Original Article

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Comparison of bond strength between two resin cement systems used in modified tube technique for separated endodontic instruments removal

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Objective: The aim of this study was to compare bond strength between two resin cements including Super Bond Universal and RelyX U200 used for separated instrument removal with modified tube technique

Materials and Methods: Thirty ProTaper Next X1 NiTi rotary files were embedded into the fixing mold then cut at 5 mm from the instrument tip in order to simulate the separated instrument engaged in the root canal. The samples were divided into 2 experimental groups according to chemical agents for adhesion in tube and glue technique, Group 1: Super Bond Universal, and Group 2: RelyX U200 (n = 15/group). Resin cements were prepared and loaded into the hypodermic tube before insertion onto each separated instrument at 2 mm overlapping length. After the complete setting of the material, all samples were subjected to pull-out test at a constant speed of 2 mm/min. using the Universal Testing Machine. The failure load of each sample was recorded and the data was analyzed using Shapiro-Wilk Test and Levene's Test to confirm the normality and homogeneity of variance following by an independent samples t-test for group comparison.

Results: Mean failure loads of group 1 and group 2 were 1.62 N and 15.50 N. The bond strength of RelyX U200 was significantly higher than Super Bond Universal ($p<0.001$).

Conclusion: Due to the compositions affected the molecular structure of this resin cement together with convenient application, the bond strength of RelyX U200 is significantly superior over that of Super Bond Universal.

Keywords: bond strength, modified tube technique, resin cement, separated instrument removal

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Introduction

Separation of endodontic instruments can occur as a common complication during root canal treatment. The presence of separated instruments might impair effective root canal disinfection, thereby decrease the treatment's success rate [1-4]. Hence, retrieval of the separated instrument should be performed to accomplish the ultimate goal of cleaning and shaping the root canal. Dentists have to make a decision whether to retrieve or retain the separated instruments in the root canal system, considering the balance between risks and benefits. When the separated instrument removal tends to be possible, retrieving the fragment will obtain the great benefit [5-8].

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Nowadays, there are several techniques available for the removal of separated instruments. Modified tube technique or tube and glue technique is a method using the hypodermic tubes combined with chemical agents to remove separated instruments. The chemical agent must be applied to the tube before it is inserted over the cutting edge of the separated instrument. After allowing the material to set, the hypodermic tube, along with the separated instrument, is gently extracted. The chemical agent, which fills the gap between the hypodermic tube and the separated instrument, plays a crucial role in removing the instrument from the root canal [9-11].

The bond strength of the chemical agent is also a significant variable that contributes to the successful retrieval of separated instruments in tube and glue technique [10, 12]. Various types of materials are already used as chemical bonding agents, such as cyanoacrylate and flowable resin composite. Cyanoacrylate, which has wide availability and affordability, is commonly used for tube and glue technique. However, it has some drawbacks, such as low viscosity and high flow, possibly resulting in the extrusion of the chemical agent from the hypodermic tube and obstructing the root canal. Moreover, cyanoacrylate exhibits low tensile strength, leading to reduced efficiency of instrument removal [12-13]. Flowable resin composite has already been used in tube and glue technique as a glue aid since it has many superior properties over cyanoacrylate, including a lower incidence of bond failure and higher shear bond strength [14-15]. Nevertheless, there are studies demonstrated the failure of instrument retrieval when using flowable resin composite without adhesives or surface modification [15-16].

Nowadays, there are several resin cements in the fields of dentistry. Based on their polymerization mechanism, luting cements could be classified into three types: light-cure resin cement, dual-cure resin cement, and chemical-cure resin cement. However, light-cure resin cement may have some disadvantages for tube and glue technique because of the limited curing light accessed through the hypodermic tube. Dual-cure resin cement and chemical-cure resin cement, which possess self-curing ability, have a high potential for tube and glue technique [17].

