

Effect of Irrigant Activation Techniques on Calcium Silicate Sealer Bond to Dentin

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Article Info	ABSTRACT
Article History Received: 17.01.2025 Accepted: 06.06.2025 Published: 29.08.2025 Keywords: Calcium silicate, Root canal filling materials, Root canal obturation.	Aim: The aim of this study was to investigate the effect of irrigant activation techniques on the push-out bond strengths of calcium silicate-based sealers. Material and Methods: 65 single-canal teeth were instrumented with ProTaper rotary instruments. The specimens were randomly divided into two groups according to sealer type. Each group was randomly subgroup into four groups according to the final irrigation activation protocol; Nd:YAG laser, Vibringe, EndoActivator, Conventional Endodontic Needle Irrigation (n=6). The root canals were obturated with single gutta-percha and BioRoot RCS or MTA Fillapex. The teeth were sectioned into 1-mm-thick disc by using Isomet. Push-out force was applied until dislocation of obturating material and the bond strength value obtained was recorded as Newton. Data were analyzed using two-way Anova and Tukey HSD test (P = 0.05). Results: A significant interaction was found between sealers and irrigation methods (p <0.05), with the highest bond strength in the apical conventional needle irrigation group and in the coronal Nd:YAG laser and control groups. The BioRoot RCS had higher bond strength values than the MTA Fillapex (p = 0.00). Higher bond strength values were observed from apical to coronally (p <0.05). Conclusion: Based on the present results, within the limitations of this study, calcium silicate-based sealer (MTA Fillapex and BioRoot RCS) showed better bond strength in the presence of the smear layer.

Farklı irrigasyon Aktivasyon Tekniklerinin Kalsiyum Silikat Esaslı Patların Dentine Bağlanma Dayanımı Üzerine Etkisi

Makale Bilgisi	ÖZET
Makale Geçmişi Geliş Tarihi: 17.01.2025 Kabul Tarihi: 06.06.2025 Yayın Tarihi: 29.08.2025 Anahtar Kelimeler: Kalsiyum silikat, Kök kanal dolgu materyalleri, Kök kanal dolumu.	Amaç: Bu çalışmanın amacı, farklı irrigasyon aktivasyon tekniklerinin kalsiyum silikat esaslı kanal dolgu patlarının push-out bağlanma dayanımı üzerindeki etkisini araştırmaktır. Gereç ve Yöntemler: 65 adet tek kanallı diş ProTaper döner aletlerle şekillendirildi. Örnekler dolgu patlarına göre rastgele iki gruba ayrıldı. Her grup, son irrigasyon aktivasyon protokolüne göre rastgele dört gruba ayrıldı; Nd:YAG lazer, Vibringe, EndoActivator, Konvansiyonel Endodontik İğne İrrigasyonu (n=6). Kök kanalları tek kon gutta-perka ile BioRoot RCS veya MTA Fillapex kullanılarak dolduruldu. Dişler Isomet cihazı kullanılarak 1 mm kalınlığında kesilerek disklere ayrıldı. İtme kuvveti, kanal dolgu materyali yerinden oynayana kadar uygulandı ve elde edilen bağ gücü değeri Newton olarak kaydedildi. Veriler iki yönlü Anova ve Tukey HSD testi kullanılarak analiz edildi (P=0,05). Bulgular: Sızdırmazlık maddeleri ve irrigasyon yöntemleri arasında önemli bir etkileşim bulundu (p <0,05), en yüksek bağ mukavemeti apikalde konvansiyonel iğne irrigasyon grubunda ve koronalde Nd:YAG lazer ve kontrol gruplarında bulundu. BioRoot RCS, MTA Fillapex'ten daha yüksek bağlanma dayanımı değerlerine sahipti (p = 0,00). Apikalden koronale doğru daha yüksek bağlanma dayanımı değerleri gözlemlendi (p <0,05). Sonuç: Mevcut sonuçlara dayanarak, bu çalışmanın sınırlamaları dahilinde, kalsiyum silikat bazlı sızdırmazlık maddesi (MTA Fillapex ve BioRoot RCS) smear tabakasının varlığında daha iyi bağlanma değeri gösterdi.

