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Original Article

Effectiveness of Self-adhesive Resin Cements for Splinting Traumatized Deciduous Teeth

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The purpose of this study was to compare bending strength and shear bond strength to deciduous enamel of self-adhesive resin cements with conventional etch-and-rinse resin cements. Three self-adhesive resin cements (RelyXUnicem (RE), Clearfil SA Luting (CL), G-Luting (GL)) were tested. A conventional resin cement (Super-Bond C&B (SB)) was used for comparison. Bending strength of the resin cements was measured according to ISO4049. Nine rectangular specimens for each cement were immersed in deionized water at37℃ for 24 hours. They were subjected to three-point bending using a universal tensing machine at a crosshead speed of 0.75 mm/min. The shear bond strength of the resin cements to deciduous enamel was measured according to ISO/TS11405. The labial surface of deciduous teeth was wet ground with silicon carbide abrasive paper up to 600grits, and the resin cements were applied to the enamel surface without pressing. RE, CL, and GL were light-cured. SB was applied after acid etching.After being completely cured, the bonding specimens were immersed in deionized water at 37℃ for 24 hours, and the shear bond strengthwas measured at a crosshead speed of 0.5 mm/min. The mean bending strength values of RE, CL, and GL were significantly greater than that of SB. The mean values of bending strength for RE and GL were significantly greater than that of CL; however there was no significant difference between RE and GL. The mean values with SD (Standard Deviation) of shear bond strengths of GL and SB were 5.2±3.8 MPa and 6.3±2.1 MPa, respectively. However, there was no significant difference between these values. The mean shear bond strength of SB was significantly higher than those of CL and RE. The mean value of the shear bond strength of GL was significantly higher than that of RE.Failure mode analysis demonstrated that all specimens of SB failed at cohesive failures in cement layer, and GL bonded specimens failed either at cohesive failures or at mixed mode failures. However, in CL and RE bonded specimens, the occurrence of cohesive failures in cement layer was not observed. The results of this study suggest that GL, one of the self-adhesive resin cements, is effective as a splinting material for fixation of luxated deciduous teeth.

Key words: self-adhesive resin cements, bond strength, splinting, deciduous teeth, traumatic injury

Introduction

Traumatic injuries to deciduous teeth are commonly encountered in clinical practice. According to an epidemiological survey in Japan[1], the incidence of tooth trauma is highest among children aged 1–3 years. The most common types of trauma were loosening and concussion, followed by luxation. These types of traumatisms accounted for almost 65% of total injuries of deciduous teeth, many of which require fixation. Methods of deciduous tooth fixation are as follows: resin fixation; wire and resin fixation; bracket and wire fixation; and resin splint. Of these methods, wire and resin fixation is the most commonly used[1].

Major causes of fixation failures in the wire and resin fixation method are moisture contamination and inadequate drying of the tooth surface, which is often exacerbated by the child's unwillingness to cooperate during treatment. The lengthy curing period of at least five minutes required for chemically cured resin cements also make fixation operations more difficult. Furthermore, when conventional etch-and-rinse resin cements or resin composites are used, teeth require surface treatments such as etching and/or bonding.These processes are often time-consuming, and make fixation operations technique-sensitive. If the surface treatment is insufficient, the bond to the enamel will be impaired. From the patient management and safety perspective, the fixation of traumatized teeth in younger children should ideally be performed both quickly and securely. Therefore, development of fixation materials that require single step application been eagerly anticipated.

Self-adhesive resin cements have been recently developed to deal with the problem of conventional resin cements requiring time-consuming surface treatment. Self-etching resin cements do not require any surface treatment of the teeth and restorations. However, two opposite results have been reported about the bonding strength of the self-adhesive resin cements to the enamel of permanent teeth. De Muncket al[2] demonstrated that the microtensile bond strength of a self-adhesive resin cement (Re-

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lyXUnicem) to the enamel was significantly lower than that of the conventional resin cement (Panavia F). On the other hand, Hikita, et al[3] concluded that etch-and-rinse resin cements and self-etching resin cements are equally effective in bonding to the enamel if the correct application procedure is followed. Moreover, there is little information on the bond strength of self-adhesive resin cements to deciduous enamel, so the effectiveness of self-adhesive resin cements in the fixation of traumatized deciduous teeth is not clear. The purpose of the present study was to assess the effectiveness of three self-adhesive resin cements in the fixation of traumatized deciduous teethby comparing the bending strength and shear bond strength with a conventional etch-and-rinse resin cement.

