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Comparative Microstructural and Chemical Evaluation of Pozzolan-Based MTAs and ProRoot MTA Using SEM, EDS, and FTIR

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ABSTRACT

Aim: This study aimed to compare the microstructural and chemical characteristics of three available mineral trioxide aggregate (MTA)–based cements using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and Fourier transform infrared spectroscopy (FTIR).
Materials and Methods: ProRoot MTA, Endocem MTA, and Endocem Zr were evaluated. Samples were prepared in silicone molds, set at 37 °C for 24 hours, ground into powder, and stored at 37 °C under 100% humidity for 72 hours. SEM analyses were performed at magnifications ranging from ×1000 to ×20.000. EDS spectra were obtained from two randomly selected regions of each specimen. FTIR analysis was conducted over a wavenumber range of 400–4400 cm^{-1}.
Results: SEM analysis demonstrated differences in surface morphology. ProRoot MTA showed small, uniformly distributed particles, indicating a homogeneous microstructure. Endocem MTA exhibited larger, irregular particles with a heterogeneous appearance, whereas Endocem Zr presented an intermediate particle size and a smoother surface. EDS confirmed calcium, silicon, and oxygen as major elements in all materials. Bismuth was detected in ProRoot MTA and Endocem MTA, while zirconium was identified exclusively in Endocem Zr. Minor elements including aluminum, magnesium, sodium, potassium, and iron were also observed. FTIR spectra revealed absorption bands related to Bi–O, Zr–O, Si–O–Si, Si–O–Ca, Ca–O, C–O, and Al–O bonds.
Conclusion: Material-dependent microstructural and chemical differences were observed among pozzolan-based MTAs. Endocem Zr may be preferable in esthetically demanding cases, whereas ProRoot MTA may be favored when enhanced mechanical and physical properties are required.

Pozzolan Bazlı MTA'lar ile ProRoot MTA'nın SEM, EDS ve FTIR Kullanılarak Mikroyapısal ve Kimyasal Olarak Karşılaştırmalı Değerlendirilmesi

Makale Bilgisi

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Anahtar Kelimeler:
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Taramalı elektron mikroskobu.

ÖZET

Amaç: Bu çalışmada, üç farklı Mineral Trioksit Agregatının (MTA) mikroyapısal ve kimyasal özellikleri, taramalı elektron mikroskobu (SEM), enerji dağılımlı X-ışını spektroskopisi (EDS) ve Fourier dönüşümlü kızılötesi spektroskopi (FTIR) yöntemleri kullanılarak değerlendirilmiştir.
Gereç ve Yöntem: ProRoot MTA, Endocem MTA ve Endocem Zr incelenmiştir. Numuneler silikon kalıplarda hazırlanmış, 37 °C'de 24 saat sertleşmeye bırakılmış, toz haline getirilmiş ve %100 nemli ortamda 37 °C'de 72 saat bekletilmiştir. SEM analizleri ×1000–×20,000 büyütmelerde yapılmış, her örnekten rastgele seçilen iki bölgeden EDS spektrumları elde edilmiştir. FTIR analizleri 400–4400 cm^{-1} aralığında gerçekleştirilmiştir.
Bulgular: SEM analizleri, materyaller arasında belirgin yüzey morfolojisi farklılıkları olduğunu göstermiştir. ProRoot MTA küçük ve homojen partiküller sergilerken, Endocem MTA daha büyük ve düzensiz partiküllerle heterojen bir yapı göstermiştir. Endocem Zr ise orta düzey partikül boyutlarıyla daha düzgün ve homojen bir yüzey morfolojisi sunmuştur. Tüm gruplarda kalsiyum, silisyum ve oksijen tespit edilmiş; ProRoot ve Endocem MTA'da bizmut, Endocem Zr'de ise zirkonyum belirlenmiştir. Ayrıca alüminyum, magnezyum, sodyum, potasyum ve demir gibi eser elementlere rastlanmıştır. FTIR analizlerinde Bi–O, Zr–O, Si–O–Si, Si–O–Ca, Ca–O, C–O ve Al–O bağlarına ait karakteristik piklerde materyaller arasında farklılıklar gözlenmiştir.
Sonuç: Pozzolan esaslı MTA'lar arasında belirgin mikroyapısal ve kimyasal farklılıklar bulunmaktadır. Endocem Zr estetik gereksinimlerin ön planda olduğu durumlarda tercih edilebilirken, ProRoot MTA mekanik ve fiziksel özelliklerin önemli olduğu klinik uygulamalarda avantaj sağlayabilir.

