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THE EFFECT OF HEAT ON BOND STRENGTH AND BIOACTIVITY OF CALCIUM SILICATE-BASED SEALERS. A NARRATIVE REVIEW

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INTRODUCTION

Root canal obturation primarily aims to prevent microbial recontamination following canal disinfection. This is accomplished through the use of gutta-percha in combination with sealing agents, which collectively create a seal that prevents the penetration of bacteria and their harmful by-products. Successful root canal obturation focuses on maximizing the use of gutta-percha and minimizing the amount of sealer applied. Ideally, the sealer should remain confined to the periphery of the gutta-percha cone, thereby enhancing the apical seal, trapping residual microorganisms, and inhibiting their movement toward the periapical region.

An ideal endodontic sealer is expected to exhibit biocompatibility with periapical tissues, demonstrate a prolonged working time, set without dimensional changes, possess antimicrobial properties, avoid discoloration, be soluble in commonly used solvents when necessary, and remain stable when exposed to oral and tissue fluids.

A range of sealing materials has been utilized in endodontics, such as zinc oxide eugenol-based, calcium hydroxide-based, glass ionomer-based, and resin-based sealers. Of these, epoxy resin-based sealers are recognized for their favorable handling characteristics and robust physical performance, making them a frequent choice for comparative studies.

Calcium silicate-based sealers have recently become increasingly prominent in endodontic practice owing to their superior biological compatibility and sealing effectiveness. These materials often include components like alumina, zirconia, hydroxyapatite, bioactive glass, and calcium phosphates, contributing to their bioactivity and suitability for tissue regeneration applications.

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The durability of the sealer-dentin interface is crucial, especially under mechanical stresses like post-space preparation or functional loading. Therefore, the sealer's ability to adhere to root dentin is crucial for ensuring the sustained success of endodontic treatment.

Besides the selection of sealer, the technique used for canal obturation significantly influences the sealing quality. Techniques such as warm vertical compaction, hydraulic condensation, and cold lateral compaction aim to achieve three-dimensional filling of the canal system.

Bioactivity is a key characteristic of certain sealers, particularly calcium silicate-based materials, which can form hydroxyapatite upon interaction with physiological fluids. This reaction enhances the integration of the material with surrounding dentin, contributing to biological sealing.

Although various investigations have explored the bioactive behavior of calcium silicate sealers, there remains a lack of evidence regarding how different obturation methods influence their performance, particularly in comparison to resin-based alternatives.

Although numerous studies have evaluated the bond strength of calcium silicate-based sealers, limited information exists on how different obturation techniques influence their adhesion compared to conventional resin-based sealers.

Therefore, this study aims to examine the effect of heat application on both the bond strength and bioactive properties of Neosealer.

REVIEW OF LITERATURE

Completely eliminating the microbiological infection, preventing additional reinfection, and curing apical periodontitis are the objectives of the root canal procedure.

To achieve this goal, effective sealing is essential to inhibit bacterial growth. Kakehashi (1965) demonstrated that microorganisms and their byproducts are the primary cause of apical periodontitis. Therefore, successful treatment largely depends on eliminating these microorganisms and their toxic byproducts.[1]

To provide a hermetic seal and fluid-tight barrier against microorganisms in the oral cavity, the obturation stage of the treatment involves filling the root canal.

An effectively obturated system should fulfill all three of these primary functions.[2]

1. It must inhibit coronal leakage and block the ingress of nutrients that could facilitate bacterial survival and proliferation.
2. Prevent fluids from periapical or periodontal tissue.
3. Prevent the proliferation of bacteria that survive from debridement and disinfection stage by their entombment.

Gutta-percha has long been the primary core material for root canal obturation; however, it lacks adhesive properties to dentin and cannot effectively penetrate or seal the dentinal tubules. This limitation is addressed through the root canal sealers, which occupy the spaces between the gutta-percha and the canal walls, as well as the voids between the master cone and accessory cones. It fills irregularities, anatomical complexities, lateral canals, and dentinal tubules.[3]

In 1958, Grossman suggested the ideal properties of root canal sealer.[4]

1. Able to create a completely airtight seal.
2. Maintains dimensional stability upon setting, with slight expansion being advantageous.
3. Does not lead to tooth discoloration.
4. Possesses a prolonged setting time.
5. Does not dissolve when exposed to bodily fluids.
6. Acts to reduce or eliminate microbial presence.