Super Bond Universal is a resin cement that is widely used and has a self-curing process. It contains 4-META (4-Methacryloxyethyl trimellitate anhydride), which is accountable for bonding to metal surfaces. Super Bond Universal gains significantly high bond strength in comparison to other dental adhesives [17-18]. Another resin cement commonly used is RelyX U200, which is a dual-curing resin cement. This resin cement is remarkable in its convenient application as it is easy to dispense and deliver [19].

Resin cement may have good bond strength that could minimize the depth of bonded area and consequently reduce the loss of tooth structure. Moreover, no study has been conducted on the application of resin cements as chemical agents for tube and glue technique. Therefore, the purpose of this study was to compare the bond strength between two different resin cements (self-cure resin cement and dual-cure resin cement) for instrument removal using tube and glue technique.

Materials and Methods

Sample size calculation

The sample size for this study was determined using a power analysis based on an independent t-test and designed with a statistical power of 95% and a significance level of 0.05. Based on the pilot results, the variance in the mean failure load for both cement types was 1.09764 N and 15.14953 N. The sample size was

calculated by G*Power software Version 3.1.9.6 (Heinrich-Heine-Universität Düsseldorf, Düsseldorf, Germany). This analysis indicated a minimum sample size of '3' per group and the sample size for the experiment was set to 15 samples per group.

Experimental model preparation

For experimental model, thirty samples were prepared (n=15/group). The separated instrument was embedded into the fixing mold to simulate the separated instrument engaged to the root canal wall. The preparation of the sample was described as followed.

Separated instrument preparation

ProTaper Next X1 rotary file (Dentsply-Maillefer, Ballaigues, Switzerland) was measured by using a Digital Vernier caliper (CD-6'' CSX, Mitutoyo, Kanagawa, Japan) under the measuring microscope (Nikon MM-11: 5X magnification, Tokyo, Japan). Three reference levels were labeled on each instrument including cutting level, overlapping length level, and embedded levels (Figure 1). The cutting level was marked at 5 mm from the instrument tip to simulate the separated instrument. The embedded level, the fixing length of the instrument embedded into the fixing mold, was created at 2 mm from the instrument tip. Finally, the overlapping length level was marked 2 mm below the cutting level in order to simulate the insertion depth of the hypodermic tube. All of the measured reference levels were labeled by the extra fine tip permanent marker (Sharpie Ultra Fine Tip Black Marker Pen: line size 0.20 mm, Newell Office Brands, USA).

Table 1 Materials

Material	Composition	LOT number
Super Bond Universal	Super-Bond Catalyst V: Partially oxidized tri-n-butyl borane, others Super-Bond Quick Monomer: Methacrylate monomers, others Super-Bond Universal Polymer (Radiopaque): Poly methyl methacrylate, others	FF0397
RelyX U200	Base: Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, initiators, stabilizers, rheological additives Catalyst: Methacrylate monomers, alkaline fillers, silanated fillers, initiator componenets, stabilizers, pigments, rheological additives	9813459
ProTaper Next X1 Rotary File	Nickel-titanium alloy (M-Wire®)	1788769
Hypodermic Needle Gauge 21	Stainless steel	23 C 09
Ortho Jet Acrylic Powder	Methyl methacrylate, N, N-Dimethyl-p-Toludine	1380-21AW
Ortho Jet Acrylic Liquid	Polymer	1408-22AA