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INTRODUCTION

Mineral trioxide aggregate (MTA) has long been considered a gold standard material in endodontics due to its superior biocompatibility, sealing ability, and bioactivity.[1,2] MTA has been widely employed in various clinical applications such as root-end filling, pulp capping, and apexification, owing to its favorable physicochemical characteristics and ability to promote hard tissue formation.[3,4] Among commercially available MTAs, ProRoot MTA (Dentsply Tulsa Dental, USA) is extensively studied and serves as a benchmark against which newly developed calcium silicate-based cements are evaluated.[2,3]

The core components of calcium silicate-based cements typically include tricalcium silicate and dicalcium silicate, which undergo hydration to form calcium silicate hydrate (C-S-H) and portlandite, responsible for the material's strength and alkalinity.[5] Radiopacifiers such as bismuth oxide or zirconium oxide are incorporated to enhance radiographic visibility, though their impact on hydration kinetics and material properties remains a topic of ongoing investigation.[5-6] For instance while bismuth oxide used in ProRoot MTA has been reported to potentially interfere with hydration reactions, zirconium-based additives as in Endocem Zr (Maruchi, Korea) have been proposed as alternatives to improve biocompatibility and reduce discoloration risks.[5,7]

Recent developments have focused on modifying the physical and chemical properties of MTA to overcome certain limitations, including long setting times and handling difficulties.[2,6,8] Materials like Endocem MTA (Maruchi, Korea) and Endocem Zr have been introduced with altered compositions and particle morphologies aimed at enhancing setting kinetics, reducing cytotoxicity, and improving handling characteristics.[7,9,10] However, a comprehensive understanding of how these compositional changes affect the microstructure and elemental profile of the materials is still evolving.

Advanced analytical techniques such as scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and Fourier transform infrared spectroscopy (FTIR) are essential tools for characterizing the morphological and chemical features of calcium silicate-based cements.[6-7-11] SEM provides insight into particle size and surface homogeneity, EDS allows elemental quantification, and FTIR detects functional groups involved in the setting reactions, offering a holistic view of material behavior at the microstructural level.[6-7-11]

The aim of this study was to evaluate and compare the physical and chemical properties, surface morphology, and elemental composition of three commercially available MTAs (ProRoot MTA, Endocem MTA, and Endocem Zr) using SEM, EDS, and FTIR techniques. By comparing these materials with the well-established ProRoot MTA, this investigation seeks to provide a deeper understanding of how formulation modifications influence the microstructural integrity and chemical profile of calcium silicate-based cements.

MATERIALS AND METHODS

Materials

Three commercially available calcium silicate-based cements were used in this study: ProRoot MTA, Endocem MTA and Endocem Zr.

Sample preparation

All materials were handled under aseptic conditions. Each cement was mixed according to the manufacturer's instructions using sterile glass slabs and spatulas. The mixed materials were placed into sterile cylindrical silicone molds (5 mm diameter × 2 mm height) and covered with moist gauze. The samples were allowed to set for 24 hours at 37 °C temperature. After initial setting, the samples were removed from the molds and pulverized using a sterile porcelain mortar and pestle until a fine powder

was obtained. To ensure complete chemical maturation, the powdered samples were incubated at 100% relative humidity and 37 °C for 72 hours in a sealed environment.