To cite this article: Mutlu ŞN, Ünverdi Eldeniz A. Effect of Irrigant Activation Techniques on Calcium Silicate Sealer Bond to Dentin. NEU Dent J. 2025;7:151-62. <https://doi.org/10.51122/neudentj.2025.150>

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INTRODUCTION

Root canal therapy primarily aims to thoroughly cleanse and form the canal system while achieving a three-dimensional seal to isolate it from periapical tissues.¹ In the latest years obturating materials and sealers have been developed on the basis of dentin adhesion techniques in an attempt to seal the root canal more effectually.²

Mineral trioxide aggregate (MTA)-based root canal sealers have been introduced due to their advantageous biological properties.^{3,4} However, without the addition of chemicals that produce adequate flow, MTA's handling properties make it impossible to use as a sealer.³ To improve cement manipulation, ingredients like gels or water-soluble polymers have been introduced.^{5,6} The distinct physical and biological properties of calcium silicate and calcium phosphate-based bioceramic sealers have attracted a lot of attention lately.^{7,8} They have a chemical composition with a crystalline structure resembling that of tooth and bone apatite materials, and they include calcium phosphate, which increase the setting capabilities.⁹

MTA Fillapex (Angelus, Londrina, Brazil) is a sealer made of salicylate resin that contains silicon dioxide, bismuth oxide, and tricalcium silicate particles.¹⁰ It has low solubility, good sealing, and an appropriate flow^{11,12} and recommended for use with both warm and cold root filling methods.¹³ Salicylate resin's organic component decreased cell survival rates¹⁴ and increased cytotoxicity.¹⁵

A calcium silicate-based sealer, BioRoot RCS (Septodont, St. Maur-des-Fossés, France) can be applied using cold lateral condensation or single cone methods. The liquid is an watery solution of calcium chloride and polycarboxylate; the powder is composed of tricalcium silicate, povidone, and zirconium

oxide.¹⁶ Upon hydration, calcium hydroxide is released, which reacts with tissue fluid phosphates, resulting in the formation of calcium phosphate or calcium carbonate precipitates on the surface.^{17,18} It was reported that BioRoot RCS formed hydroxyapatite following contact with phosphate-buffered saline solution.¹⁷ It has been documented that BioRoot RCS stimulates human periodontal ligament cells to produce angiogenic and osteogenic growth factors,¹⁶ moreover, BioRoot RCS has biocompatibility and antimicrobial activity, may induce hard tissue deposition.^{17,19}

Endodontics has continually evolved with efforts to enhance irrigant delivery and agitation methods for more effective root canal irrigation. These techniques contain activation with gutta-percha cones, lasers, brushes, negative pressure irrigation technic, and sonic and ultrasonic machines.²⁰

The fundamental method of root canal irrigation is needle irrigation, and the most important aspect influencing solution penetration is the depth at which the needle tip is inserted into the canal.²¹

The EndoActivator System (Dentsply Tulsa Dental Specialties), operates using sonic energy to create dynamic intracanal fluid motion, significantly improving irrigation effectiveness compared to traditional needle techniques. It occurs a portable handpiece and three kind of disposable flexible polymer tips of variant sizes that do not cut root dentin.²²

One irrigation tool that can be used to sonically activate irrigation solution is the Vibringe system (Vibringe B.V.Corp, Amsterdam, Netherlands).²³ This device uses sonic flow technology in conjunction with acoustic streaming, and it runs at a lower frequency (2-3 kHz) than ultrasonic devices (25-40 kHz).^{24,25}

Laser-activated irrigation (LAI) has emerged as a modern technique, utilizing pulsed energy to activate irrigation solutions. In general, the literature demonstrate that mid-infrared erbium lasers have been used for the activation of irrigants^{26,27} and there are any written articles regarding Diode²⁸ and Nd: YAG laser²⁹ on the LAI.