7. Biocompatible with periapical tissues.
8. Demonstrates tackiness when mixed, ensuring adequate adhesion to the canal walls upon setting.
9. Consists of a fine powder that mixes easily with liquid and is soluble in common solvents to allow for retreatment if necessary.

Numerous methods are available for obturation of the root canal system. Among these methods, the cold lateral compaction (CLC) technique is commonly employed and is frequently regarded as the conventional standard. It is relatively straightforward, cost-effective, and allows for good control at the apical end. However, this method has certain limitations, such as the formation of spreader tracts, poor integration of gutta-percha cones, insufficient adaptation to the canal walls, potential inconsistency in the density of the filling, and an inability to adequately seal irregularities within the canal.[5]

Introduced by Schilder in 1967[6], the Warm Vertical Compaction technique (WVC) allows thermo-plasticized gutta-percha to be used to fill internal resorption defects and accessory apical anatomy. The main challenge with this technique is controlling the apical extrusion of the softened gutta-percha.[7]

To improve the seal, stop microleakage, and fill in irregularities within the root canal system, the obturation technique selection is crucial. Obturation technique has been shown to be an efficient way to control sealer thickness, which in turn influences bond strength, fracture resistance and the rate of sealer penetration into dentinal tubules.[8]

Calcium silicate-based materials in endodontics

Epoxy resin-based sealers are frequently utilized in clinical settings as root canal sealers.Schroeder created the AH series sealer in 1957; it had excellent physical characteristics and sealing ability. The issue of AH 26 producing formaldehyde during setting was resolved by AH Plus. Because of its dimensional stability and resistance to resorption, AH Plus has been regarded in numerous studies as the gold standard sealer.[9] On the other hand, it has negative effects such as cytotoxicity, mutagenicity, and inflammatory response. Additionally, because of its hydrophobicity and retained dental moisture, AH Plus may have flaws in its adhesion to the canal walls.[10]

The development of bio-ceramic materials in recent years has signaled the start of a new era in endodontics. iRoot SP, the first sealer based on calcium silicate, was introduced in 2007. Its superior physical qualities make it appealing to dentistry. Since then, the market has seen the introduction of numerous Calcium silicate-based sealers that are radio-opaque, biocompatible, osteoconductive, capable of achieving a superior hermetic seal, insoluble, and can be easily handled.[11] They exhibit a slight expansion upon setting and are dimensionally stable.[12] Because of their thin film thickness, small particle sizes (less than 2 micrometers), high flowability, and hydrophilicity, which contribute to their better penetration effect, bio-ceramics offer exceptional chemo-physical qualities.[13]

The material contains antibacterial properties before setting, and it becomes biocompatible and even bioactive after setting. Through the process of hydration, they produce calcium hydroxide, which then reacts with tissue phosphate to generate hydroxyapatite.[14] Glass ceramics, hydroxyapatites, zirconia, bioactive glass, and calcium phosphate are all components of bio-ceramics. Based on how they interact with the surrounding tissue, Calcium silicate-based materials have been categorized as either bioactive or bioinert. Bioactive substances like glass and calcium phosphate promote the growth of surrounding tissues. Materials that are bioinert, such as zirconia and alumina, have no physiological or biological effects.[15]

Calcium silicate-based sealer used in endodontics are either calcium silicate based or calcium phosphates-tricalcium phosphate-hydroxyapatite based.[16]

It is uncertain how the bio-ceramic-based sealer bonds to root canal dentin.

Nevertheless, the following concepts have been suggested:

1. The sealer achieves mechanical interlocking by infiltrating the dentinal tubules.
2. Mineral infiltration of the sealer into intertubular dentin after collagen fiber denaturation by an alkaline sealer.
3. The creation of hydroxyapatite when phosphate reacts with calcium hydroxide and calcium silicate hydrogel in the presence of moisture.

LeGeros et al.[17] was the pioneer in utilizing bio-ceramic materials as dental restorative cement.. Bio-ceramic was first applied as a root canal sealer by Krell and Wefel[18] two years later. When they examined Grossman's sealer and calcium phosphate sealer in extracted teeth, they discovered that while there was no discernible difference in dentinal adhesion or adaptation, calcium phosphate sealer was not as successful in sealing apically as Grossman's sealer. Both premixed (iRoot, Ceraseal, Endoseal MTA) and powder-liquid (Bioroot RCS, Proroot ES) forms of bio-ceramic sealers are available. Mixing of powder and the liquid system is technique sensitive and time consuming. However, premixed sealer, paste, and putty provide uniform consistency and minimal waste they are all hydrophilic.[19]

NeoSealer is a premixed, calcium silicate-based bioceramic sealer composed of calcium silicates, zirconium oxide, and a thickening agent. It features a high pH of 12.73, a setting time of up to 3.5 hours, and a radiopacity below 8 mm. The material demonstrates substantial calcium ion release, along with excellent cell viability and a high rate of cellular migration.

