experiment the specimens were stored in air environment at 37^{o}C to make the conditions of the resin cement to be close to *in vivo*.

After all the specimens were produced and stored in the conditions described above, the tests for diametral tensile strength and fracture toughness were performed.

For brittle materials, such as dental resin cements, a diametral compression test is a common method to measure the diametral tensile strength [15]. The test is simple and gives information about internal coherence of brittle polymer-based material [22]. Therefore, the comparison of materials by the diametral compression test is a useful method of comparative evaluation of different materials [23].

Among investigated resin dual-cured cements, the value of diametral tensile strength was found to be highest in Panavia F2.0 (37.77 ± 2.72 MPa). At the same time, Clearfill SA luting (26.37 ± 6.75 MPa) showed the lowest values. As described in the other studies, the diametral tensile strength values for resin cements are in mean value range between 33.6 MPa to 46.9 MPa [5] and between 29.4 MPa to 45.1 MPa [6].

The variation in values in diametral tensile strength can be explained by impurities in the reactant [24], the difference between polymeric matrix, size of fillers and bond between fillers and matrix [20]. However, Clearfill SA luting cement had the greatest standard deviation. This may give us the clue, that even when the conditions of producing the specimens were the same, in some part, the specimens were different.

The next parameter, chosen in this experiment for evaluation of the resin dual-cured cements was fracture toughness. Different resin cements contain different amount of fillers. According to previous studies, increase in filler content improves the fracture toughness [18,22]. Each of investigated cement had a different chemical composition. However, in Panavia F2.0 the total amount of inorganic filler is 59%. In Zirconite and Rely-X Unicem the total amount of inorganic filler is 50%, and in Clearfill SA Luting cement this amount is only 44%.

The value of K_{IC} defines the critical stress intensity level at which a catastrophic failure occur [9]. For the resin cement, the K_{IC} value is important especially at the cusp region and margin of the restoration. The bite force applied at the cusp of the restoration redirects the load to the cement, as supporting medium between the restoration and the abutment. In case of low fracture toughness, the crack will occur, and the longevity of the restoration will be affected. This is reasonable especially for ceramic inlays or onlays, because the durability of such restoration is less than durability of full crown restoration, what encircles the abutment. Also, the ferrule effect present in full crown prosthesis restoration is not present in other restorations, such as 3/4 crowns, inlays or onlays. In such restorations the ability to withstand the loads occurred during mastications or parafunctional movements depends not only on resistance form of the cavity, but on the physical or mechanical properties of bonding cement. However, the study about fracture toughness for dual-cured resin cements was not found in the literature.

In the aim to prove the value of the experiment the SEM pictures of the surfaces of the specimens of each kind of investigated cement were taken and analyzed. The fracture line of the specimen lies right over the sharp notch, dividing the area into two parts: the first one (lower part in the micrograph) is relatively rough while the other (the upper part) is smoother, as it is seen in Fig. 5a-d. For three samples the crack propagating along the boundary between smooth and rough areas is observed. Another particularity observed in micrographs taken at high magnifications (Fig. 6a-b). The particles of inorganic filling poor adhere (poor encapsulated in the organic matrix) to the organic component of the paste for Panavia F2.0, while the best adhesion of inorganic filling to the organic component (best encapsulation) is observing for Rely-X Unicem. This conclusion follows from the fact that inorganic particles in Panavia F2.0 (Fig. 6a) are not as wetted with organic component as for Rely-X Unicem, (Fig. 6b). The other two samples demonstrate intermediate adhesion strength of inorganic filler to the inorganic component (Figs. 6c, d). The explanation of the above observations in terms of physics and materials science may shed a light on the comparative properties of the investigated cements.