Following chemomechanical preparation, the root canal walls develop a 1- to 2-mm thick smear layer, which is an amorphous and uneven surface. It is composed of both organic materials that contain pieces of odontoblastic processes, microbes and their byproducts, and necrotic pulp tissues, as well as inorganic dentin debris.³⁰ The root canal irrigation solutions and medications may not be able to enter the dentinal tubules due to the smear surface.³¹ Eliminating the smear layer could strengthen the filling material's attachment to the canal walls.³² But according to Saleh and Ruyter,³³ a stronger bond was not linked to the endodontic sealers' ability to penetrate the dentinal tubules after the smear layer was removed. To extract the organic and inorganic fragments of the smear surface, a final irrigation procedure utilizing sodium hypochlorite (NaOCl) and chelating chemicals such as EDTA is recommended.³⁴

The push-out bond strength test is typically used to assess resistance to endodontic sealers coming loose from the root dentine wall.^{35,36} In this mechanical test, a tensile load is applied longitudinally to the root sample's long axis until the sealer and core material are forced out or dislodged. Due to its great reproducibility and the fact that fractures happen parallel to the dentine bonding surface, the push-out bond strength test can be used to evaluate parallel-sided samples even in situations when bond strength is low.^{37,38} Additionally, the push-out test has the advantage of being able to test materials inside the root canal without the need

for extremely complex equipment, making it more advantageous than other bond strength tests like shear and tensile testing.³⁹ Using the push-out test, numerous studies have been carried out to ascertain the dislodgement resistance of different endodontic sealers; nevertheless, the reported results differ significantly.^{35,36}

Calcium silicate-based sealers' ability to adhere to dentine may be impacted by the physical and chemical changes that irrigation, with or without activation, may bring about on the surface of the dentin. This study researched the effects of various irrigant activation techniques on the calcium silicate-based sealers' push-out bond strength. The null hypothesis was that the binding strength between calcium silicate-based sealers and root dentine treated with various irrigant activation methods would be the same.

MATERIALS AND METHODS

Specimen Preparation

This study was approved by the Selçuk University Faculty of Dentistry Non-Interventional Clinical Research Evaluation Commission with the decision number 05, subject 2013/04, dated 07.05.2013. In this investigation, 65 human teeth that had been removed and had a single canal were used. To get rid of any leftover tissue debris, all teeth were submerged in a 2.5% sodium hypochlorite (NaOCl) solution for 24 hours. They were then kept in distilled water at 4°C until they were needed. Under constant water chilling, specimens were decoronated using a diamond bur (MANI Inc., Tochigi, Japan) to a standard root length of 12±1 mm. A size #15 K-file (Mani Inc., Tochigi, Japan) was used, and the working length was adjusted 1 mm short of the apical foramen. ProTaper (Dentsply, Maillefer, Ballaigues, Switzerland) NiTi rotary files were used to prepare the root canal. From SX to F4,

the ProTaper files were utilized. Between the files, a 30-gauge endodontic irrigation needle (1-side vent) (Transcodent, Sulzer, Germany) was inserted 1 mm short of the working length (WL) to irrigate the canals with 2 mL of 2.5% NaOCl (Caglayan Kimya, Konya, Türkiye).

Specimens (n=30) were randomly divided into two main groups according to the type of root canal sealer used: Group A was filled with BioRoot RCS, and Group B was filled with MTA Fillapex.

Sticky wax (StickyWax KerrLab USA) was used to glue the tooth root ends to the glass petri dishes. Each main group was then further divided into five subgroups (n=6 each) based on the final irrigation protocol used:

Group 1A / 1B: Nd:YAG Laser Activation (n=6); Using a 30-gauge endodontic irrigation needle, the samples were irrigated with 3 ml of 3% NaOCl (Caglayan Kimya, Konya, Türkiye) and 3 ml of 17% EDTA (Merck KGaA, Darmstadt, Germany). After each irrigation solution, 3x10 sec laser application was performed into the root canal. In accordance with Moon, Kim, et al.,²⁹ a 1064 nm Nd:YAG laser (Fotona, Madrid, Spain) was employed for laser irradiation. In a 200-µm flexible endodontic fiber, the usual settings were 100 mJ/pulse and 15 Hz (average power = 1.5 W). The laser fiber was held 1 mm away from the WL and moved in a circular motion in the apical-coronal direction during irradiation. Three times, the laser fiber was entered and removed for ten seconds each, separated by fifteen seconds.