Push out bond strength of the bio-ceramic sealer.

Grossman emphasized that a fundamental property of root canal filling materials is their capacity to adhere to radicular dentin. Strong adhesion helps prevent fluid leakage Along the junction between the filling and the canal walls. The gutta-percha material used for obturation helps maintain the sealer-dentin bond under both static and dynamic conditions. It ensures close adaptation of the obturation material to the root canal dentin, which helps prevent fluid leakage when the tooth is at rest. Additionally, it withstands mechanical forces, such as those from tooth flexure or post-space preparation, which could otherwise dislodge the filling material.[21] Various methods such as the push-out test, micro-tensile bond strength test, and shear bond strength test are commonly employed to evaluate the bond between root canal sealers and the radicular dentin. . Among these, push-out test is particularly valued for its ability to simulate clinical shear stresses at the dentin-sealer interface. It provides more consistent stress distribution and lower variability compared to the other tests. Additionally, it allows for reliable assessment even when bond strength values are minimal, and its simplicity makes it suitable for repeated testing without the need for advanced equipment. A key benefit of this method is its capacity to evaluate bond strength in standardized 1-mm-thick root slices at different canal levels.[22]

The composition, properties, and intended uses of a root canal sealer are crucial factors in choosing the most suitable one for a specific clinical situation. Both bio-ceramic sealers and epoxy resin-based sealers offer strong adhesion to the dentinal surface.[23]

The effect of sealer type on push-out bond strength

Numerous studies have demonstrated that the chemical covalent link that forms between the open epoxide rings in the sealer and the exposed amino groups in the dentin collagen matrix gives AH + sealer a high resistance to dislodgment.[24]

To compare the push-out bond strength in root canals, Mokhtari et al.[25]used two distinct obturation techniques—single-cone and cold lateral compaction using gutta-percha with AH plus sealer. In comparison to the single-cone method, they discovered that the cold lateral compaction technique produced a higher bond strength. Furthermore, Gade et al. carried out a study comparing the push-out bond strength of EndoSequence BC Sealer with that of AH Plus, using two different obturation techniques: thermoplasticized and cold lateral compaction. Their results showed that the greatest bond strength was obtained when AH Plus sealer was applied using the cold lateral condensation technique.[26]

Nevertheless, in 2019, In a systemic review, Endosequence BC sealer's push-out bond strength was greater than AH Plus sealer's when the thermoplasticized approach was applied. They examined the push-out bond strength of root canal sealers calcium silicate and epoxy resin. The analysis concluded that epoxy resin sealers often showed higher push-out bonds than paste-to-paste and premixed calcium silicate.[27]

In studies evaluating the effect of different irrigation protocols on the push-out bond strength of various sealers—including epoxy resin-based, calcium silicate-based, and silicone-based types—it was reported that the use of EDTA followed by NaOCl enhanced the bonding performance of epoxy resin-based sealers, which demonstrated superior bond strength values compared to the other tested materials.[28] The same was true for Celik et al., who looked into how various irrigant combinations used in final irrigation affected the root canal sealers' push-out bond strength, such as AH Plus, Tech Biosealer Endo, and Endoseal MTA. They found that AH Plus benefited from the final irrigation protocol and had higher push-out bond strength values.[29]

Silva et al.'s study aimed to evaluate the bond strength of EndoSeal MTA, a newly developed injectable pozzolan-based root canal sealer, in comparison to MTA Fillapex and AH Plus when bonded to root dentin. AH Plus demonstrated a significantly higher resistance to dislodgment than other sealers. EndoSeal has also demonstrated Acceptable bond strength for use in endodontic therapy, showing better performance when compared to MTA Fillapex.[30]

A comparative analysis was conducted between polydimethylsiloxane-based sealers, such as GuttaFlow 2 and GuttaFlow Bioseal, and the epoxy resin-based sealer AH Plus, to assess differences in their push-out bond strength. The push-out bond strength was measured using a universal testing machine. It was concluded that AH plus exhibited the highest push-out bond strength among the tested sealers. .[31]