SEM and EDS analysis

The microstructural morphology and elemental composition of the materials were examined using a SEM (Thermo Fisher Scientific Quattro S, Czech Republic) equipped with EDS. The specimens were placed on carbon conductive adhesive tape. SEM imaging was performed using secondary electron (SE) mode at 10 or 20 kV accelerating voltage, 9–14 mm working distance, and magnifications of ×1000. ×5000. ×10.000. and ×20.000. Particle size was assessed from micrographs using image analysis software, which was calibrated according to the scale bars provided. For EDS analysis, spectra were collected from two randomly selected regions of each sample to evaluate reproducibility and elemental distribution. Particle size measurements from SEM images were performed using ImageJ/Fiji (NIH, USA) image analysis software. At least 30 individual particles were analyzed per sample on images acquired at 5.000× magnification containing a 10 µm scale bar, and the obtained data were used to generate histograms. The results are expressed as mean ± standard deviation (SD). Particles were categorized as "small" (<2 µm) or "large" (>2 µm) in accordance with dentinal tubule penetration thresholds.[12]

FTIR analysis

Fourier transform infrared (FTIR) spectroscopy was employed to characterize the chemical composition of the samples. FTIR spectra of ProRoot MTA, Endocem MTA, and Endocem Zr were obtained using a Shimadzu IRSpirit-T model spectrometer, operating within the wavenumber range of 400–4400 cm.[1] For analysis, specimens were directly placed onto the ATR crystal without further preparation. FTIR is a widely used vibrational spectroscopic technique that enables the identification of both organic and inorganic components based on their characteristic absorbance peaks. Based on the compositional data reported in previous studies, a summary table was prepared (Table 1) listing the expected compounds present in ProRoot MTA,[13] Endocem MTA,[14] and Endocem Zr.[15] FTIR analysis was then conducted to verify the presence of these compounds by identifying their corresponding characteristic peaks in the spectra.

Table 1: Chemical Compounds in MTA-Based Cements

Material	Reported Chemical Compounds
ProRoot MTA	Calcium silicate (Tricalcium silicate, Dicalcium silicate), Calcium hydroxide, Bismuth oxide, Silicon dioxide, Aluminum oxide, Magnesium oxide, Calcium carbonate, Calcium oxide.
Endocem MTA	Calcium silicate (Tricalcium silicate, Dicalcium silicate), Calcium hydroxide, Bismuth oxide, Calcium carbonate, Silicon dioxide, Aluminum oxide, Magnesium oxide, Calcium oxide.
Endocem Zr	Calcium silicate (Tricalcium silicate, Dicalcium silicate), Calcium hydroxide, Zirconium oxide, Calcium carbonate, Silicon dioxide, Aluminum oxide, Calcium oxide.

