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# In Vitro Microleakage Evaluation of Bioceramic and Zinc-Eugenol Sealers with Two Obturation Techniques

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**Abstract:** Aim of the study is to compare the quality of the apical seal offered by a zinc-eugenol and a tricalcium-silicate-based sealer, both used with the single-cone or with the continuous wave of condensation technique. Forty central incisors were divided into four groups (n = 10), according to the two sealers and the two obturation techniques under investigation, and their outer surface was isolated with nail varnish. After endodontic treatment, samples were immersed in methylene blue dye for 72 h, then included in self-curing resin and sectioned to longitudinally expose the canal apical third. The depth of dye penetration was measured in each group. Mean values were compared by two-way-ANOVA test. Multiple comparisons were performed by Tukey test. The level of significance was set at 0.05 in all tests. The continuous wave of condensation technique led to reduced microleakage. Moreover, dye penetration values were reduced for the tricalcium-silicate sealer. In terms of microleakage, the warm continuous wave of condensation technique seems promising even when combined to a bioceramic sealer.

**Keywords:** bioceramic; continuous wave condensation; microleakage; single-cone; zinc-oxide-eugenol



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## 1. Introduction

The ultimate aim of the root canal treatment, after an adequate disinfection carried out by mechanical instruments [1] and chemical solutions [2–5], is to fill in a three-dimensional way the endodontic space, in order to obtain a fluid-tight barrier, stable over time, with the purpose of protecting the periradicular tissues from microorganisms present in the oral cavity.

The most common endodontic filling material is constituted by a combination of a semi-solid core (gutta-percha) and a fluid sealer [6], which have been classically used following cold gutta-percha techniques (single-cone or lateral condensation) warm compaction techniques. In cold obturation techniques, the three-dimensional effectiveness of the apical seal relies exclusively upon the sealer [7,8], while in warm techniques a hermetic apical seal is achieved both owing to the sealer and the heated gutta-percha.

The ideal properties of an effective endodontic sealer were first suggested by Grossman in 1978 [9]. Several sealers have been proposed over time: zinc oxide–eugenol formulations, calcium hydroxide sealers, glass ionomers, epoxy resin-based, methacrylate resin-based, and the recently introduced calcium silicate-based sealers [10].

Zinc oxide–eugenol-based sealers are obtained mixing a liquid preparation of eugenol with a powder containing zinc oxide stably crystallized in a hexagonal wurtzite structure, as confirmed by X-ray diffraction (XRD) analysis [11,12]. The setting reaction of zinc oxide–eugenol is a classical acid–base reaction giving a salt (amorphous chelate of zinc eugenolate) and water [13]. They are widely used in endodontics due to many advantages: antimicrobial activity [14,15], resorption in case of extrusion into periradicular tissues [16], and conveniently slow setting time [17]. However, they also have some disadvantages: possible discoloration of tooth structure [18], shrinkage on setting [19], cytotoxic potential, and interference with adhesive procedures due to the free eugenol release in tissue fluids and dental tissues [20,21].

With the recent introduction of calcium silicate-based endodontic sealers, a more biological approach has taken hold [22]. As confirmed by XRD phase observations, these innovative materials are based on tricalcium silicate, calcium phosphate, zirconium oxide, and calcium hydroxide [23], and hence they are also known as "bioceramics" or "hydraulic calcium silicate-based sealers" [24] or "calcium phosphate-based root canal sealers" [25]. Bioceramics raised great interest because of their bioactivity when in contact with tissue fluids [22], their high biocompatibility (with no irritation of periradicular tissues) [26], their slight expansion after setting (which helps to create a hermetic barrier) [27], and their content of calcium phosphate (which allows to create a crystalline structure similar to tooth and bone apatite materials, improving adhesion to the root dentin) [28].