Conversely, bio-ceramic sealers can slightly expand when exposed to the natural moisture found in dentinal tubules, which results in the production of calcium hydrogel and calcium hydroxide. This chemical bonding to mineralized tissue on the dentin-sealer layer gives the bio-ceramic sealer a high bond strength.[32] According to a meta-analysis study, calcium silicate-based sealers are either inferior to traditional resin-based sealers or comparable with them.[33] In addition, push-out tests were conducted to assess the bond strength of two types of endodontic sealers. Bio-ceramic and methacrylate resin-based sealers. The findings showed that, in comparison to the other groups, endoseal MTA and gutta-percha showed noticeably stronger resistance to dislodgement. In comparison to RealSeal SE and Resilon, EndoSequence BC sealer and BC Point also demonstrated noticeably higher push-out bond strength values.[34]

The push-out bond strength of different obturation systems was evaluated, including conventional gutta-percha with AH Plus, the Epiphany/Resilon system, and Smart Seal C-Point with a bioceramic sealer. Results showed that the C-Point with bioceramic sealer exhibited the highest bond strength, followed by gutta-percha with AH Plus, and lastly the Epiphany/Resilon system.[35]

In a study by Pawar et al., the push-out bond strength of root canals filled using either the traditional gutta-percha with AH Plus or C-Point with EndoSequence BC sealer was evaluated after preparing oval root canals .Cold lateral compaction was used for obturation in all groups. The results demonstrated that the highest bond strength was achieved in canal filled with C-Point and BC sealer, whereas the lowest values were observed in canals obturated using gutta-percha and AH Plus.[36]

A universal testing machine was employed to assess the push-out bond strength of AH Plus in comparison with two recently developed calcium silicate-based sealers: iRoot SP and MTA Fillapex. The findings revealed that both AH Plus and iRoot SP exhibited greater bond strength values than MTA Fillapex.[37]

The effect of the obturation technique on push-out bond strength.

The bond strength of calcium silicate-based sealers has been shown to vary depending on the obturation technique used. Although manufacturers generally recommend the single-cone technique, the use of heat in procedures such as warm vertical compaction may affect the physical and chemical properties of the sealer.Exposure to heat can enhance hydration and promote earlier hydroxyapatite formation. However, a reduction in setting time may compromise the material's flow characteristics, ultimately leading to decreased adhesion to root canal dentin.[38]

Concerning how the obturation method affects the bio-ceramic sealer's push-out bond, the push-out bond strength of several sealers and obturation systems was compared by Yap et al.[39] at two weeks and three months after obturation. The TotalFill BC obturation system and TotalFill BC sealer combined with gutta-percha demonstrated bond strength comparable to that of AH Plus/gutta-percha, with the bond strength increasing over time. . In contrast, the push-out bond strength of the EndoREZ sealer and EndoREZ obturation system was low and gradually reduced. Additionally, the push-out bond strength of two endodontic obturation materials—Epiphany/Resilon and gutta-percha/AH Plus—was assessed by Element Obturation unit. The study concluded that Resilon/Epiphany self-etch did not produce stronger bond strength than AH Plus/gutta-percha.[40]

Several studies have explored how different obturation techniques influence the push-out bond strength of AH Plus sealer and the premixed bioceramic sealer TotalFill BC when applied to root canal dentin. Results indicated that TotalFill BC exhibited superior bond strength compared to AH Plus. Additionally, while the performance of AH Plus was notably affected by the obturation method used, TotalFill BC appeared to maintain consistent bonding regardless of the technique.[41]

Furthermore, Nouroloyouni et al. used a universal testing machine to examine the push-out bond strength of cold lateral compaction and single cone obturation with AH-Plus and Sure Seal Root. Compared to AH-Plus, the lateral compaction/Sure Seal group showed the highest push-out bond strength and the fewest voids.[42]

In a study by Retana-Lobo et al., the push-out bond strength was evaluated in canals obturated either with gutta-percha and sealer using the single-cone technique or with sealer alone. The findings revealed that bioceramic sealers demonstrated enhanced bond strength when used without a gutta-percha core, suggesting that sealer-only obturation may offer improved adhesion in such materials.[43]

