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Spectrophotometric Analysis Evaluating Apical Sealing Ability in Retrograde Filling Using Proroot MTA and Endocem MTA Premixed Regular: An In-Vitro Study

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Abstract: ***Root-end filling aims to achieve an effective apical seal by preventing the leakage at the root-end cavity interface. Adaptation between the dentinal walls and root-end filling material contributes to marginal integrity and helps establish a three-dimensional impermeable seal. This in-vitro study aimed to evaluate and compare the sealing ability of ProRoot MTA and Endocem MTA Premixed Regular as root-end filling materials using ultraviolet spectrophotometric dye extraction analysis. Fifty extracted single-rooted human teeth were selected, and standardized root canal preparation, obturation, apical root-end resection, and retrograde cavity preparation were performed in all samples. The specimens were randomly divided into two groups based on the root-end filling material used: Group I (ProRoot MTA) and Group II (Endocem MTA Premixed Regular). Following placement of the materials, all samples were subjected to dye penetration using methylene blue to evaluate apical microleakage. The absorbed dye was extracted from the specimens and analyzed using UV Spectrophotometer by measuring optical density values in the absorbance range of 500–700 nm. The recorded data were subjected to statistical analysis to compare the sealing ability of the two groups. Student's t test was used to compare the mean optical density of Endocem MTA Premixed and ProRoot MTA. It was found that Endocem MTA Premixed demonstrated lower mean optical density (0.545) compared with ProRoot MTA (0.682). Endocem MTA Premixed Regular exhibited significantly better sealing ability than ProRoot MTA.***

Keywords: ProRoot MTA; Endocem MTA Premixed Regular; UV spectrophotometry

1. Introduction

The primary objective of periapical surgery is to establish a fluid-tight apical seal between root canal and periradicular tissues. The root end filling is the procedure by which an inert, biocompatible material is packed into the root end cavity to prevent the leakage of microorganisms and provide a three-dimensional apical seal. Various root end filling materials i.e. amalgam, gutta percha, intermediate restorative, glass ionomer and composites were used previously, however they failed to demonstrate all of the desired ideal properties (Srikumar et al. 2022, Eid et al. 2023).

ProRoot MTA (Dentsply, Tulsa, OK, USA) was the first type of hydrophilic bioceramic material used for root-end filling (Park et al. 2023). ProRoot MTA is composed of Portland cement, bismuth oxide, calcium sulfate dihydrate, tetracalcium aluminoferrite, gypsum and calcium oxide. It has superior physico chemical and biological properties (Kang et al. 2021). It has sealing ability, osteogenic potential and biocompatibility due to its ability to release calcium ions, form an adherent interfacial layer and trigger the precipitation of hydroxyapatite on its surface (Bird et al. 2012).

Despite its favorable sealing ability, ProRoot MTA presents less than ideal working properties, since the cement resulting from the mixture of powder with liquid is difficult to handle. To overcome these limitations, various approaches have been attempted to achieve convenience of manipulation and delivery.

A new injectable bioceramic root-end filling material, Endocem MTA Premixed Regular (Maruchi, Wonju, Korea) is developed recently (Park et al. 2023). Endocem MTA Premixed Regular consists of tricalcium silicate, dodecacalcium hepta-aluminate, zirconium oxide, dimethyl sulfoxide, calcium sulfate, lithium carbonate, phyllosilicate mineral, hydroxypropyl methylcellulose and silicon dioxide. Dimethyl sulfoxide, a non-aqueous vehicle which improves the adhesive bonding to coronal dentin and decrease leakage by increasing dentin wettability. It also provides greater plasticity and higher flow. It eliminates the need for mixing time, and it has also demonstrated improved solubility and high compressive strength, making it a potentially favorable option for clinical use. It can induce biomineralization by forming hydroxyapatite crystals inside the dentinal tubules. The phyllosilicate component provides a hemostatic effect, making it easy to use without bleeding control. Collectively, these properties contribute to its excellent sealing ability, outstanding biocompatibility, and stable dimensional changes (Jang et al. 2023, Park et al. 2023).

Various studies have emphasized the importance of root-end fillings in outcomes of apicoectomies by reporting that teeth with root-end fillings showed favorable results compared with those without root-end fillings. The quality of apical seal achieved by root-end filling materials has been assessed by various methods like the degree of dye penetration, dye extraction, radioisotope penetration, bacterial penetration, electro- chemical means and fluid filtration techniques. In dye extraction method, all the dye that leaked through the apex is recovered by dissolving in acid. It also quantitatively measures the optical density of the solution by the use of a spectrophotometer thus provides reliable results in microleakage studies (Nepal et al. 2020).