This effect together with the crack that propagates along the boundary between rough and smooth surfaces for Panavia F2.0 points at its more brittle nature. Actually, since the inorganic particles are chipping out of the organic matrix and deep crack forms within the Panavia F2.0 cement as it follows from Fig. 6a, it points to its more brittle nature compare to other cements under investigation. Mechanical test of the cements supports microscopic observations. Indeed, brittle materials possess higher hardness and just this effect we see for Panavia F2.0 which possesses maximal hardness (tensile stress and thus maximal hardness) among other cements under investigation. Relative hardness of the cements can be evaluated based on their tensile stress, which is as follows:

Panavia F2.0 (37.77 ± 2.72 MPa), Rely-X Unicem (31.98 ± 2.92 MPa), Zirconite (30.43 ± 2.06 MPa), Clearfill SA luting (26.37 ± 6.75 MPa).

In this experiment, value of fracture toughness for Panavia F2.0 (1.52 ± 0.16 $MNm^{-1.5}$) was the highest. However, for the rest of the tested cements the fracture toughness was found to be similar.

From the results of the tests, we can conclude, that the resin cement with a high diametral tensile strength has the high fracture toughness. On the other hand, the cement with lowest diametral tensile strength had similar fracture toughness with other cements. This study provides the valuable information for the clinicians using CAD/CAM zirconia restoration cemented with dual-cured resin materials.

In the experiment, the specimens with visible defects were excluded from the test; however, each specimen was not evaluated using the microscope after the test was performed. Thus, the small artifacts, such as minor air bubbles, or micro cracks could be present in some samples. In addition, the results were obtained *in vitro*, however, *in vivo* conditions the situation might be completely different due to difficulties of moisture control, temperature changes during first hours of setting, accurate mixture of the cement in clinical conditions, early application of the force on the restoration after placement, etc. All these factors may cause catastrophic failure of the restoration, what were not observed in this study. However, due to limitations of current study, such hypotheses were not tested. Therefore, these estimates could be taken in mind as the reasons for high standard deviation rate and, as a result, for the test outcomes. In the further experiments these factors should be taken in account and verified to provide more accurate data. Also, clinical experiments should be included as the subject of research in further studies for resin cements.

V. CONCLUSION

Among investigated dental resin cements the highest diametral tensile strength and fracture toughness were found for Panavia F2.0. In cementation of CAD/CAM zirconia restorations Panavia F2.0 is believed to withstand load forces better, than other investigated cements, demonstrating high resistance to fracture.

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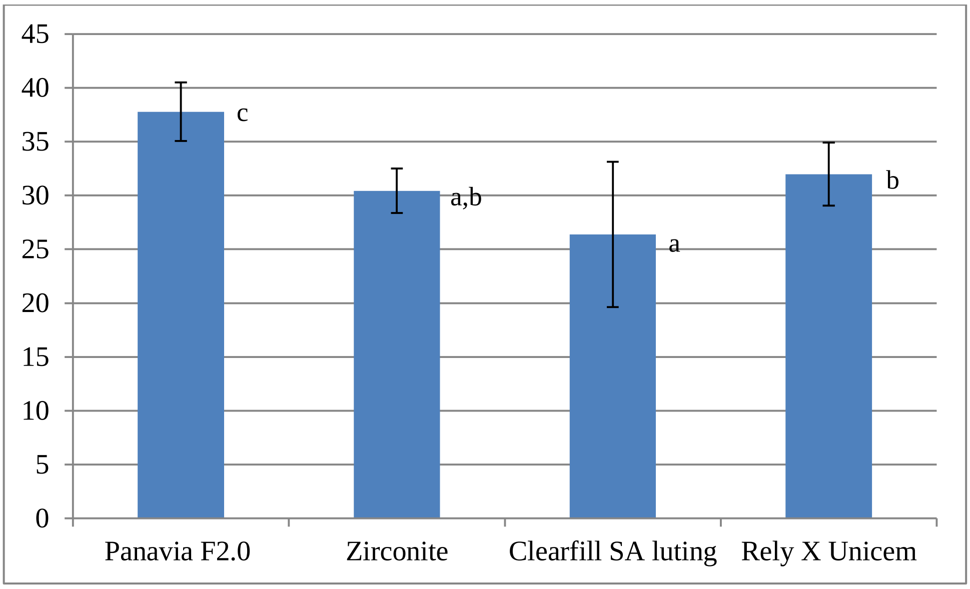
Table 1. Resin cements investigated in this study.
4-META: 4-Methacryloxyethyl trimelliticc anhydride