Numerous research investigations examined the impact of heat used during the warm vertical compaction obturation technique on the bond strength of calcium silicate-based sealers. The push-out bond strength of three different obturation systems was tested : gutta-percha combined with AH Plus using the lateral condensation technique, C-Points with a bioceramic sealer applied via the single-cone method, and thermoplasticized gutta-percha (non-carrier based CALAMUS system) backfilled with AH Plus. Among these, the C-Point and bioceramic sealer group exhibited the highest resistance to dislodgement, whereas the gutta-percha with AH Plus using lateral compaction showed the lowest bond strength values.[44]

In a study by Dabaj et al., the influence of obturation technique on the bond strength of two sealer types was explored. The investigation compared a calcium silicate-based sealer (EndoSequence BC Sealer) applied using a thermoplasticized injection method with an epoxy resin-based sealer (AH Plus) placed via cold lateral compaction. The results indicated that the thermoplasticized injectable technique adversely affected bond strength, with a more pronounced impact observed for EndoSequence BC Sealer.[45]

Bioactivity of Calcium Silicate-based bioceramic materials.

Bioactivity refers to a material's capacity to initiate the formation of bone-like apatite and develop a biologically active layer when placed in a physiological environment.[46] The formation of apatite acts as a biological foundation in bone-related clinical applications and may facilitate the regeneration of damaged tissues by promoting new bone formation.[47] Materials capable of initiating the formation of apatite or its precursors on their surfaces may contribute positively to cell differentiation, aiding processes such as tissue healing, bone regeneration, and cementum formation. Bioactivity can be evaluated in vitro by immersing the material in simulated body fluid (SBF) to observe the development of apatite-like layers.[48]

The effect of sealer type on bioactivity

The study by Natale et al. aimed to evaluate the mechanical properties and ion release characteristics of two calcium silicate-based cements, MTA Angelus and Biodentine, as well as a calcium hydroxide-based cement, Dycal.. In addition to evaluating the mechanical qualities, such as compressive strength, elastic modulus, and flexural strength, the ion release was measured at various pH levels. The materials all consistently released calcium over 28 days, according to the results; however, depending on the pH, Dycal released calcium at a much lower rate than Biodentine and MTA Angelus. When compared to MTA Angelus and Dycal, biodentine showed much higher strength and modulus, indicating better stress-bearing properties.[49]

Bioactivity and hydration were evaluated using scanning electron microscopy combined with energy dispersive X-ray spectroscopy (SEM/EDX), comparing a cement composed of 80% tricalcium silicate and 20% zirconium oxide to Biodentine™ and MTA Angelus. All materials tested demonstrated bioactivity, evidenced by the formation of hydroxyapatite on their surfaces after exposure to simulated body fluid.[50]

Moreover, another study evaluated MTA PLUS's ability to generate apatite crystals in comparison to Proroot after 28 days in a solution similar to human fluids at 37°C and 99% humidity. The samples were analyzed with an environmental SEM/EDX. According to the findings, MTA Plus exhibited apatite deposition, demonstrating that it is a bioactive material.[51]

The bioactivity of various calcium-based materials—including calcium hydroxide and several calcium silicate formulations, was examined by evaluating their ability to form apatite in simulated body fluid (SBF). All tested calcium silicate-based materials exhibited substantial calcium ion release, with Tech Biosealer Capping, MTA Plus Gel, and Biodentine releasing the highest concentrations. In contrast, Lime-Lite demonstrated the lowest calcium ion release among the materials evaluated. Morphologically, ProRoot MTA presented with prominent spherulitic crystal deposits, while TheraCal produced smaller, denser formations. These findings highlight the dual nature of calcium silicate-based cements as both bio-interactive (ion-releasing) and bioactive, Indicating their potential to stimulate dentin bridge formation and contribute to improved clinical healing outcomes through rapid apatite deposition and calcium ion availability.[52]

The effect of environmental conditions on sealer behavior

The behavior of EndoSequence BC RRM putty when exposed to blood and simulated body fluid was analyzed using SEM, EDX, and XRD techniques, both before and after contact with water, HBSS, and heparinized whole blood. The results revealed that samples exposed to blood exhibited a poorly crystalline surface structure and showed additional elemental peaks for calcium, phosphorus, and chlorine. These findings imply that the surrounding environment significantly affects the material's hydration behavior.[53]

An experimental study explored the interaction between Biodentine and MTA with root canal dentin in a simulated physiological environment. Dentin sections, each 1.5 mm thick, were prepared from extracted human teeth using Gates-Glidden drills. These segments were randomly allocated to receive either MTA or Biodentine, after which they were immersed in a phosphate-rich solution simulating the composition of blood plasma.

Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray (EDX) analyses were employed to assess the interfacial layer's thickness and the calcium-to-phosphate (Ca/P) ratio. While both materials exhibited bioactivity through the formation of an interfacial layer, MTA produced a notably thicker layer than Biodentine. Amorphous calcium phosphate deposits were observed in the interfacial regions of both groups. Due to its comparable biointeractive behavior, Biodentine may serve as a viable alternative to MTA for root repair procedures.[54]

The calcium phosphate deposition capability of NeoMTA Plus was assessed in Hank's Balanced Salt Solution (HBSS) and compared with that of commercially available MTA. Elemental analysis was performed using SEM-EDX to evaluate ion deposition. Results indicated that NeoMTA Plus released calcium and hydroxyl ions in greater amounts and over a longer duration than MTA Plus, With results indicating a statistically notable difference between the materials. NeoMTA Plus, a newly developed calcium silicate-based root-filling cement, provides sufficient radiopacity along with a prolonged setting time. Its improved ion release profile and capability to form calcium phosphate may contribute to improved root-filling integrity and support both endodontic and periodontal tissue regeneration, reflecting its strong bioactive and biocompatible characteristics.[55] In a comparative study, the potential of NeoMTA Plus and MTA Angelus to promote apatite crystal formation was investigated. Dentin discs containing standardized perforations were filled with either material and incubated in phosphate-buffered saline (PBS) for 15 days. Bioactivity was then evaluated using Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy coupled with Energy Dispersive X-ray (SEM/EDX) analysis. The surface precipitates were assessed for crystallinity and carbonate/phosphate (CO_3/PO_4) ratios via FTIR, while the Ca/P ratios were determined using EDX. Statistical comparisons were made using ANOVA and t-tests. Each of the materials, being tricalcium silicate-based, react to form calcium silicate hydrate. NeoMTA Plus exhibited greater apatite deposition, higher crystallinity, and elevated Ca/P ratios, though it demonstrated a lower CO_3/PO_4 ratio compared to MTA Angelus. SEM analysis revealed that NeoMTA Plus had a globular microstructure with finer particles, whereas MTA Angelus displayed larger, spherical particles. Due to its rapid setting, superior crystallinity, and enhanced bioactivity, NeoMTA Plus shows promise as an effective material for root repair applications.[56]

Tomás-Catalá et al. performed an in vitro study to evaluate the biological behavior of human dental pulp stem cells (hDPSCs) to MTA-Angelus, MTA

Repair HP, and NeoMTA Plus. The assessment involved evaluating cell viability and migration in response to material eluates. Cell morphology and adhesion to the tested materials were analyzed through immunocytofluorescence and scanning electron microscopy, respectively. Furthermore, EDX analysis was carried out to assess the elemental content of each material. The results demonstrated that all three materials influenced key cellular behaviors, including proliferation, morphology, migration, and attachment, indicating their biological activity toward hDPSCs.[57]

Do Carmo et al. evaluated the calcium ion release and apatite-like precipitate formation of MTA, Biodentine, and various calcium aluminate cement formulations shortly thereafter immersion in phosphate-buffered saline (PBS). The study also examined the impact of these materials on bond strength within root-end cavities. Ten cylindrical specimens of MTA, Biodentine, CACb, and a calcium-enriched variant (CACb+) were fabricated using silicon molds and immersed in either PBS or deionized water for 14 days. Surface analyses were performed using SEM, EDS-X, and FTIR. Additionally, eighty standardized root-end cavities were filled with the tested cements (ten per group) and similarly stored for 14 days. All materials demonstrated crystal formation consistent with hydroxyapatite development, confirming their bioactivity following exposure to PBS.[58]

The pH, setting time, and calcium ion release of MTA Angelus, MTA Angelus+ nano-oxides, and an experimental nano-hybrid MTA were assessed by Zanjani et al., Using a variety of methods, specimens of each cement were made and examined. The biocompatibility was evaluated using the MTT test. The cements were poured into polypropylene tubes, and each tube's weight was recorded. Measurements of pH and calcium ion release were made at various intervals during the specimens' incubation in deionized water. Using a Gilmore needle, setting time was ascertained. Atomic absorption spectroscopy, pH meter, XRD, SEM/EDX, and FTIR spectroscopy were all used in the study's investigation. The experimental group had the least setting time, according to the results, whereas MTA Angelus had the longest. Every material has the ability to release calcium and was alkaline. When nanoparticles were added to MTA Angelus, the setting time was greatly reduced without affecting pH or calcium ion release.[59]