Endocem MTA Premixed Regular is relatively new to the market, having been introduced in 2023, and information about it remains limited particularly in comparison with established materials such as ProRoot MTA and only few studies have investigated its sealing ability.

Therefore, the objective of the present study was to assess and compare the sealing ability of ProRoot MTA and Endocem MTA Premixed Regular by using UV Spectrophotometric analysis.

2. Methods

This in-vitro study was approved by the Dental Research Ethics Committee of the Punjab Government Dental College and Hospital, Amritsar affiliated to Baba Farid University of Health Sciences, Faridkot (No. BFUHS/2k25/p- TH/9355). All patients and/or legal guardians had given their written informed consent for the use of their teeth for research purposes.

Fifty human mandibular premolar teeth with single root were collected. The calculus and soft tissue debris were removed from the surface of the teeth by mechanical debridement using ultrasonic scaler (Guilin Woodpecker Medical Instrument Co., Ltd., China). The teeth were stored in a sodium hypochlorite (NaOCl) solution diluted 1:10 with distilled water until the time of experimental usage.

To ensure standardized root lengths, the roots were sectioned 12.00 mm from the apex using a diamond disc (Wuhan Jinguang Medical Technology Co., Ltd., China) attached to straight hand piece under constant water cooling (NSK, Japan). The canal patency was determined by No. 10 K file (Mani, Tochigi, Japan) and working length was determined by subtracting 0.5mm from the length achieved with the tip of the trial file just visible at the apical foramen of each root canal. The root canals were instrumented using a rotary instrument system ProTaper Next (Dentsply Maillefer, Ballaigues, Switzerland) up to size F3. During instrumentation, the root canals were irrigated with 2 mL of 1% NaOCl using a 27-gauge side-vented needle. The smear layer formed on the root canal wall was removed by using 1 mL of 17% ethylenediaminetetraacetic acid for 2 minutes. After that, the root canals were irrigated with 2 mL of distilled water and dried with paper points (Dentsply, Malliefier) followed by obturation with single cone technique using AH Plus sealer after verifying master cone X ray.

The access cavities in all the teeth were restored with resin composite filling (Spectrum, Dentsply Sirona, Konstanz, Germany). The specimens were placed in 100% relative humidity at 37° C for 24 h. The apical 3 mm of each root was resected at 90° angle to the long axis of the root with straight fissure bur (Dentsply Detery, Konstanz, Germany) in a high-speed contra-angled handpiece (NSK, Japan) along with water coolant. The teeth were randomly divided into two experimental groups i.e. Group I and Group II according to type of root-end filling material used.

Group I: ProRoot MTA (Dentsply, Tulsa, OK, USA) Retrograde preparations were made in the teeth uniformly to a depth of 3 mm using Pro-ultrasonic tip No. 2 (Dentsply Maillefer, Dentsply India Pvt. Ltd, Mumbai) (figure 1). The depth of the cavity was checked using a calibrated periodontal probe. Root-end cavity was irrigated with normal saline, dried with absorbent paper points. ProRoot MTA was manipulated according to the manufacturer's instructions. The mixing was done on a paper pad with distilled water in 3:1 powder ratio. When the mixture exhibited putty like consistency after about mixing of 30 seconds, ProRoot MTA was dispensed into the root end cavity using MTA carrier (S S White, New Jersey) (figure 2) and compacted using a small plugger. Any excess material was removed and the surface of the root was cleaned with a moist piece of gauze.

Group II: Endocem MTA Premixed Regular Retrograde preparations were made in the teeth with same method as in Group I. The Endocem MTA Premixed Regular was injected into the prepared retrograde cavity (figure 3) and apically placed using paper points. A cotton pellet fully soaked with distilled water was placed on it to facilitate setting reaction.