Materials and Methods

Three self-adhesive resin cements (RelyXUnicem(RE); 3 M ESPE, Clearfil SA Luting (CL);Kurare Medical, G-Luting (GL);GC) were tested and compared with a conventional etch-and rinse resin cement (Super-Bond C&B (SB), Sun Medical). The application protocols are listed in Table 1.

To evaluate the bending strength, the three-point bending strength of rectangular specimens (2 mm x 2 mm x25 mm) was measured according to ISO4049:2000[4]. Rectangular specimens were prepared as follows: the three self-adhesive resin cements (RE, CL and GL) and one conventional resin cement (SB) were mixed according to the manufacturer's instructions, and the cements were put into a Teflon mold. RE, CL and GL were light cured using an LED light unit (Elipar S10, 3M ESPE). The SBwas allowed to self-cure. Nine specimens were prepared for each. After one hour, the prepared specimens were immersed in deionized water at 37℃ for 24hours, and they were subjected to the three-point bending test using a universal testing machine (EZ-Graph, Shimadzu) at a crosshead speed of 0.75mm/min.

Twenty-eight extracted human non-carious deciduous teeth were stored in 0.5% Chloramine T solution at 4 ℃ and used within three months of extraction. Table 2 shows the type of teeth used for measuring the shear bond strength of the cements to the enamel. The teeth were gathered following informed consent approved by the committee for Human Research Ethics of Asahi University (No. 22094), and were randomly divided into four groups of seven each.

Figure 1 shows the specimens for preparation of shear bond test. The teeth were embedded in chemically cured acrylic resin (SpeciFix 20, Struers). The labial surface of the enamel was wet ground with silicon-carbide abrasive papers up to 600 grit to obtain a 3 mm in diameter flat labial enamel surface. The cements were filled without pressing in a tygon tube (3355-L) with an internal diameter of 2 mm and a height of 3 mm, and RE, CL, and GL were light-cured. SB was filled after acid etching for 30seconds according to the manufacturer's instruction. For standardization, the bonding sites were demarcated by attaching a piece of masking tape with a 1.8mm diameter hole on each surface. After one hour, the specimens of shear bond strength were immersed in deionized water at 37℃ for 24 hours and the shear bond strength to deciduous enamel was measured according to ISO/TS11405:2003[5] using by the universal testing machine at a crosshead speed of 0.5 mm/min.

Table 1 List of cements used in this study

Product/Lot no./ Manufacturer	Code	Application/Surface Treatment of enamel
Self-adhesive resin cement		
Clearfil SA luting/ 011296, 011333/ Kuraray Medical	CL	Hand mixing for 10 seconds. Apply light-cure for 20 seconds per part. Rinsed for 15 seconds and air-dried for 10 seconds with a three-way syringe.
Rely X Unicem/56838/ 3M ESPE	RE	Hand mixing for 20 seconds. Apply light-cure for 20 seconds per part. Rinsed for 15 seconds and air-dried for 10 seconds with a three-way syringe.
G-luting/1001291/GC	GL	Hand mixing for 10 seconds. Apply light-cure for 10 seconds per each part. Rinsed for 15 seconds and air-dried for 10 seconds with a three way syringe.
Self-curing adhesive resin cement		
Super-Bond C&B/VK1/ SUN MEDICAL	SB	Mixed powder and liquid with disposable tip. Allowed to self-cure. Etchant was applied to enamel surface for 30 seconds, then rinsed for 15 seconds, and air-dried for 10 seconds with a three way syringe.

Table 2 Numbers of deciduous teeth used in this study

51, 61	71, 81	52, 62	72, 82	53, 63	73, 83
4	10	4	6	2	2

The names of individual teeth are abbreviated by Viohl's Two-digit System.