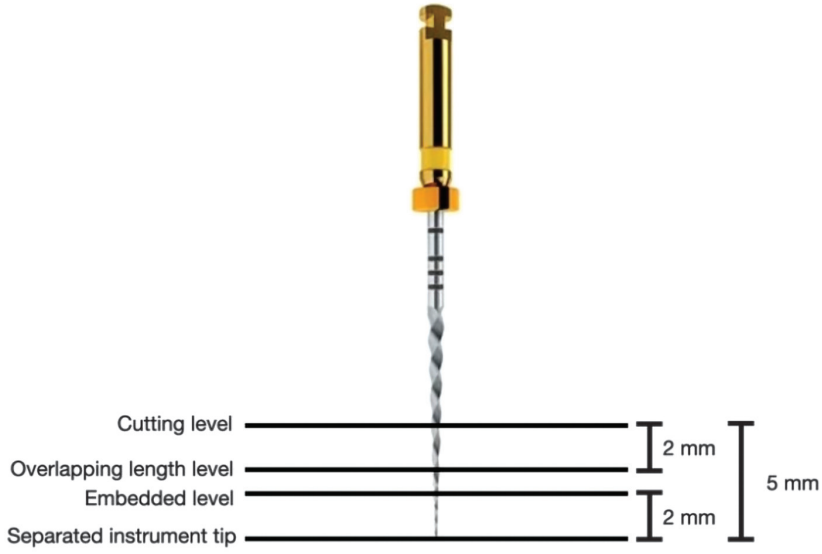


Figure 1 Labeled reference levels for each ProTaper Next Size X1

Fixation of the separated instrument into the fixing mold

Each separated instrument was fixed by the Vicat Apparatus over the PVC pipe for controlling the embedded length and angulation of the instrument that should be most parallel to the long axis of the Universal Testing Machine (Instron Ltd., Buckinghamshire, England). After that, orthodontic self-curing acrylic (Lang Dental Manufacturing. Co., Inc., Illinois, USA) was prepared following the manufacturing instructions. The mixture was poured into the Polyvinyl chloride (PVC) pipes. Each fixed separated instrument on the Vicat Apparatus was loaded immediately into unset acrylic until reach the embedded level as marked (Figure 2). After the setting period of acrylic, each fixed instrument in the fixing mold was cut at the cutting level by a single-sided diamond disc (Diamond Disc 915DF 104 190, Jota, Rüthi, Switzerland) under the measuring microscope (Nikon MM-11: 5X magnification, Tokyo, Japan).

Preparation and cementation of the hypodermic tube onto the separated instrument

Hypodermic tubes gauge twenty-one are chosen as the tube part. Measured and marked from the hub for twenty mm, the hypodermic tubes were cut by Diamond Disc 915DF bur and became non-beveled.

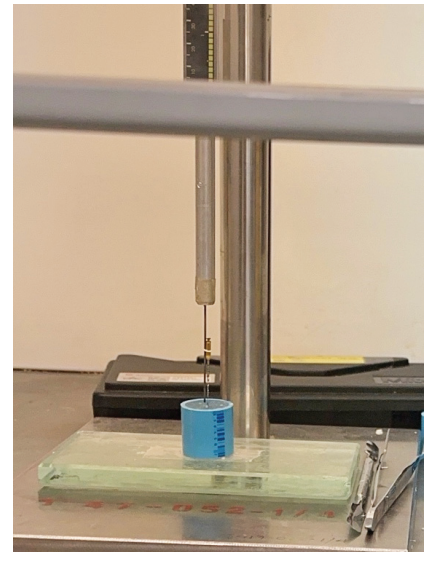


Figure 2 Setting of an embedded separated instrument with Vicat

Separated instruments embedded in the fixing molds were divided into 2 experimental groups (n=15/ group) based on the type of resin cements. In group 1, Super Bond Universal was applied while RelyX U200 was used in group 2. The overlapping length of hypodermic tubes was 2 mm in both groups

The mixing of each type of resin cement followed the manufacturer's directions. For Super Bond Universal, polymer powder was mixed

directly to the activated liquid by bulk-mix technique. The activated liquid was prepared on the chilled dispensing dish then the polymer powder was added and stirred lightly with the brush. The mixing ratio of monomer, catalyst V, and polymer was 4:1:1. All components were used immediately after taking out from the refrigerator to control the same mixing temperature. Prepared resin cement was placed on a mixing pad and was loaded into the hypodermic tube by scratching technique. The volume of resin cement added into the hypodermic tube was controlled by 30 times of scratching in every sample.

For RelyX U200, the material was dispensed and mixed thoroughly onto the mixing pad before use. The prepared resin cement was loaded into the hypodermic tube as previously mentioned for Super Bond Universal.