Group 2A / 2B: Vibringe Activation (n=6); A 30-gauge endodontic needle was utilized with a Vibringe tool. The needle was positioned 2 mm below WL. Using a Vibringe, 3 ml of 3% NaOCl and 3 ml of 17% EDTA solutions were used to irrigate the teeth for 30 seconds.

Group 3A / 3B: EndoActivator (EA) (n=6); EA was administered 2 mm behind the WL to the irrigated samples in group 1 after each irrigation. The EndoActivator System was utilized for 30 seconds at 10.000 cycles, with a 25/02 polymer tip for each solution

Group 4A / 4B: Conventional Needle Irrigation (CNI) (n=6); A syringe was used to apply irrigation solutions, which included a 30-gauge closed-end tip and a needle with side-port aperture. The irrigation needle was put 1 mm short of its WL. All samples were then irrigated with 3 ml of 3% NaOCl and 3 ml of 17% EDTA solutions, each of which was activated using an irrigation needle.

Group 5A / 5B Control group (n=6); Samples in the control group were irrigated with a conventional needle and 6 milliliters of distilled water for 1 minute. Activation was not done.

In addition, one extra sample from each subgroup (not filled) was prepared to evaluate smear layer removal using scanning electron microscopy (SEM). Samples that had been irrigated but not filled were allowed to air dry at room temperature for the whole night before being sputter coated with gold or palladium for three minutes. To evaluate the removal of the smear layer, samples were seen using a scanning electron microscope (SEM) (Leo 440, Electron Microscopy Ltd., Cambridge, UK). SEM was used for the observations, with a magnification of 1.000× and a voltage of 20 kV.

To get rid of any leftovers from the solutions other than the control group, the root canal was irrigated with 3 milliliters of distilled water following the last irrigation treatments. Four Protaper F4 paper cones (Dentsply, Maillefer, Ballaigues, Switzerland) were used to dry the root canals. Following the manufacturer's instructions, a mixture of powder and liquid BioRoot RCS canal sealer was created.

A single gutta-percha cone (ProTaper Universal F4, Dentsply Maillefer) was coated with BioRoot RCS sealer and inserted into the root canal of the WL. Following root filling, the coronal orifice was filled with a temporary filling substance (Cavit, 3M; ESPE, St. Paul, MN), and all specimens were kept in an incubator at 37°C for seven days.

MTA Fillapex Group; The same treatments were followed as in the BioRoot RCS groups, but with MTA Fillapex sealer. MTA Fillapex canal sealer, consisting of two packs, was mixed according to the manufacturer's recommendation.

Push-Out Bond Test

An Isomet saw (Buehler Ltd., Lake Bluff, IL) was used to slice the teeth into a 1-mm-thick disc perpendicular to the long axis while being lubricated with water. A total of 36 specimens were obtained from each group, 18 from the apical and 18 from the coronal.

A digital caliper was used to measure each disk's thickness precisely, to within 0.1 mm. The plungers were attached to a universal testing machine's load cell. In order to closely match the size of the filling material, three plungers of varying sizes were used. On the root canal filling, a vertical load was applied at a rate of 0.5 mm/min in an apical to coronal orientation. Push-out force was applied until dislocation of obturating material and the bond strength value obtained was recorded as Newton

(N). Using the formula $\text{Area} = 2\pi rh$, where $p = 3.14$ and r and h are the measured height and radius of the pushed-out filler material in millimeters, respectively, the area of the bonded interface was computed.