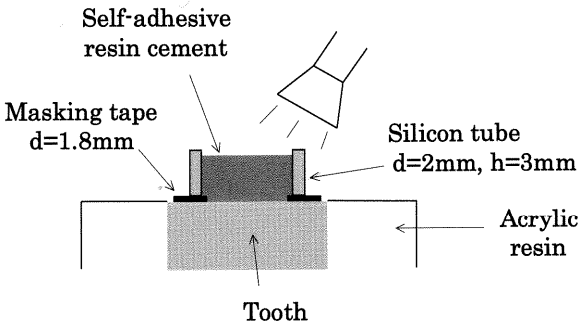

Fig. 1 Schematic illustration of shear bond strength test sample.

Following shear bond testing, all fracture surfaces were observed under a digital microscope (VHS-200, Keyence) at 100x magnification to determine the mode of failureand classified into one of three categories: adhesive failure at the cement-enamel interface, cohesive failure in the cement layer and mixed adhesive failure and cohesive failure, respectively. After sputter-coating with osmium coater (HPC-ISW, Shinkuu Devices), the fracture surfaces were also observed using a scanning electron microscope (SEM: S-4500, Hitachi) with an acceleration voltage of 5 kV.

The results ofthe three-point bending strengths and the shear bond strengths were analyzed at a significance level of 0.05 using one-way analysis of variance (ANOVA) and Fisher's multiple comparisons test. All statistics were performed using a statistical software package (StatView, J 5.0, SAS Inc).

Results

Figure 2 shows the mean bending strength values of the resin cements immersed in distilled water at 37℃ for 24 hours. The mean values of bending strength for three self-adhesive resin cements were significantly higher than that of SB. The mean values of RE and GL were significantly greater than that of CL, however, there was no significant difference between RE and GL.

The means of shear bond strength to deciduous enamel are shown in Table 3. The mean values with SD for GL and SB were 5.2 ± 3.8MPa and 6.3 ± 2.1MPa, respectively. However, there was no significant difference between these values. The mean shear bond strength of SB was significantly higher than those of CL and RE. The mean shear bond strength of GL was significantly higher than that of RE.

Table 4 shows the mode of failure of the resin cements bonded to deciduous enamel after shear bond testing. Failure mode analysis demonstrated that all specimens of SB failed due to cohesive failures in the cement layer, and GL bonded specimens failed either due to cohesive failures or mixed mode failures. However, in CL and RE bonded specimens, the occurrence of cohesive failures in the cement layer was not observed.

Figure 3 shows the SEM photographs of the fracture surfaces of the GL(a) and SB(b) bonded specimens. The

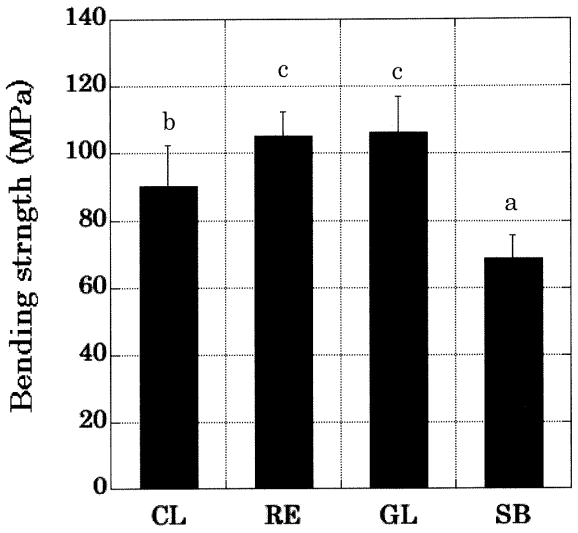

Fig. 2 Three-point bending strength of the cements immersed in distilled water for 24hours at 37℃. Letters marked with same superscript letter are not significantly different ($p<0.05$). ($n=9$)

Table 3 Shear bond strength of the resin cements to deciduous teeth. Letters marked with same superscript letter are significantly different ($p<0.05$). ($n=7$)

	Mean (n = 7)	SD	*
CL	3.0	2.0	b
RE	2.2	1.5	a, c
GL	5.2	3.8	c
SB	6.3	2.1	a, b

Values are mean and standard deviation (SD) in MPa. * Letters marked with the same superscript letter are significantly different ($p < 0.05$).