The hypodermic tube containing each resin cement was inserted onto each separated instrument embedded in the fixing mold until achieved the 2 mm overlapping length levels. All samples in the Super Bond Universal group were left for 12 minutes to confirm the complete setting of the materials. For RelyX U200 group, all samples were left for 6 minutes with light-curing (VALO, Ultradent Products Inc., Utah, USA) applied through the hub of hypodermic tubes.

Pull-out test by Universal Testing Machine

Each prepared sample was mounted onto the Universal Testing Machine. The hub of hypodermic tube was vertically grabbed by the grip of the testing machine. Pull-out tests were performed with a constant speed of 2 mm/min. Failure load results were measured in Newton digitally and recorded.

The dislodged resin cement was evaluated based on its mode of failure. Adhesive failure was identified when the resin cement detached from the hypodermic tube or the separated instrument. Cohesive failure was considered when the cement detached within its own layer. A mixed failure was noted when both adhesive and cohesive failures were observed. Five samples from each group were randomly selected to determine the failure mode.

Statistical analysis

The data was tested to compare the failure load of two resin cements. The Shapiro-Wilk test and the Levene's test were used to confirm the normality and equality of the variances. Since the data have normal distribution and homogeneity of variances, an independent sample t-test was performed. All collected data were analyzed by SPSS v.22 for MAC (SPSS Inc, Chicago. IL. USA) at $P<0.05$.

Results

In the pull-out test, the failure load between 2 groups of resin cement types was significantly different ($p<0.001$). The mean failure load of the Super Bond Universal group and RelyX U200 group were 1.62 N and 15.50 N. According to the statistical analysis, the pulling force to break the connection between hypodermic tubes and separated instruments in RelyX U200 group was more than Super Bond Universal group (Table 2).

The failure mode of each randomly selected sample was observed. In the Super Bond Universal group, adhesive failure was detected between the resin cement and the hypodermic tube in three of the five samples, while mixed failure was observed in two of the five samples. In the RelyX U200 group, four of the five samples showed adhesive failure between the resin cement and the hypodermic tube, while only one of the five samples exhibited mixed failure. Cohesive failure did not occur in any of the randomly selected samples.

Analysis of the failure modes revealed that adhesive failure between file and resin cement was the predominant pattern in both experimental groups (Figure 3).

Table 2 Failure load from pull-out test (n = 15/ group)

Groups	Cement types	Failure load (N) Mean ± SD	P-value
1	Super Bond Universal	1.62 ± 0.37	< 0.001
2	RelyX U200	15.50 ± 2.80	

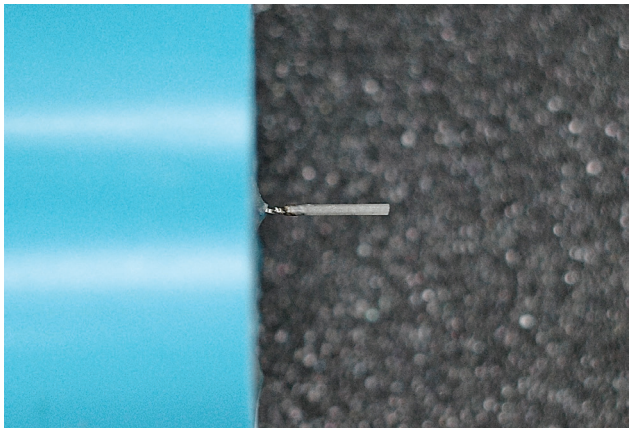


Figure 3 The characteristics of resin cements dislodged from the hypodermic tube

Discussion

Within this investigation, all experimental steps were concisely controlled including the measurement of the simulated separated instruments, the embedding of NiTi rotary files into the fixing molds, and the consistent mixing ratio of the materials. Furthermore, each sample underwent the placement of a hypodermic tube over the separated instrument to facilitate the complete curing of the resin cements according to the manufacturer's instructions. There were two resin cement systems used in this study to represent two types of resin cement and to prove their advantage over flowable resin composites that require light curing for complete polymerization [20]. Super Bond Universal, a self-curing resin cement, hypodermic tubes were left in place for 12 minutes while tubes containing RelyX U200, a dual-curing resin cement, were retained for 6 minutes with additional light curing to ensure thorough material setting, following the instructions Therefore, the failure load assessed in this study was intentionally from shear bond strength of each resin cement at complete setting stage.