Statistical Analysis

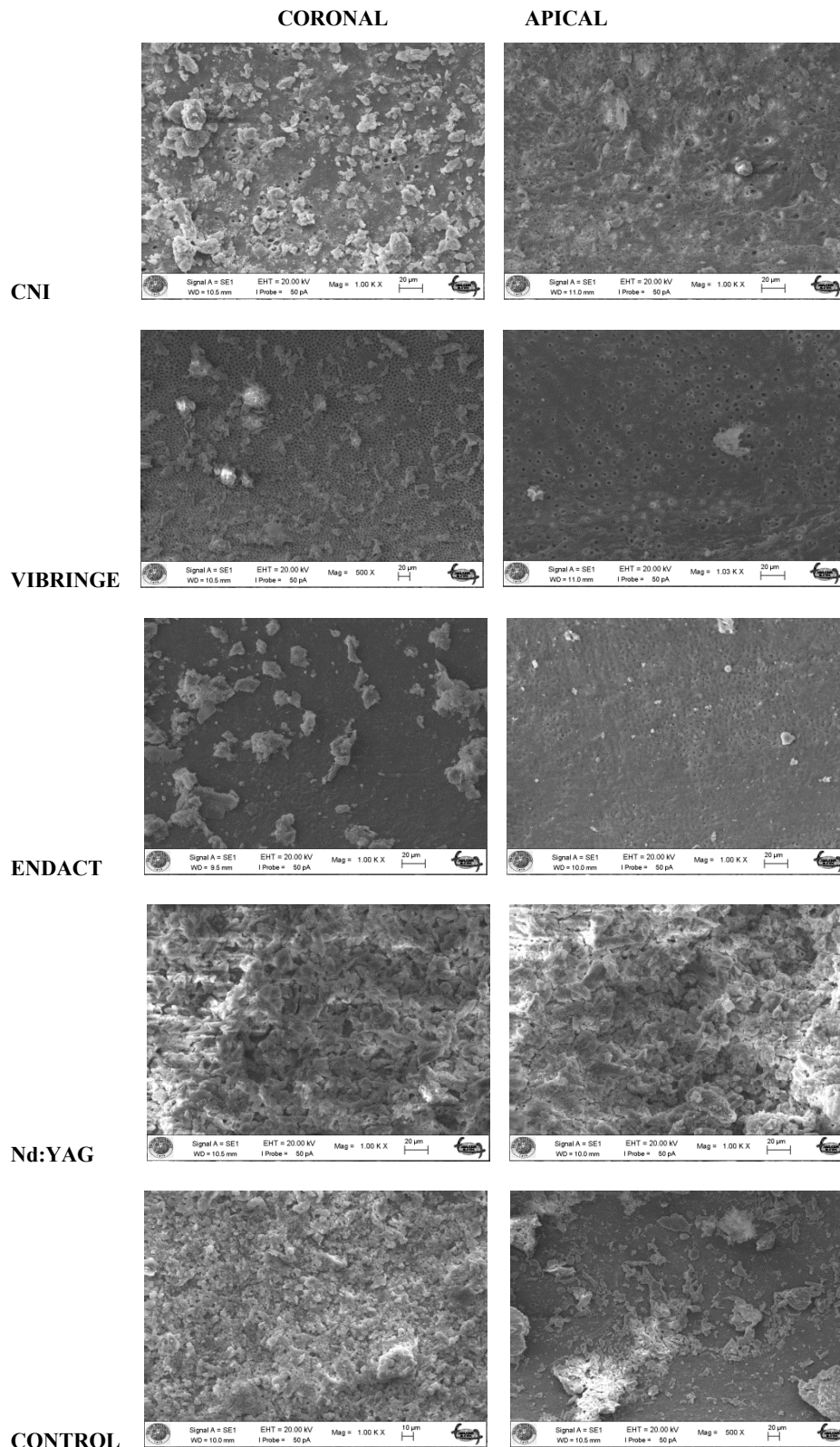
The statistical analysis of the data was carried out using the SPSS (Statistical Package for Social Sciences) 17.0V software. The two-way Anova Test was used to evaluate the results gathered from the bond strength test of root canal filling sealers. The Tukey HSD test was used to statistically evaluate irrigation techniques, sealers, and regional differences. The accepted significance threshold was $p=0.05$.