RESULTS

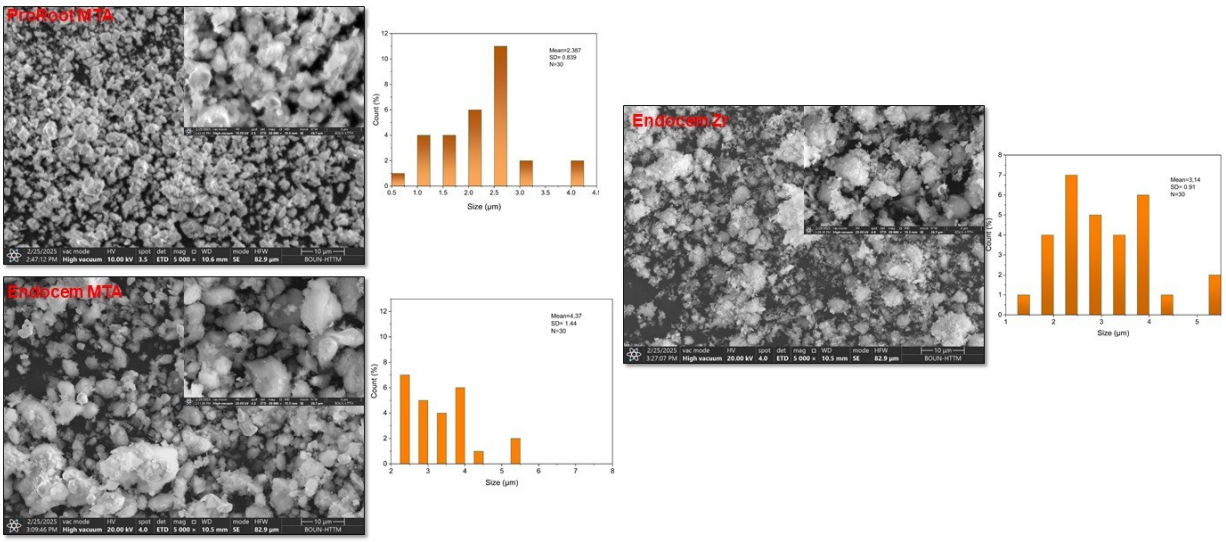
SEM Analysis

Particle Size Distribution

The morphological characteristics of the materials were compared based on the SEM micrographs, which display representative surface structures of each material. Comparative analysis demonstrated notable variations in particle size and distribution homogeneity among the investigated materials. ProRoot MTA exhibited the smallest and most homogeneous particles, with sizes ranging from 0.94 to 4.43 µm, corresponding to a fine and uniform microstructure. In contrast, Endocem MTA showed larger particles, ranging from 2.35 to 7.57 µm, with a heterogeneous and irregular morphology. Endocem Zr demonstrated an intermediate particle size distribution between 1.41 and 5.11 µm and a smoother, more homogeneous surface.

Quantitative histograms based on 30 randomly selected particles per material, together with representative SEM images and the corresponding mean ± SD values, are presented in Figure 1.

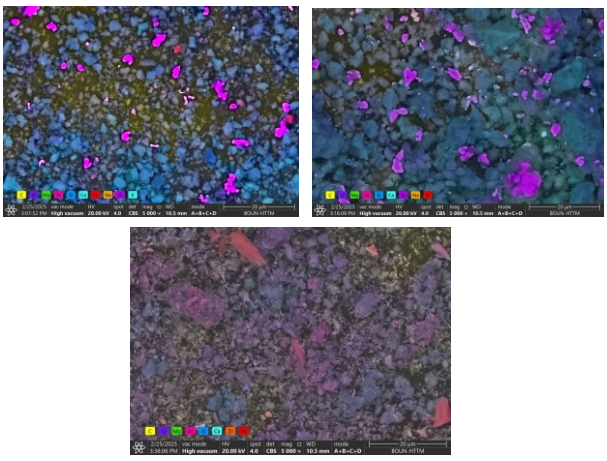
Figure 1. Representative SEM images and corresponding particle size distribution histograms of the tested calcium silicate–based materials.



EDS Analysis

Energy-dispersive X-ray spectroscopy (EDS) analysis of ProRoot MTA samples revealed calcium (Ca), silicon (Si), oxygen (O), and prominent bismuth (Bi) peaks, confirming the presence of bismuth oxide (Bi_2O_3) used for radiopacity. Elemental maps obtained from different areas showed consistent distribution, indicating homogeneity across the surface. In Endocem MTA, calcium (Ca), silicon (Si), oxygen (O), and bismuth (Bi) were also detected, suggesting a similar radiopacifier composition to ProRoot MTA. EDS spectra of Endocem Zr demonstrated the presence of calcium (Ca), silicon (Si), oxygen (O), and a significant zirconium (Zr) peak, consistent with the use of zirconium oxide instead of bismuth oxide for radiopacity. In all groups, trace elements such as aluminum (Al), magnesium (Mg), iron (Fe), sodium (Na), and potassium (K) were identified. The elemental composition of the three materials, as revealed by colored SEM-EDS imaging, is presented in Figure 2.

Figure 2. Elemental composition of the three materials, as visualized by colorized SEM-EDS analysis at ×5000 magnification: (A) ProRoot MTA, (B) Endocem MTA, (C) Endocem Zr.



FTIR analysis

According to the FTIR spectrum of ProRoot White MTA, the shoulder observed at 1453 cm^{-1} corresponds to the Bi–O bond, while the peak at 1413 cm^{-1} is associated with the asymmetric C–O stretching band within the calcium silicate structure. The band at 873 cm^{-1} is related to Ca–O bonds and carbonate vibrational modes. A distinct asymmetric Si–O–Si stretching band is observed at 1111 cm^{-1}, whereas the peak at 922 cm^{-1} corresponds to Si–

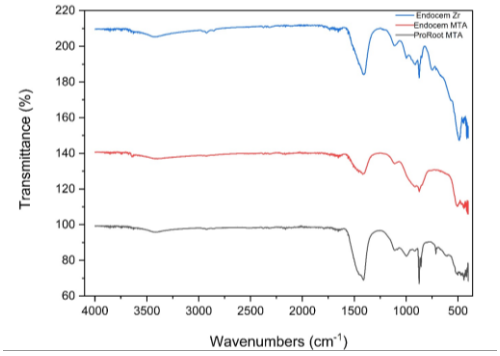
O–Ca bonds. The band at 997 cm^{-1} is attributed to Al–O stretching vibrations in the aluminate structure. The peak at 512 cm^{-1} corresponds to Bi–O bond vibrations.