In some particular situations, such as in presence of widely oval canals or extremely complex anatomies [29], cold gutta-percha techniques have been criticized as they would hardly allow the complete filling of the whole endodontic space [30], while warm obturation techniques would appear more suitable to achieve a hermetic and three-dimensional apical seal [31]. However, most manufacturers, in agreement with recent literature reviews [32], strongly recommend the use of bioceramics according to the single-cone obturation technique. Many properties of tricalcium-silicate-based sealers render them particularly suitable to be used in combination with such a technique: they show no shrinkage, no resorption, and they may undergo some degree of expansion upon setting [33]. Several in vitro studies [33–39] investigated the possible improvement of the apical seal quality by combining tricalcium-silicate-based root canal sealers to the warm vertical compaction, but leading to contrasting results. Camilleri [36] keeps on recommending the use of these type of sealers exclusively with cold obturation techniques; Viapiana at al. [38], on the other hand, claimed the absence of obvious differences when using bioceramic sealers with cold or warm obturation techniques. Thus, further research seems still required to help clarifying such an ongoing debate.

On the above bases, the aim of the present in vitro study is to compare the extent of methylene blue dye penetration through the apical foramen following endodontic obturation procedures performed employing a zinc-eugenol and a bioceramic root canal sealer, in combination with the single-cone and with the continuous wave of condensation techniques.

The null hypotheses to be tested were that there were no statistically significant differences in terms of microleakage between the tested sealers and between the obturation techniques.

## 2. Materials and Methods

The present in vitro research has been conducted in full accordance with the World Medical Association Declaration of Helsinki and with the rules of the local ethic committee of "G. D'Annunzio" University of Chieti, Italy. All human biological materials were obtained during routine dental procedures, with patients' agreements. All teeth used for the experimental procedures were extracted for periodontal reasons and they would have been extracted anyhow, even if not included in the present investigation: they were not thrown away but were kept with the aim of being employed for the experimental procedures, with patients' agreement.

Forty extracted human upper central incisors (n = 40) were selected. All teeth were inspected with a stereomicroscope 20× (MDG17, Wild Heerbrugg, Heerbrugg, Switzerland) to detect the presence of caries, open apices, fractures, or resorption areas, that represented exclusion criteria. The specimens were carefully cleansed with ultrasound to remove extraneous tissue and calculus, and then stored in an aqueous solution of 0.4% chloramine-T before the use.

The crowns were horizontally sectioned at the canal entrance level using a cylindrical diamond bur operated with a high-speed electric handpiece (200,000 rpm) and under abundant irrigation, so that the length of all roots was adjusted to approximately 12 mm. The work length was determined by a K-file # 15 (Dentsply Maillefer, Ballaigues, Switzerland) introduced in the canal space until the tip was just visible at the apical foramen. Then the roots were cleaned and shaped with Mtwo rotary system files (Sweden & Martina SpA, Padova, Italy) alternating each instrument with a profuse irrigation of a 5.25% sodium hypochlorite (NaOCl) solution. All canals were enlarged to size 50, 0.04 taper, to working length. After preparation, the canals were irrigated with 5 mL 5.25% NaOCl followed by 5 mL 17% ethylenediaminetetraacetic acid (EDTA) for 60 s to remove the smear layer. Successively, the specimens were irrigated with distilled water to avoid the prolonged effect of the EDTA and NaOCl solutions, then the canals were dried with paper points. Finally, external surfaces of the roots were coated with three layers of clear nail varnish except for the apical foramen, which was kept patent by introducing a size 25 k-file in the apex prior to the application of the nail varnish.

The roots were randomly divided into four groups, according to the root canal sealer and the obturation technique used.

Information about the endodontic sealers investigated in this study (Figure 1) are summarized in Table 1.


(**a**) (**b**)

**Figure 1.** Root canal sealers investigated in the study: (**a**) zinc-oxide-eugenol-based canal sealer; (**b**) tricalcium-silicate-based sealer.

**Table 1.** Information about the root canal sealers investigated in the study.

| Type | Product Name (Manufacturer, Country) | Composition | | Lot. No |
|---|---|---|---|---|
| Zinc-oxide-eugenol-based sealer | Pulp Canal Sealer (Kerr, Orange, CA, USA) | Powder<br>Liquid | Zinc oxide, precipitated silver, oleoresin, thymol iodide<br>Oil of cloves, Canada balsam | 7,929,829 |
| Tricalcium-silicate-based sealer | BioRoot RCS (Septodont, Saint-Maur-des-Fossés, France) | Powder<br>Liquid | Tricalcium silicate, zirconium oxide<br>Aqueous solution of calcium chloride | B26389 |