This study aimed to evaluate the bond strength of resin cements applied in tube and glue technique when a NiTi rotary file separates during root canal treatment. NiTi rotary files are known to have a higher incidence of separation compared to other endodontic instruments. In addition, studies have shown a clear trend towards more separations in rotary files with continuous rotating motion compared to those with reciprocating motion [21-23]. Therefore, the present study was specifically to investigate the bond strength achieved with continuously NiTi rotary files. Among this file type, ProTaper is the most frequently studied according to prior research [24]. Furthermore, an in vitro study identified the ProTaper Next X1 as the most susceptible size to separate within root canals [25]. Therefore, ProTaper Next X1 was selected as the simulated separated instrument for this study.

Previous research has directed attention towards examining the failure load of flowable resin composites, as potential alternative adhesive aids. A study investigating the bond strength of Surefil SDR, a flowable resin composite, applied in the tube and glue technique with various pre-treatment protocols, demonstrated similar or higher mean failure loads compared to the current study. Specifically, the failure load of Surefil SDR without pre-treatment was recorded at 12.62 N, which is comparable to the failure load of RelyX U200 observed in our investigation [16].

The present study presented distinct findings regarding the performance of two resin cement types as chemical agents used in hypodermic tubes. Specifically, the failure load observed in the RelyX U200 group significantly surpassed that of the Super Bond Universal group. To our knowledge, this is the first investigation to assess the failure load associated with the removal of NiTi rotary files utilizing resin cements with the tube and glue technique. A recent study compared the breaking force among resin cement (Maxcem Elite), cyanoacrylate, and glass ionomer cement in the removal of the stainless steel files (K-file and H-file). Their finding reported a higher mean failure load across all experimental groups, ranging from 20.64 N to 96.85 N [15]. In contrast to our findings of 1.62 N and 15.50 N. Discrepancies in results may be attributed to the differences in material composition, the utilization of dissimilar separated instruments as well as the divergent study protocol.

To remove separated instruments by tube and glue technique, there are three possible factors which can affect the success rate of instrument removal: Mechanical interlocking from NiTi rotary files, chemical bond between NiTi rotary file and resin cement, and chemical bond between resin cement and hypodermic tube. Firstly, mechanical interlocking of resin cement filled in flutes of rotary NiTi file tends to obviously impact the capacity of separated instrument removal by tube and glue technique. When resin cement sets completely in the flutes of file, mechanical interlocking will be created. This was evidenced by the cylindrical shape of dislodged resin cement that remained adhered to the NiTi rotary files after testing (Figure 3). The predominant adhesive failure pattern observed between separated files and resin cement in both experimental groups can be explained by the chemical properties of the materials involved. However, NiTi rotary files are made from non-precious alloys, and one of their chemical properties is chemical inertness [26]. Therefore, the strength of the chemical bond between resin cement and NiTi rotary file might not be the major factor of successful separated instrument removal by tube and glue technique. The last factor affecting the success rate of instrument removal is the chemical bond between the resin cement and the hypodermic tube. Similar to NiTi alloy, the tube made from stainless steel is also chemically inert [27]. This was confirmed by stereomicroscope examination showing minimal resin cement retention on the inner walls of the hypodermic tubes. These findings suggest that mechanical interlocking, rather than chemical bonding, is the primary mechanism contributing to the success of the tube and glue technique.

The notable discrepancy in failure load between RelyX U200 and Super Bond Universal can potentially be accounted by the different compositions of these resin cements. Super Bond Universal consists primarily of methyl methacrylate (MMA) and 4-metacryloxyethyl trimellitate anhydride (4-META). MMA, characterized by a short and small molecular structure, along with 4-META, possesses only one methacrylate group.