Furthermore, the ability of MTA, Endocem-MTA, and NeoMTA Plus to deposit apatite crystals in an acidic or neutral environment was examined using elemental analysis and EDX. On the surface of the cements subjected to butyric acid, prismatic crystalline formations were observed. EDX examination revealed a distinct peak of Cat from NeoMTA-Plus and Endocem-MTA in acidic and physiological conditions. In conclusion, in acidic environments, both materials demonstrated increased calcium release.[60]

Shalabi et al. investigated the effect of blood on the bioactivity and bonding strength of Biodentine when used as a root-end filling material. .Single-rooted teeth that had been cleaned, shaped, and obturated were used in the investigation. Next, the apical third of the root end is removed. Next, a retro-cavity is prepared using a retro trip that is connected to an ultrasonic device, and it is then filled with the material that has been tested. Half of the specimens were immersed in blood, whereas the remaining half were placed in deionized water. To test the ion-releasing capabilities, all samples were immersed in HBSS to simulate body fluids. Apatite formation was monitored at intervals of 1, 7, and 30 days using scanning electron microscopy (SEM) for surface morphology and energy-dispersive X-ray spectroscopy (EDX) for elemental composition. The Ca/P ratios were calculated, and X-ray diffraction (XRD) was employed to identify the crystalline phases. The findings indicated that blood contamination significantly affected apatite deposition on both the surface of the material and at the material–dentin interface. The Ca/P ratios observed in the contaminated samples exceeded those typical of stoichiometric hydroxyapatite. Furthermore, the presence of blood during the setting of Biodentine was associated with reduced apatite nucleation compared to uncontaminated conditions. [61]

The bioactivity of Neo MTA Plus, ProRoot MTA, Biodentine, and glass ionomer cement as root-end filling materials was compared using 1% methylene blue dye penetration as a tracer method. A total of eighty human single-rooted teeth were divided into four groups, with each group receiving a different material. Dye penetration depth was evaluated on days 1 and 7 under a stereomicroscope at 2× magnification to assess the sealing ability and bioactivity of the materials. All tested materials exhibited some level of dye penetration. However, among the groups, Neo MTA Plus and Biodentine demonstrated superior performance, supporting their suitability as effective root-end filling options. [62]

Three commercial calcium silicate-based endodontic materials—MTA, BRRM putty, and BiodentineM—was examined by Talabani et al for their capacity to deposit minerals. Twelve 3 mm-thick discs derived from human single-rooted teeth were obturated with the three tested materials (BiodentineTM, ERRM putty, and MM-MTA) and left in PBS for 7 and 30 days. Morphology, surface deposits, and interfacial dentin were assessed by SEM. When compared to MTA, biodentine and ESRRM putty exhibited much higher calcium ion release. This indicated that the materials under test are bioactive.[63]

Comparing NeoPutty MTA, NeoMTA Plus, and MTA Angelus's ability to release ions in response to human dental pulp cells. Sample discs from all examined materials were used in the investigation, along with material eluents in the ratios of 1:1, 1:2, and 1:4. Human dental pulp cells (HDPCs) were isolated and cultured in the presence of the evaluated materials. ICP-MS and SEM-EDX were used to analyze surface deposition and elemental analysis. The results showed that MTA released calcium ions at a substantially higher rate than NeoPutty and NeoMTA Plus.[64]

Rabello C Z et al. measured the setting time, solubility, pH, calcium ion release, and radiopacity of MTA-A, MTA-HP, and BD.The sample size was established using previous investigations. Various tests and measurements were carried out using defined procedures.It was observed that all materials had favorable physicochemical properties and may be used as bioceramic cements in dental applications.[65]

The effect of obturation technique on bioactivity

Endoseal MTA, Well-Root ST, EndoSequence BC Sealer, EndoSequence BC Sealer HiFlow, and AH Plus were exposed to a temperature of 100°C for one minute to assess how heat influences their physical characteristics. Changes in setting time, flow, and film thickness were recorded, along with pH measurements. Additionally, SEM and EDS analyses were conducted to assess the structural and elemental composition of the set materials. Exposure to 100°C led to a significant reduction in both the setting time and flow properties of all the sealers tested, with Endoseal MTA being the most affected. In addition, film thickness increased significantly in Endoseal MTA under thermal exposure. pH values and SEM/EDS findings remained unchanged. These results suggest that elevated temperatures, as encountered during warm vertical compaction, could negatively impact the handling and performance of calcium silicate-based sealers due to altered physical characteristics.[66]