Table 4 Failure mode distribution of the shear bond strength test

	Adhesive failure at cement-enamel interface	Mixed failure	Cohesive failure in cement
CL	4	3	0
RE	3	4	0
GL	1	4	2
SB	0	0	7

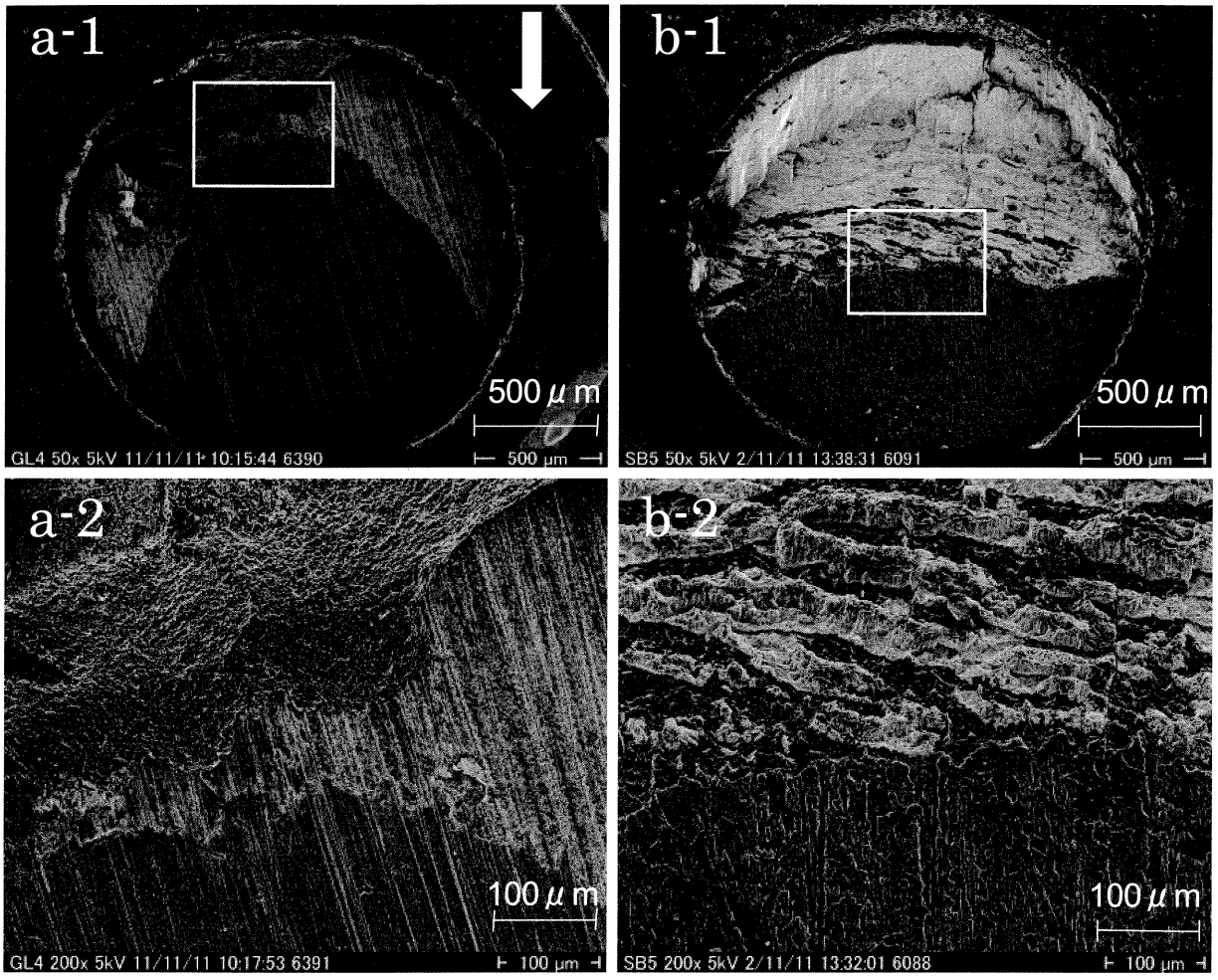

Fig. 3 SEM images of the fractured surface of GL (a) and SB (b) after shear bond strength test. The direction of loading for shear bond strength test was upper side to lower side of specimen (arrowed). Mixed failure is apparent (a-1, b-1).
At higher magnification, remnants of adhesive resin cement are evident on the enamel of adhesive failure area in SB (b-2). Many numbers of oblique lines which were caused by grounding process of specimen are evident in GL (a-2).

white arrow shows the loading direction. For GL bonded specimens, it is clear that the failure starts in the cement layer and propagates through the interface between the enamel and the cement, because many oblique lines due to grinding are observed in the SEM photographs. On the other hand, for SB bonded specimens, it was found that the interface between the enamel and the cement was covered by the cement. The SEM observation clearly shows that for SB bonded specimens, all of the fracture patterns were found cohesively in the cement layer.

Discussion

The effectiveness of self-adhesive resin cements as a splinting material for traumatized deciduous teeth was investigated in this study. At first, the three-point bending strength of three self-adhesive resin cements was evaluated, because the strength of the splint materials themselves are considered to have some influence on effectiveness of fixation. The strength results after 24hours were compared with the conventional etch-and-rinse resin cement. All self-adhesive resin cements had greater bending-strength than the conventional etch-and-rinse resin cement (SB). This finding shows that from the viewpoint of bending strength, all self-adhesive resin cements are considered to be effective in the fixation of traumatized deciduous teeth.

In the present study, the mean values of the shear bond strength of GL and SB were 5.2MPa and 6.3MPa, respectively. However, there was no significant difference between these values. The mean shear bond strength of SB was significantly higher than those of CL and RE. The results of the shear bond strength proved to be considerably lower than the values of previous studies that measured the shear bond strengthto the human molar enamel[8,9]. One of the reasons for the low bond strength is that the self-adhesive resin cements were applied to ground enamel without pressing, because it is difficult to apply a splint material to traumatized deciduous teeth with pressing. Chieffiet al[7] showed that the application of sustained seating pressure during luting procedures improves the final bond strength of the resin cement. Although it is not known how much bond strength is required to prevent fixation failure, the shear bond strength of cements used on orthodontic brackets will serve as a good index. It has been reported