### 2.1. Group 1: Zinc-Oxide-Eugenol-Based Canal Sealer and Single-Cone Technique

A single cone of gutta-percha tapered with diameter and conicity corresponding to the final shaping instrument (# 50.04) was tried in the root canal after visual and tug-back control. The accuracy of the working-length was assessed by inspecting the tooth through the apex with a stereomicroscope (20× magnifications) and by periapical radiography. The canal was dried and the sealer (Pulp Canal Sealer, Kerr, Orange, CA, USA) was mixed according to the manufacturer's instructions. Then, the single cone was coated with sealer and inserted to the working-length with slow movement in order to allow the sealer to flow back coronally and avoid apical extrusion. The coronal excess of gutta-percha was finally removed with a heated instrument.

### 2.2. Group 2: Tricalcium-Silicate-Based Sealer and Single-Cone Technique

In this group all the sample preparation procedures were exactly the same as those described for to the previous group, but a tricalcium-silicate-based sealer (BioRoot RCS, Septodont SAS, Saint-Maur-des-Fossés, France) was used.

### 2.3. Group 3: Zinc-Oxide-Eugenol-Based Canal Sealer and Continuous Wave of Condensation Technique

A master gutta-percha cone tapered with diameter and conicity corresponding to the final shaping instrument (# 50.04) was selected, then the tip was trimmed with a scalpel in order to obtain a proper tug-back at 0.5 mm from the working length. The zinc-oxide-eugenol-based sealer was manipulated with 1:1 proportion according to the manufacturer's instructions. After setting the System B (Sybrondental, Orange, CA, USA) temperature at 200 °C, a Buchanan plugger was mounted on the device's handle and inserted at 5 mm short of the working length without binding on the canal walls; at this level, a rubber stopper was positioned to mark the coronal reference point. The master cone was then coated with sealer and fitted into the canal space in order to place the material on the dentinal walls up to the apical level. Afterwards, the plugger was activated and inserted into the canal filled with the gutta-percha point until the rubber stopper reached the coronal reference point. The heat source was deactivated and the plugger was pushed in apical direction for 10 s with the aim of compensating the gutta-percha shrinkage and allowing the penetration of the thermoplastic gutta-percha (and eventually of the sealer) in the accessory canals. The power of the heat source was activated again for 1 s to separate the plugger from the compacted gutta-percha; then the plugger was extracted and a manual plugger was immediately inserted to verify the presence of the core material at the apical level and its adequate compaction [40]. Back-filling of the rest of the canal space was achieved by injecting (Obtura II; Obtura Corporation, Fenton, MO, USA) and compacting warm gutta-percha with a pre-fitted plugger.

### 2.4. Group 4: Tricalcium-Silicate-Based Sealer and Continuous Wave of Condensation Technique

In this group the root canal obturation was achieved following the technique described for Group 3, but the same tricalcium-silicate-based root canal sealer used in Group 2 was employed.

Specimens from every group were stored at 37 °C and 100% humidity for 72 h, prior to be subjected to the subsequent experimental procedures.

After completing the isolation of the root by covering the coronal surface with three additional layers of nail varnish, the teeth were immersed in an aqueous solution of 2% methylene blue dye and stored in an incubator, at 37 °C for 72 h. Following exposure to dye, the roots were meticulously rinsed under running water.

The samples were included in self-curing acrylic resin and then put on a coverslip. The experimental teeth were sectioned longitudinally using a low-speed diamond saw (Micromet M; Remet S.p.A., Casalecchio di Reno, Italy) in a direction approximately parallel to the long axis of the tooth and through the apex in order to expose the canal space. Sectioned samples were placed on a piece of millimetric paper and examined under a stereomicroscope at 20× magnifications. Images were captured and then saved using a digital device. To estimate the microleakage, the images were imported in a specific software (Image J version 1.42q; National Institutes of Health, Bethesda, MD, USA) and analyzed after setting a scale using the millimetric paper on the background of every sample. The calculation of the microleakage was made by measuring the distance (mm) between the apical foramen and the most coronal point of dye infiltration.