MDP: 10-methacyloxydecyl dihydrogen phosphate

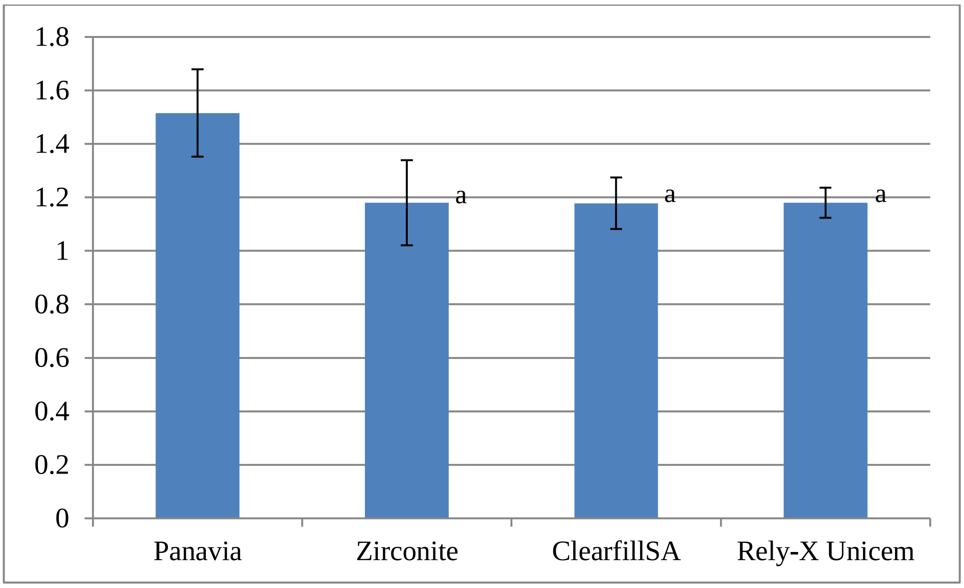
Brand name	**Lot No.**	**Composition**	**Manufacturer**
Panavia F2.0	00405A	MDP	Kuraray Medical Co., Osaka, Japan
Clearfill SA luting	143BA	MDP	Kuraray Medical Co., Osaka, Japan
Zirconite	4146HQBARCZ	4-META, Methacrylated Phsophric acid esters, [3-(Methacyloyloxy) propyl] trimethoxysilane	BJM Lab., Or Yehuda, Israel
Rely X Unicem	3922517	Phosphoric acid modified methacrylate monomer	3M ESPE, St Paul, MN, USA

Fig. 1. Mean diametral tensile strength (MPa) and standard deviations of materials investigated.



The same scripts demote no significant difference ($P < 0.05$).

Fig. 2. Fracture toughness ($MNm^{-1.5}$) and standard deviations of materials investigated.



The same scripts demote no significant difference ($P < 0.05$).

Fig. 3. Dimension of cylinder-shaped specimen. φ= 4 mm, L = 6 mm, P= load.

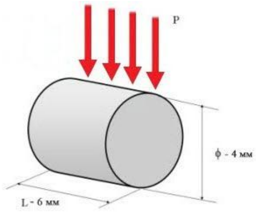
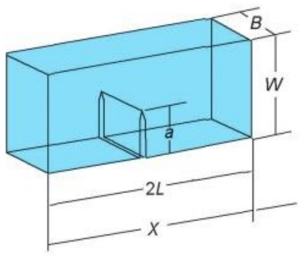


Fig. 4. Single-edged, notched specimen used in the study; B = 4 mm; W = 6 mm; X = 20 mm; L = 10 mm; a = 4 mm.