This condition contributes to the formation of linear molecules and brittle polymers within Super Bond Universal. This molecular configuration ultimately diminishes bond strength as evidenced by previous studies [28-30]. In contrast, RelyX U200 is composed of Dimethacrylate dihydrogenphosphate monomers [31]. These monomers exhibited a structure containing methacrylate groups similar to the methacryloyloxydecyl dihydrogen phosphate (MDP). Several studies have shown that resin cements containing MDP or subjected to MDP primer pre-treatment demonstrated superior bond strength compared to those lacking MDP or no MDP pre-treatment [30-31]. Moreover, the polymer structure of RelyX U200, comprising two methacrylate groups and one phosphate group within the Dimethacrylate dihydrogenphosphate molecule,

facilitates the formation of a cross-linked molecular structure, thereby enhancing its mechanical properties. This cross-linked arrangement not only contributes to higher bond strength but also influences the polymerization rate, ultimately supporting material strength. Consequently, RelyX U200 exhibits superior bond strength compared to Super Bond Universal due to its robust cross-linked molecular structure [29, 31, 34-35].

However, the present study has some limitations. This study was primarily designed to investigate the failure load of chemical agents, focusing solely on the bond strength of the materials. The process of removing separated instruments within the root canals differs from the experiment conducted in fixing mold. Another limitation lies in the constrained accessibility of light curing in clinical situations.

The light-curing protocol is also a factor that influences the high bond strength of chemical agents. Particularly, the intensity of light exposure onto the resin cements significantly impacts the mechanical properties of the material which requires light-curing. Higher light intensity correlates with increased polymerization rates and reduced volumetric contraction [36]. Adequate light intensity also mitigates the occurrence of microporosity and interfacial gap [35]. Furthermore, sufficient light activation facilitates the initiation of polymerization by free radicals, leading to an accelerated polymerization rate [37-38]. In the context of tube and glue technique, previous studies have applied an optic fiber inserted through the hypodermic tube as a light-curing source [12, 16]. This method is to ensure the maximum light intensity exposed to the materials since flowable resin composites strictly require adequate light-curing for complete polymerization [17]. However, in clinical situations, the challenge arises from the inaccessibility of the light to the chemical agent when using the tube and glue technique. Therefore, the utilization of self-curing or dual-curing resin cements emerges as a practically prominent option.

Technical sensitivity is another factor related to the different outcomes in this study, especially for the Super Bond Universal group. The more separated components of resin cement might result in an increase in error in each step of preparation. The discrepancy of mixing and working time, the amount of resin cement loaded into the tube, and the different consistency of mixed resin cement in each sample is entirely factors that could affect the failure load in our study. Accordingly, the resin cements requiring less preparation steps tend to have more constant bond strength than those of the types composed of a more separated composition.

Based on the findings of this study and the various influencing factors discussed, the application of self-curing or dual-curing resin cements appears to offer greater clinical feasibility due to their mode of curing. When comparing the two resin cements assessed in this study, RelyX U200 showed superior bond strength over Super Bond Universal, a distinction that can be attributed to their compositions and molecular characteristics. Moreover, convenient usage with a precise mixing ratio associated with RelyX U200 contributes to reduce technique sensitivity, rendering it a more appealing option. Further investigations may focus exclusively on the self-curing mode for both types of resin cement. In addition, exploring the bond strength of other self-curing or dual-curing resin cements employed as chemical agents in the tube and glue technique is also interesting.

Conclusion

Within the condition of the study, the bond strength of RelyX U200 was significantly superior over that of Super Bond Universal. RelyX U200 is one of the attractive alternative chemical agents for tube and glue technique.

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Dr. Sirapatra Likittummawong is a student in the Master of Science Program in Dentistry (International Program) Major in Endodontics, Faculty of Dentistry, Mahidol University

Conflict of interest

The authors declare that they have no conflict of interest.

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