For microleakage evaluation, the samples were first coated with two layers of nail varnish, leaving the apical 3 mm uncoated. After the varnish had dried, the samples were placed in 50 airtight containers, each containing 5 mL of 2% methylene blue solution. These containers were then stored in incubator at 37 °C for duration of 72 h. After the incubation period, the samples were washed under tap water to remove any traces of the dye, dried and nail varnish removed with scalpel. Each sample was transferred to a test tube containing 2 ml of freshly prepared 65% HNO_3 and left for another 72 h to denature and completely dissolve the teeth (figure 4). The solution obtained from each tube was transferred to Effendorf tubes and centrifuged (Eppendorf MiniSpin centrifuge) at 3500 revolution per minute (RPM) for 5 min. From each sample, 2ml of the supernatant layer was collected and transferred into plastic cuvettes and then analysed using an UV Spectrophotometer (Shimadzu UV-1900 UV-Visible Spectrophotometer) at 500-700 nm wave length with 65% concentrated nitric acid as the blank (figure 5). The readings were recorded for each specimen as Absorbance Units (AU). The absorbance values indicated the dye leakage and sealing ability of root end filling materials.

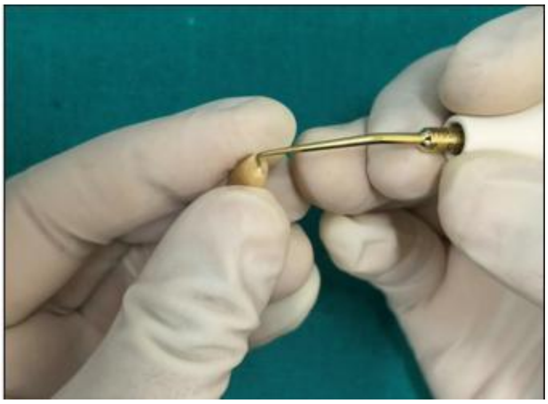

Figure 1: Retrograde preparations to a depth of 3 mm using pro-ultrasonic tip no. 2

Figure 2: Proroot mta injected into the prepared retrograde cavity with mta carrier

Figure 3: Endocem mta premixed regular injected Into the prepared retrograde cavity

Figure 4: Sample after 72 h showing complete denaturation and dissolution of the teeth

Figure 5: Analysis using an UV Spectrophotometer

3. Results

The data collected in Group I (ProRoot MTA) and Group II (Endocem MTA Premixed Regular) were tabulated and statistically analyzed using the student's t-test using SPSS version 23.0 (IBM Corp., Armonk, NY, USA). A p value < 0.05 was considered indicative of statistical significance.

Table 1: Spectrophotometric analysis of optical density for Group I and Group II in absorbance units

Group	No. of observations	Mean optical density	SD
Group I	25	0.682	0.122
Group II	25	0.545	0.206

SD-standard deviation

As per the table 1, the basic statistical analysis was computed for Group I (ProRoot MTA) and Group II (Endocem MTA Premixed), with 25 observations in each group. Group I showed a higher mean optical density (0.682) than Group II (0.545)

Table 2: Comparison of means of two groups using t-test

t (test statistic)	Degree of freedom(df)	p value	Mean Difference	Std. Error Difference	95% Confidence Interval of the Difference	
					Lower	Upper
2.850	48.000	0.006	0.136	0.048	0.040	0.233

*p>0.05; not significant, p≤0.05; significant; p<0.001 highly significant

As per the table 2, statistical analysis by using t test and p value demonstrated statistically significant difference in absorbance values between Group I and Group II (p<0.05). Endocem MTA Premixed (Group II) showed significantly better sealing ability than ProRoot MTA (Group I).

4. Discussion

Apical microleakage remains one of the primary causes of endodontic failure; therefore, achieving an effective apical seal using an appropriate root-end filling material is critical for long-term treatment success (Khandelwal et al. 2015). The main aim of root end filling is to prevent the movement of the bacteria and diffusion of bacterial products from the root canal into periapical tissues and vice versa. (Ravichandra et al. 2014). The root end filling material should possess ideal properties such as biocompatibility, dimensional stability, radiopacity, ability to set in a wet environment, antibacterial properties, easy handling, adequate compressive strength and hardness, osteoinductive and osteoconductive properties and adherence to the canal walls to provide a good apical seal (Soundappan et al.

2014).

Many materials have been used for root-end fillings in endodontic surgery. However, there is no one material that is universally accepted as the best. Mineral trioxide aggregate is considered an ideal retrograde filling material. It shows the formation of calcium phosphate precipitation at the interface which reduces the risk of marginal percolation and gives promising long-term clinical success. (Mandava et al. 2015, Ravichandra et al. 2014). It has favorable properties suitable for a root end filling material such as excellent sealing ability, biocompatibility, good compressive strength (67 Mpa), periradicular regeneration, osteoinductive ability, insoluble in fluids once set, radiopacity and antibacterial effect. It has also been shown to induce hard tissue formation, including deposition of cementum. However, it also has certain drawbacks like prolonged setting time (2 h 45 min), difficulty in manipulation, technique sensitivity, and it is quite expensive as well (Khandelwal et al. 2015, Angitha et al. 2023).