RESULT

The removal of the smear layer

Specimens analyzed with a 1000x magnification scanning electron microscope are demonstrated in Figure 1. The apical and coronal areas of the SEM samples from the group that received the Nd:YAG laser treatment displayed recrystallization and the development of smear layers. On the dentin surface, EndoActivator-activated groups exhibit some debris and a smear layer. Neither the control group nor the traditional endodontic irrigation needle were able to eliminate the smear layer on the dentin surface. The vibringe group exhibited open dentinal tubules in the coronal and apical thirds in contrast to the other groups.

Figure 1: Representative scanning electron microscopy pictures for the various irrigant activation techniques (magnification x1000) displaying selected samples from the coronal and apical portions



Push-Out Bond Strength

The BioRoot RCS has considerably higher binding strength values than the MTA Fillapex ($p = 0.00$).

Bond strength values were higher in the apical region than in the coronal region, according to inter-regional comparisons ($p < 0.05$).

The bond strength values were higher in the apical region (mean=0.79), than the coronal (mean = 0.37) in both sealer groups ($p > 0.05$).

Table 1: Mean and standard deviation values of irrigation activation methods

Techniques	Means
CNI	0.79±1.05 ^a
Vibringe	0.47±0.46 ^b
Endact	0.42±0.30 ^b
Nd:yag	0.64±0.47 ^{ab}
Control	0.57±0.47 ^{ab}

The difference between the means carrying different letters on the same line is significant ($p < 0.05$)

There was statistical interaction between the techniques ($p < 0.05$) (Table 1). Despite the fact that the CNI group (mean = 0.79)

demonstrated the strongest binding, there was no discernible difference between the control group, Nd:YAG laser, and CNI group ($p > 0.05$).

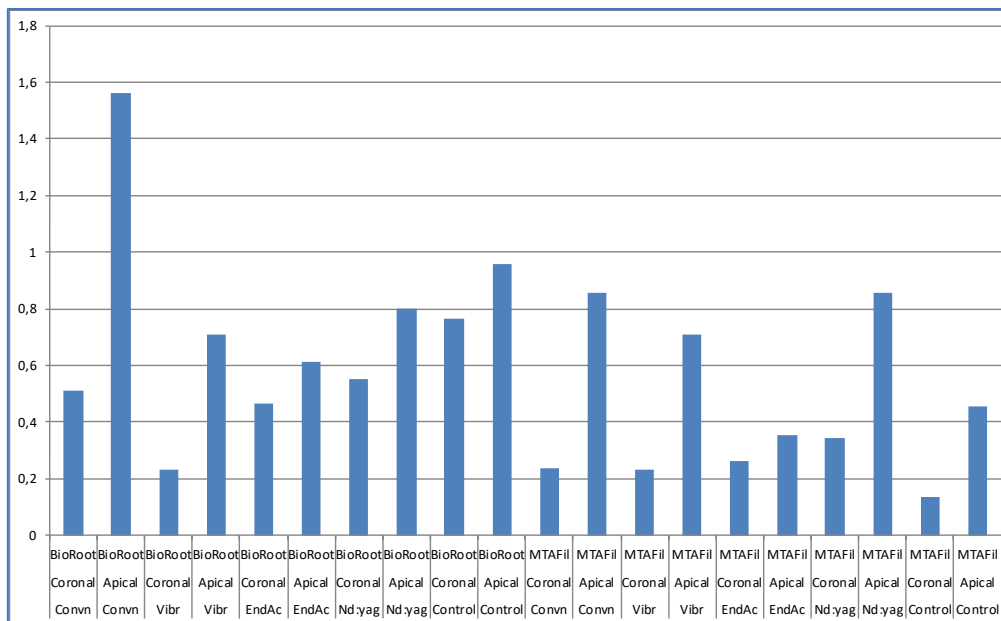
When the regions and techniques were examined, it was seen that the bond strength of the apical region of the teeth was better with manual activation ($p < 0.05$). The bond strength that was the lowest was seen in samples using EA in the apical region and Vibring in the coronal region. The highest bond strength in the coronal region was measured in Nd: Yag laser and control groups.