that the clinically acceptable bond strength between brackets and the enamel is 6-8 MPa[10]. In the present study, the shear bond strength of the GL was 5. 2MPa. After the fixation of traumatized teeth, most dentists usually instruct the guardian to refrain from giving the child hard food to allow healing of the affected area.Therefore, the bond strength of 5. 2MPa is likely to be sufficient forsplinting a traumatized deciduous tooth.

Previous studies on the shear bond strengths and etching patterns of self-adhesive resin cements to permanent incisor enamel[11] suggested that the pretreated tooth surface would improve the bond strength of self-adhesive resin cements. Abdelnady[12] showed the difference inthe enamel bond strength according to different surface treatments. The pretreatment of the enamel surface with phosphoric acid will enhance the bond strength of self-adhesive resin cement to deciduous teeth. However, this study showed that there was no significant difference in the shear bond strength between SB and GL. GL consists of methacrylic acid ester, fluoro-amino-silicate glass, a phosphate monomer, and silica filler.SB, on the other hand, consists of MMA, 4-META, and TBB.GL contains a phosphate monomer,MDP. The functional monomer may serve various functions, including etching tooth substrates and enhancing monomer penetration[13]. However, the effect of the etching of the phosphate monomer on the bonding between the cement and the enamel may not be sufficient. As shown in Figure 3 , for GL bonded specimens, it is clear that the failure starts in the cement layer and propagates through the interface between the enamel and the cement, because many oblique lines caused by grinding are observed.These findings suggest that GL does not affect a structural change on the enamel and removing GL after the fixation period. On the other hand, the pretreatment of the enamel surface with phosphoric acid will enhance the bond strength of the conventional etch-and-rinse resin cement (SB) to deciduous teeth. As the etching procedure was performed before cementing, resin cement infiltrated into pits of the enamel surface of the SB group. However, the flexural strength of the etch-and-rinse resin cement was inferior to those of all self-adhesive resin cements. Therefore, as shown in the SEM photograph, for SB bonded specimens, all of the fractures patterns were observed cohesively in the cement layer.