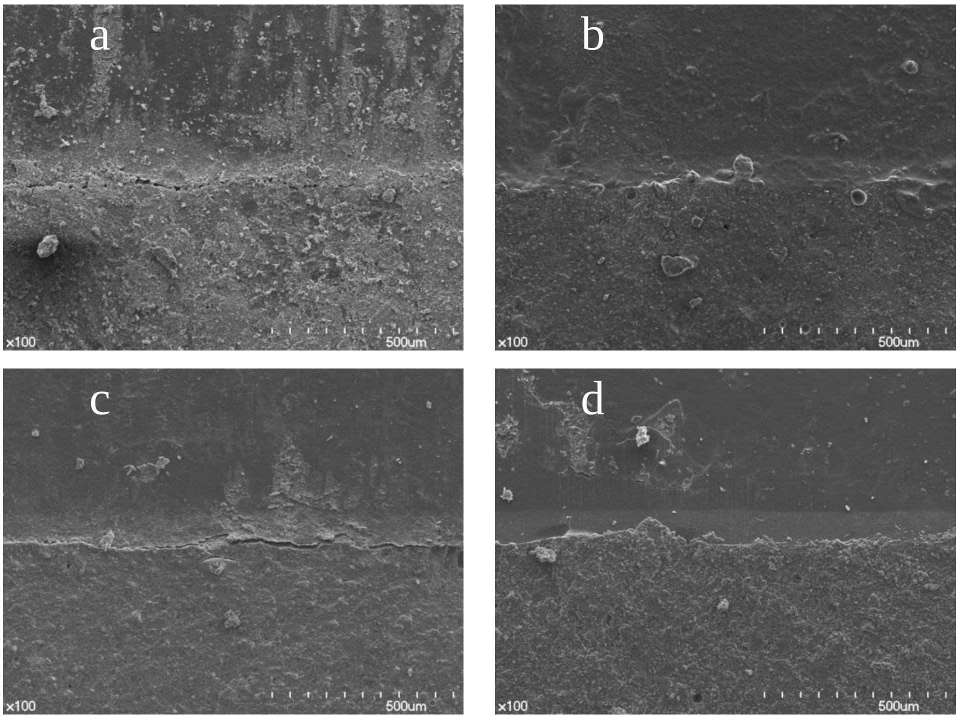


Fig. 5. SEM pictures of microstructure of fractures of the specimens with × 100 magnification. The difference between microstructures of upper and lower parts of the samples is clearly seen in all specimens: a) Panavia F2.0, b) Rely-X Unicem, c) Clearfill SA luting, d) Zirconite.