A significant interaction ($p < 0.05$) was observed between the sealers and the irrigation methods. The apical CNI approach yielded the highest bond strength values, whereas in the coronal region, the highest values were observed in the Nd:YAG laser and control groups.

When the bonding strength of the sealers was evaluated according to the techniques ($p < 0.05$), the bond strength of BioRoot was higher. Vibringe samples were equally measured in both sealers.

There was no statistical interaction between the sealers, regions and techniques ($p > 0.05$) (Figure 2).

Figure 2: Bond strength findings by sealers, techniques and regions



DISCUSSION

Good filling ability is a desirable feature of a root canal sealer. The purpose of this study was to investigate the effects of sodium hypochlorite and EDTA solutions activated by the EndoActivator, Nd:YAG Laser, Vibringe, and conventional endodontic needle irrigation on the bond strength of bioceramic-based BioRoot RCS and MTA Fillapex sealers to root dentin in removing the smear layer. The null hypothesis proposed that the binding strength between calcium silicate-based sealers and root dentine treated with various irrigant activation methods would be the same. However, based on the results of this study, the null hypothesis was rejected. Significant differences in the push-out bond strength were observed among the different irrigant activation methods. A common method for assessing the root canal filling materials resistance to dislodgement is the push-out test.⁴⁰ While dislodging, a selection of two calcium silicate based sealers was investigated in order to get push-out bond strength and failure mode. To remove the smear, 17% EDTA was applied to the dentine. There are two types of adhesion mechanisms: mechanical and chemical. Smooth surfaces tend to have stronger adhesion when it comes to chemical bonding. Conversely, for micromechanical bonding to occur, the dentin matrix's surface must have non-uniforms that let the material to pass through.⁴¹

Among canal filling procedures, cold lateral and vertical compaction methods have proven beneficial because they can force the sealer into the uninstrumented sections where bacteria are still present.⁴² The application of these techniques is time consuming, it is important for the dentist to be experienced and must be visually supported by radiographs during canal filling. The single cone approach is really employed by the majority of dentists because it is easy and fast to apply. Using nickel-titanium files, it is very advantageous to use gutta-percha cone suitable for the angle and apical diameter of the last applied file. Placing the sealer on the apical portion of the root with a single cone is possible since the gutta-percha

cone and the final file used are compatible. The space left from the gutta-percha in the canal is filled with patches and spaces.⁴³ Therefore, the single cone technique may not be considered reliable. Martins, Leoni⁴⁴ stated that the single-cone technique leaves a much larger area for the sealer in the morphologically irregular coronal and middle trinity, and this increases polymerization shrinkage and causes the sealer to separate from the root canal walls. However bioceramic sealers can provide a durable three-dimensional obturation along all root canals. When used with compatible gutta percha, they can spread to areas that instruments cannot reach or any irregular root canal with their perfect wetness and fluidity. In addition, due to their adhesive properties to dentin, they also reduce root fractures that occur over time. BioRoot RCS sealer is one of these new bioceramic materials. BioRoot RCS should be considered not only as a sealer but as a complete root canal filler. BioRoot RCS is clinically very successful, especially as a filler in the single-cone filling technique.⁴⁵ Because of these advantages, we chose the single cone technique in our study, where we used bioceramic-based sealers.

BioRoot RCS paste (Avg=0.72) showed higher bond strength than MTA Fillapex paste (Avg=0.44). ($p=0.00$). MTA Fillapex is known to have weaker bond strength than sealers like AH Plus and iRootSP.⁴⁶⁻⁵⁰ According to Sagen Ustun,⁴⁸ MTA Fillapex's low bond strength is due to the weak adhesion capacity of tag-extension-like structures.