The fixation of a traumatized tooth using the conventional adhesive resin cement requires 30 seconds of phosphoric acid etching followed by 5 to 10 seconds of rinsing with water. Moreover, it takes more than 5 minutes until the resin completely cures.In contrast, GL does not require any surface treatment and it takes only 10 seconds for light-cure of each part. Even when cementing four anterior teeth, the curing process can be completed within one minute when curing a single surface from two directions for10 seconds each. The application of GL for the fixation of traumatized teeth can dramatically reduce chair time and minimize patient burden compared to an application of the conventional etch-and-rinse resin cement. GL does not require any pretreatment, meaning that the treatment time will be reduced and enamel demineralization can be limited to the minimum area, thereby preventing the unnecessary invasion of intact enamel[14]. The measurement of bond strength in this study was performed on the flat area of the enamel. It is highly likely that the prismless outer layer enamel, which is more common in deciduous teeth than permanent teeth[15,16], was removed during the grinding process. Further research is now needed to determine the microstructural changes at the cement /enamel interface.

Conclusion

The bending strengths of the self-adhesive resin cements were superior to that of Super-Bond C&B.There was no significant difference in shear bond strength between G-Luting and Super-Bond C&B. These results suggest that G-Luting would be useful as a splinting material for fixation of luxated deciduous teeth.

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セルフアドヒーシブレジンセメントの外傷乳歯固定材としての有効性

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本研究の目的は，セルフアドヒーシブレジンセメントの曲げ強さ，ならびに乳歯エナメル質との接着強さを，従来型の接着性レジンセメントのそれと比較検討することにある．3種類のセルフアドヒーシブレジンセメント，リライエックス　ユニセム（RE），クリアフィル　SA ルティング（CL），ジールティング（GL）を使用した．従来型の接着性レジンセメントであるスーパーボンド C&B（SB）は，コントロールとして用いた．セメントの3点曲げ強さは，ISO4049に従い測定した．各セメントについて9個の角柱試料を作製し，37℃の蒸留水中に24時間浸漬した後，万能試験機によりクロスヘッドスピード0.75mm/min で，3点曲げ強さを測定した．セメントと乳歯エナメル質との接着強さは，ISO/TS11405に従い測定した．抜去したヒト乳歯頬側面を耐水研磨紙で600番まで研磨し，圧をかけないでセメントを歯面に接着させた．RE，CL，GL は，光照射して硬化させた．一方 SB は，歯面を酸処理した後に，歯面に載せ硬化させた．セメントが十分硬化してから，試料を37℃の水中に24時間浸漬し，万能試験機によりクロスヘッドスピード0.5mm/min でせん断接着強さを測定した．曲げ強さの平均値は，RE と GL が，CL と比較して有為に大きい値を示した．しかし，RE と GL 間には有意差は認められなかった．GL と SB のせん断接着強さの平均値ならびに標準偏差は，それぞれ5.2±3.8MPa，6.3±2.1MPa であったが，両者の間に有意差は認められなかった．SB のせん断接着強さの平均値は，CL，RE の平均値に比べ，有為に大きかった．GL のせん断接着強さの平均値は，RE の平均値に比べ著しく大きかった．破断様式は，SB がすべてセメント内での凝集破壊であったのに対し，GL は混合破壊と凝集破壊がみられた．しかし CL と RE では，セメント内での凝集破壊はみられなかった．これらの結果から，セルフアドヒーシブレジンセメントである GL の，外傷乳歯固定材料への応用の可能性が示唆された．

キーワード：セルフアドヒーシブレジンセメント，接着強さ，固定，乳歯，外傷

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