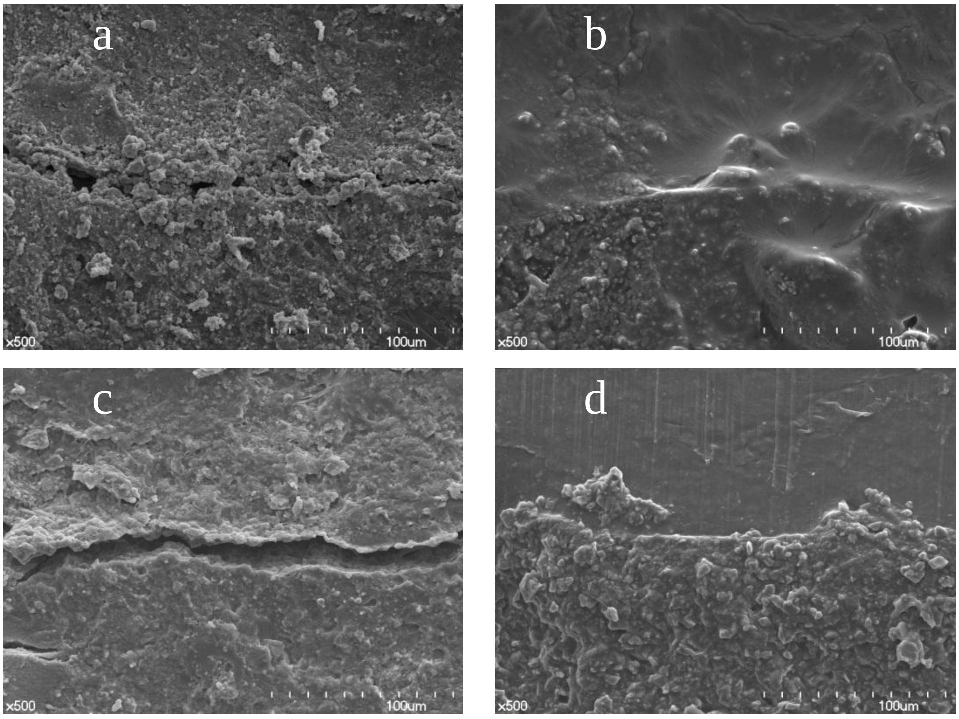


Fig. 6. SEM pictures of microstructure of fractures of the specimens at high (× 500) magnification that demonstrates the strength of adhesion of inorganic filler to the organic component in the cement: a) Panavia F2.0, b) Rely-X Unicem, c) Clearfill SA luting, d) Zirconite.

국문초록

지르코니아 수복물에 사용하는 치과용 레진 시멘트간의 간접인장강도와 파괴인성의 비교

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연구목적

이 연구의 목적은 지르코니아 수복물의 합착에 사용되는 네 가지 레진 시멘트 간의 간접인장강도와 파괴인성을 비교하는 것이다.

재료 및 연구방법

4개의 레진 시멘트 (Panavia F2.0, Zirconite, Clearfill SA luting, Rely-X Unicem) 선택하였고 간접인장강도 시험을 위해 직경 4 mm, 길이 6 mm의 원통형 스테인레스 스틸 주형을, 파괴인성 시험을 위해 20 x 6 x 4 mm의 single-edge notch가 있는 테플론 주형을 사용하였다. 모든 시편은 24 시간 동안 $37^{o}C$에서 공기 중에 보관하였다. 인스트론 만능재료시험기(Instron Corp., Ganton, MA, USA)를 사용하여 crosshead speed를 0.5 mm/min로 실패 하중을 $23 \pm 1^{O}C$에서 기록하였다. 통계 분석은 SPSS를 사용하여 유의수준 0.05로 one-way ANOVA 와 Scheffé의 다중검증을 시행하였다.

결과

Panavia F2.0(37.77 MPa ± 2.72 MPa)군은 가장 높은 간접인장강도를 보였으며 뒤이어 Rely-X Unicem(31.98 MPa ± 2.92 MPa), Zirconite(30.43 MPa ± 2.06 MPa), Clearfill SA luting(26.37 MPa ± 6.75 MPa)의 순으로 나타났다. Rely-X Unicem 과 Zirconite, Zirconite와 Clearfill SA 간에는 통계적으로 유의한 차이가 없었다.

파괴인성 측정에서도 Panavia F2.0(1.52 $MNm^{-1.5}$ ± 0.16 $MNm^{-1.5}$) 은 가장 높은 결과를 나타내었으며, 나머지 세 그룹은, Zirconite(1.18 $MNm^{-1.5}$ ± 0.16 $MNm^{-1.5}$), Clearfill SA luting(1.18 $MNm^{-1.5}$ ± 0.1 $MNm^{-1.5}$), Rely-X Unicem(1.18 $MNm^{-1.5}$ ± 0.06 $MNm^{-1.5}$), 비슷한 파괴인성을 나타내었으며 Panavia F2.0보다 통계적으로 유의하게 낮은 값을 가졌다.

결론

연구한 레진 시멘트 중에서 Panavia F 2.0이 가장 높은 간접인장강도와 파괴인성을 보

였다. CAD/CAM 지르코니아로 제작한 수복물을 접착할 때 실험에 사용된 다른 레진 시멘트들 보다 Panavia F 2.0이 높은 파절 저항성을 보여 가해지는 힘에 가장 잘 저항하는 것으로 보인다.

주요어: Diametral tensile strength, Fracture toughness, Resin cements, Zirconia.

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