Assmann, Scarparo⁴⁷ also reported that the resin in the structure of MTA Fillapex may affect the adhesion of the sealer to the dentin. In this reserach, porosity and cracks in the resin matrix were identified in the samples analyzed under a scanning electron microscope after MTA Fillapex samples were put through a seven-day solubility test in deionized water.⁵¹

In these studies, as in our study, MTA Fillapex supports low binding values. Camilleri⁵² reported that adding calcium chloride to sealers containing MTA improves sealer filling.

Calcium chloride regulates and shortens the setting time, decreases the amount of water required to set the sealer, and increases resistance to hydrostatic pressure.^{52,53} One possible explanation for the increased bond strength is that the setting accelerating calcium chloride can alter the characteristics, chemical composition and surface area of the pores of the cements.⁵⁴ The calcium chloride concentration of BioRoot RCS accounts for the difference between it and the calcium silicate-based sealer MTA Fillapex employed in this investigation.

Ingle⁵⁵ reported that the cause of endodontic treatment failure was inadequate canal filling and endodontic. Depending on the decrease in the diameter and amount of dentinal tubules in the apical region,^{56,57} the bond strength decreases.⁵⁸ Paque, Luder⁵⁹ reported that the difficulty of applying materials to the apical and sclerotic structure of dentin in the apical region makes it difficult to adhere in this region. Considering our study's findings, the reason why the apical bond strength values were better in both sealers may be that the Ni-Ti rotary file used in the preparation was filled using appropriate gutta-percha. When only the regions were compared, the bond strength in the apical region was found to be greater than that in the coronal region. As a result of this finding, the sealers we used in the experiment very advantageous.

EL-Ma'aita, Qualtrough⁶⁰ reported that When the smear layer is eliminated, the bond strength of sealers based on calcium silicate decreases. The Vibringe and EndoActivator groups in our study had weaker bonds than the smear layer because they were cleaner, but higher in the control, Nd:YAG laser and manual irrigation groups that had the smear layer. The conventional endodontic needle irrigation, Nd:YAG Laser, and control groups had the highest bond strength for both sealers in our study. Although the application of Nd:YAG laser to the dentin area increases the bonding values, in SEM analysis, it is observed that the smear layer persists and that retention is caused by recrystallized rough regions.^{61,62} This micro-thinned surface may also have augmented the

bond strength of the Nd:YAG laser groups.

The results of this study may not be fully suitable for estimating a clinical setting. However, this laboratory analysis yields trustworthy and reproducible results that can be utilized to compare novel endodontic sealers and set international standards.

In addition, the bond strength of canal sealer to the root canal is only one of the ideal properties of endodontic paste. As a result, other features of root canal sealers should be researched to provide a more reliable conclusion.

CONCLUSION

According to the results of this study, considering the limitations of the study, MTA Fillapex and BioRoot RCS, which are calcium silicate-based root canal sealers, showed higher bond strength in the presence of the smear layer. The best approach of removing the smear layer is irrigation with Vibringe and EndoActivator. Especially the smear layer remaining after irrigation activation with Nd:YAG laser and endodontic irrigation needle and the recrystallization areas created by the laser increased the bonding of calcium silicate-based sealer. This study presented a feasible and simple approach for assessing the bond strength of a calcium silicate-based sealer to the canal wall.

Ethical Approval

Ethical approval for this study was received by Selçuk University Faculty of Dentistry Non-Interventional Clinical Research Evaluation Commission (2013/04).

Financial Support

This research was supported by Selçuk University Scientific Research Projects Coordination Office with project number 13102025.

Conflict of Interest

The authors deny any conflicts of interest related to this study.

Author Contributions

Design: ŞNM, AUE, Data collection or access: ŞNM, Analysis and comments: ŞNM, AUE, Literature search: ŞNM, AUE, Writing: ŞNM.

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