Thermocouples were utilized to record the average temperatures generated by two different heat delivery devices within the root canal space. Two premixed sealers—TotalFill BC and HiFlow BC (FKG, Switzerland)—were allowed to set and subsequently analyzed. Characterization was performed at two time points: immediately after setting and after 28 days of immersion in physiological solution. Analytical techniques included X-ray diffraction (XRD), energy-dispersive spectroscopy (EDS), scanning electron microscopy (SEM), and inductively coupled plasma (ICP) to assess ion release. Fourier-transform infrared spectroscopy (FT-IR) was employed to study the organic constituents in both unset and temperature-exposed sealers, following acetone extraction. Thermographic analysis further contributed to the assessment of thermal behavior. Findings indicated that the actual temperatures achieved did not match those displayed on the device dials, with peak readings recorded at the coronal region and lower levels apically. The two sealers shared similar compositions, differing primarily in their carriers. Both materials were composed of tricalcium silicate, dicalcium silicate, and zirconium oxide. Although calcium hydroxide was not detected following immersion in a physiological solution, the release of calcium ions was still observed. Thermal exposure altered the chemical structure of both materials; however, these changes appeared reversible upon cooling.[67]

The chemical composition of an experimental sealer, along with MTA Fillapex, Apexit Plus, and AH Plus, was examined using scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS). To evaluate the effect of heat during the warm vertical compaction technique, the sealers were analyzed after being exposed to either 100°C or 37°C for one minute. X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FT-IR) were employed to identify the reaction products following the setting process. The results revealed that the experimental tricalcium silicate-based sealer and Apexit Plus exhibited calcium hydroxide peaks post-setting, which were absent in MTA Fillapex.[68]

Seven endodontic sealers—AH Plus, Adseal, MTA-Fillapex, RoekoSeal, GuttaFlow 2, GuttaFlow BioSeal, and EndoRez—were subjected to thermal stress using an endodontic obturation device at 230°C to evaluate changes in their surface morphology, chemical composition, and thermal behavior. Energy-dispersive spectroscopy (EDS) revealed modifications in elemental composition following a 5-second heat application. However, scanning electron microscopy (SEM) demonstrated that AH Plus and GuttaFlow BioSeal maintained their surface structure. In contrast, high temperatures adversely affected Adseal, MTA-Fillapex, RoekoSeal, GuttaFlow 2, and EndoRez. The minimal changes observed in AH Plus and GuttaFlow BioSeal suggest their suitability for use in thermally activated obturation techniques.[69]

A recent scoping review explored how elevated temperatures influence the chemical and physical properties of bioceramic root canal sealers, adhering to the PRISMA Extension for Scoping Reviews framework. Nineteen out of the 91 were selected for qualitative analysis following independent screening by two reviewers . The majority of the included studies—published between 2014 and 2024—were in vitro investigations examining materials such as EndoSequence BC Sealer, BioRoot RCS, and TotalFill BC under various thermal conditions. The review reported mixed outcomes: while some sealers maintained their properties under heat, others exhibited alterations in setting time, flow, and chemical structure. These variations were largely dependent on the sealers' composition and the specific experimental protocols used. The authors emphasized that although some bioceramic sealers may be compatible with thermoplastic obturation techniques, laboratory findings should be interpreted cautiously, as they may not directly reflect clinical behavior. Additional clinical studies are warranted to validate these observations.[70]

A comprehensive and structured electronic search was performed in November 2023 and subsequently updated in April 2024, adhering to PRISMA 2020 guidelines. This search focused on in vitro studies examining the physicochemical properties of calcium silicate-based sealers (CSSs). The risk of bias was assessed using the PRILE 2021 guidelines. The search initially returned 6,421 results, from which 10 studies were selected for qualitative analysis. These studies evaluated 11 physicochemical properties, with setting time and flow being the most commonly assessed. The results indicated that heat exposure accelerates setting time, reduces flow, and increases film thickness of CSSs. However, there are ongoing concerns about solubility, viscosity, radiopacity, dimensional stability, microhardness, porosity, and compressive strength, emphasizing the need for more research. Certain calcium silicate-based sealers (CSSs), such as MTA Fillapex and EndoSequence BC Sealer HiFlow, showed minimal alterations upon heat exposure, suggesting their suitability for use in warm obturation techniques .[71]

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