In the FTIR spectrum of Endocem MTA, the peak at 1411 cm^{-1} corresponds to the asymmetric C–O stretching band. The Si–O–Si stretching vibration is observed at 1109 cm^{-1}, followed by a peak at 919 cm^{-1} corresponding to the Si–O–Ca bond. The band at 872 cm^{-1} is attributed to Ca–O and carbonate vibrational modes. The peak at 512 cm^{-1}, consistent with the ProRoot spectrum, also corresponds to Bi–O bond vibrations.

In the case of Endocem Zr, the peak at 1406 cm^{-1} is assigned to the asymmetric C–O stretching band, and the Si–O–Si stretching band is again observed at 1109 cm^{-1}. The band at 1000 cm^{-1} is attributed to Al–O stretching vibrations. A peak at 916 cm^{-1} corresponds to Si–O–Ca bonding, while Ca–O vibrations are evident at 873 cm^{-1}. Notably, a distinct peak at 745 cm^{-1} corresponds to Zr–O vibrations, and the strong band at 489 cm^{-1} indicates Zr–O bond presence as well.

The FTIR spectra of the three materials are presented together in Figure 3.

Figure 3. FTIR spectra of the three MTA-based materials presented for comparative analysis.



DISCUSSION

Despite its favorable properties, the extended setting time of MTA continues to pose a challenge for clinical applications.[16] Despite this limitation, ProRoot MTA, due to its biocompatibility, sealing ability, and capacity to promote tissue healing, is widely recognized as the gold standard among endodontic materials.[4] To address limitations such as prolonged setting time and handling difficulty, alternative formulations like pozzolan based cements have been developed.[17] Its composition primarily includes tricalcium silicate and dicalcium silicate, with bismuth oxide (Bi_2O_3) incorporated as the radiopacifying agent.[5] Endocem MTA is a fast-setting MTA-based cement with a modified particle size and formulation aimed at improving handling properties and reducing setting time.[17] Like ProRoot MTA, it also utilizes bismuth oxide for radiopacity.[5] Endocem Zr is a newer generation cement designed to overcome limitations associated with bismuth oxide, particularly tooth discoloration.[17] In this formulation, zirconium oxide (ZrO_2) is employed as the radiopacifier to enhance both esthetic stability and biocompatibility.[7]

The FTIR spectra exhibited characteristic absorption bands corresponding to Si–O, C–O, and O–H vibrations, confirming the formation of calcium silicate hydrate, calcium hydroxide, and other hydration products.[18] These findings indicate ongoing hydration reactions and the coexistence of amorphous and crystalline phases within the materials.[11]

The presence of trace elements like Al, Mg, Fe, Na, and K is likely related to the manufacturing process and generally does not significantly affect the material's overall physical properties.[2] The absorption bands near 512 cm^{-1} (Bi–O) in ProRoot White MTA and Endocem MTA, and at 745 cm^{-1} and 489 cm^{-1} (Zr–O) in Endocem Zr, confirm the incorporation of bismuth and zirconium oxides as radiopacifiers, consistent with previous reports describing the use of metal oxides to enhance radiopacity in calcium silicate–based cements.[5,17]

The fine and homogeneous surface morphology observed in ProRoot MTA represents a distinctive characteristic of this formulation, reflecting its uniform particle distribution and minimal aggregation. The presence of smaller particles, frequently below

2 µm, may contribute to enhanced penetration into dentinal tubules and improved surface adaptation. In contrast, Endocem MTA exhibited a coarser and more heterogeneous surface with pronounced particle agglomeration, which may limit its mechanical interlocking with dentin. The inclusion of zirconium oxide in Endocem Zr resulted in an intermediate microstructure with moderately uniform particle dispersion and surface smoothness.[17,18] Such morphological refinement in ProRoot MTA may facilitate superior handling properties, improved adaptation to cavity walls, and more effective dentinal tubule interaction.[18]