Recently, several commercial Calcium silicate-based cements (CSCs) with their ease of use and operability, are favoured in clinical treatments (Jang et al. 2023). Endocem MTA Premixed (Maruchi, Wonju, Republic of Korea), is developed in with a quick-setting time of 4 min as per the manufacturer's specifications. The pre-mixed injectable type, Endocem premixed is considered the most effective for its antibacterial activity, sustained antibacterial effectiveness, cytotoxicity, and radiopacity. It offers several advantages, including the elimination of mixing requirements, convenient handling and enhanced applicability (Moon et al. 2023).

In the present study, UV Spectrophometric analysis was used to compare the sealing ability of ProRoot MTA (Group I) and Endocem MTA Premixed (Group II). Ultraviolet-Spectrophotometric analysis determines the amount of the absorber (Methylene blue dye) in the solution and is interpreted that absorbance of the solution is directly related to the amount of the dye leaked along the tooth cement interface and is recorded as absorbance units. The absorbance values, recorded in absorbance units (AU), indicated the extent of dye leakage and the sealing ability of the root-end filling materials. (Srikumar et al. 2022).

In Group II, Endocem MTA Premixed (0.545) revealed better sealing ability than Group I, ProRoot MTA (0.682). In Endocem MTA Premixed, less microleakage may be attributed to its smaller particle size, which facilitates deeper penetration into dentinal tubules and enhances adaptation to the root-end cavity walls. Additionally, the short initial setting time of ECPR (260 ± 17.32 s), along with its higher flow (>17 mm) and lower film thickness (50 um) compared with powder–liquid calcium silicate cements, may contribute to improved adaptation to irregularities and enhance the material's ability to seep into canal spaces. In contrast, the comparatively lower sealing ability of ProRoot MTA may be related to its larger particle size. The powder liquid ratio variation, excess water in mixed material led to void formation and increased porosity affected the sealing ability of root-end material.

Park et al. (2023) evaluated the push-out bond strength to quantify the interfacial adaptation values of the Endocem MTA premixed regular to the root canal dentin in comparison to ProRoot MTA. They found that Endocem MTA premixed regular is comparable to ProRoot MTA regarding interfacial adaptation and can be considered to show adequate performance in root-end filling. Back et al. (2023) showed that Endocem MTA Premixed Regular exhibited significant release of calcium ions after setting, which contributes to bioactivity and supports hard tissue formation. The high sulfide ion release indicating enhanced mineralization potential along with a low solubility value, which is important for providing long-term stability and preventing microleakage from periapical tissues. Moreover, EMPR had a high pH than conventional MTAs and it increased with time. An alkaline pH has antibacterial activities and mineralization effects.

Supporting these findings, Kim Y et al. (2026) reported that Endocem MTA Premixed demonstrated high biocompatibility, maintaining greater than 90% cell viability. A significantly higher ALP activity, indicating enhanced early osteogenic differentiation and promoted mineral deposition. These findings suggest that the material provides a biologically conducive environment for periradicular tissue healing and regeneration following endodontic microsurgery. Moon et al. (2023) reported that Endocem MTA Premixed have been shown to promote proliferation and differentiation of periodontal ligament stem cells (PDLSCs), which may contribute to improved periradicular healing and tissue regeneration. Furthermore, Kim Y et al. (2026) reported satisfactory setting characteristics under simulated clinical conditions, indicating that the material is capable of achieving adequate hardening and dimensional stability after placement. Such properties are clinically important because a stable and well-set root-end filling material is essential for maintaining the integrity of the apical seal, preventing microleakage, and facilitating long-term periapical healing.

Limitations of this study include the in vitro nature of the study, which does not actually mimic the clinical situation, and performing the study at a certain point in time rather than at different intervals.

5. Conclusion

The results of the present study indicate that this new injectable bioceramic root-end filling material, Endocem MTA Premixed has better sealing ability. The better handling properties of this material combined with superior biological, mechanical and physical property suggest the superiority of, Endocem MTA Premixed over other root end filling materials.

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