Although bismuth was clearly detected in the EDS analysis, the absence of a distinct Bi_2O_3 peak in the FTIR spectrum, observed only as a shoulder, may be attributed to the possible reaction of bismuth with the liquid component of the MTA during setting.[5]

Microstructural characteristics have been shown to be closely associated with the clinical performance of calcium silicate–based materials. A more homogeneous particle distribution and well-defined crystalline structure may enhance the sealing capacity by ensuring closer contact with the dentin surface, and calcium silicate–based sealers also achieve satisfactory long-term sealing through penetration into dentinal tubules.[19] In addition to microstructural features, clinical success also depends on handling properties. For instance, the correlation between flow characteristics and sealing ability in calcium silicate–based sealers has been demonstrated, and this finding is consistent with the general understanding that structural parameters such as particle size and surface homogeneity directly influence clinical manipulation and adaptation.[20]

From a biological perspective, resin-free calcium silicate–based sealers have been shown to induce a low inflammatory response and exhibit osteoconductive properties [21] and a close relationship between their physicochemical characteristics and cellular responses has also been demonstrated.[22] Taken together, these findings indicate that calcium silicate–based materials may provide biocompatibility advantages in clinical applications. With respect to aesthetics, long-term follow-up has shown that calcium silicate–based materials do not cause clinically significant discoloration.[23] Moreover, the dominance of Zr–O bands identified by FTIR analyses has been associated with zirconium oxide supplementation, which appears to confer an additional benefit in color stability compared with bismuth oxide–containing formulations.[23]

One of the primary limitations of this study is the restricted number of materials evaluated, as only three calcium silicate-based materials were included in the analysis. Additionally, all experiments were conducted under laboratory conditions, which may not fully replicate the complexities of the clinical environment. Another limitation is that only the set forms of the materials were analyzed; the unreacted powder forms were not included in the analysis. Including both set and unhydrated forms might have provided a more comprehensive understanding of the hydration behavior, as the release of calcium hydroxide could vary depending on the reaction stage of the material.[11,24] The particle size analysis was descriptive in nature and not supported by full quantitative data; hence, statistical comparison between materials was not feasible. Furthermore, no dentin–material interface was observed because no tooth substrate was used in this study. Since smaller and more homogeneous particles may positively influence the sealing ability of calcium silicate–based cements,[19,20] such interfacial properties could not be evaluated within the scope of this investigation. In addition, radiopacifying agents such as zirconium oxide and bismuth oxide have been associated with differences in discoloration potential,[23] suggesting that future research integrating optical, biological, and mechanical analyses would provide a more comprehensive understanding of these materials' clinical behavior. Therefore, further investigations combining microstructural characterization with biological and mechanical testing are recommended.

This study focused on the comparative analysis of pozzolan-based MTAs, Endocem MTA and Endocem Zr, with white ProRoot MTA using SEM-EDS and FTIR techniques. According to the results, the elemental and structural characteristics observed in the set form of ProRoot MTA were consistent with findings reported in previous SEM-EDS,[24] and FTIR [5,6] studies. To the best of our knowledge, there is a lack of published data focusing on the microstructural and chemical properties of pozzolan-based MTAs using these specific analytical techniques. This study contributes novel insights by providing a detailed characterization of Endocem MTA and Endocem Zr in this context.

CONCLUSIONS

Given its zirconium-based composition and enhanced microstructural uniformity, Endocem Zr may represent a suitable alternative in cases with high esthetic demands, as the replacement of bismuth oxide with zirconium oxide minimizes discoloration potential and promotes a more stable microstructure. In contrast, the smaller and more homogeneous particle distribution observed in ProRoot MTA may confer superior mechanical integrity and physical performance, contributing to improved handling characteristics and potentially enhanced clinical durability.

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Ethical Approval

Ethical committee approval was not required for our study. An ethics declaration form has been completed.

Financial Support

The authors declare that this study received no financial support.

Conflict of Interest

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

Author Contributions

Design: AK, Data Collection or Access: AK, EIA, Analysis and Comments: AK, DM, Literature Search: AK, EIA, Writing: AK, EIA, DM.

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