Means and standard deviations were calculated for each experimental group. The differences between the means were statistically analyzed by two-way analysis of variance (ANOVA), in order to assess the effect of the two factors under investigation (the "endodontic sealer" and the "obturation technique") on the microleakage mean values. Multiple comparisons were performed by a Tukey Test. The level of significance was set at $p < 0.05$ in all tests.

After microleakage evaluation, representative samples from every experimental group were dehydrated in progressive concentrations of ethyl alcohol (70%, 80%, 90%, and 100%) and sputter-coated with a 300-A°-thick gold layer. They were then observed under a scanning electron microscope (SEM) (EVO 50 XVP LaB6; Carl Zeiss SMT Ltd., Cambridge, UK) at 1000× and 3500× magnification, in order to assess the potential sealer penetration into the dentinal tubules.

## 3. Results

The obtained microleakage mean values (and standard deviations), organized according to the factors under investigation (endodontic sealer and obturation technique), are shown in Table 2 and graphically depicted in Figure 2.

**Table 2.** Mean values (mm) and standard deviations (mm) of the microleakage in each experimental group organized according to the considered factors: endodontic sealer and obturation technique.

| Endodontic Sealer | Obturation Technique | |
|---|---|---|
| | Single Cone | Continuous Wave |
| Zinc-oxide-eugenol-based sealer (ZOE) | Group 1<br>$3.63_{1}{}^{a}$ (0.82) | Group 3<br>$2.46_{2}{}^{a}$ (1.29) |
| Tricalcium-silicate-based sealer (BioRoot) | Group 2<br>$2.33_{1}{}^{b}$ (0.85) | Group 4<br>$1.02_{2}{}^{b}$ (0.60) |

$^{a,b}$ Different superscript letters suggest statistically significant differences in vertical comparisons. $_{1,2}$ Different numbers in pedex suggest statistically significant differences in horizontal comparisons.

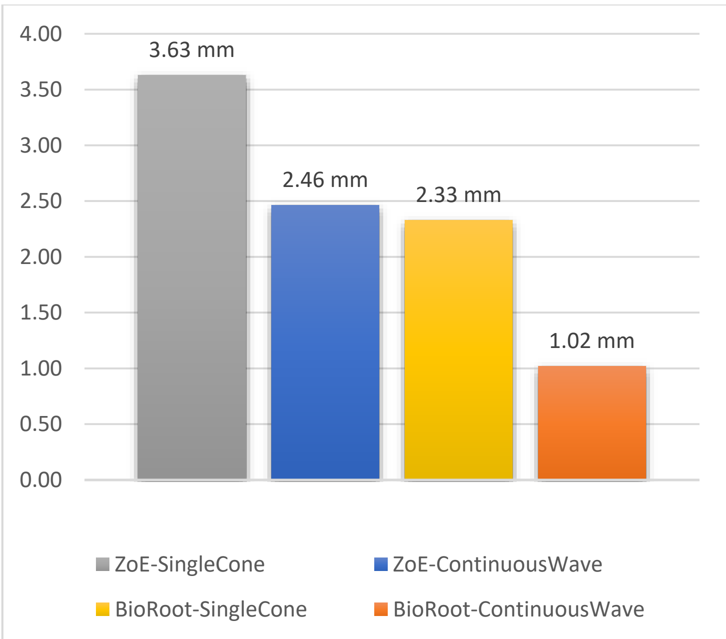


**Figure 2.** Apical microleakage mean values (mm) and standard deviations (mm) of the studied samples, organized according to the considered factors (endodontic sealer and obturation technique).

The maximum infiltration values (3.63 mm) were observed in Group 1 (zinc-oxide-eugenol-based sealer and single cone technique), significantly higher compared to all the other experimental groups.

The lowest apical microleakage mean values (1.02 mm) were found in Group 4 (tricalcium-silicate-based sealer and the continuous wave of condensation technique), significantly reduced compared to the remaining experimental conditions tested.

No statistically significant differences were observed when comparing Group 2 (tricalcium-silicate-based sealer and single-cone technique) and Group 3 (zinc-oxide-eugenol-based canal sealer and continuous wave of condensation technique).

SEM analysis showed an evident penetration of the bioceramic root canal sealer into the dentinal tubules (Figure 3), whereas in samples obturated with zinc-oxide-eugenol-based sealer dentinal tubules appeared empty (Figure 4).

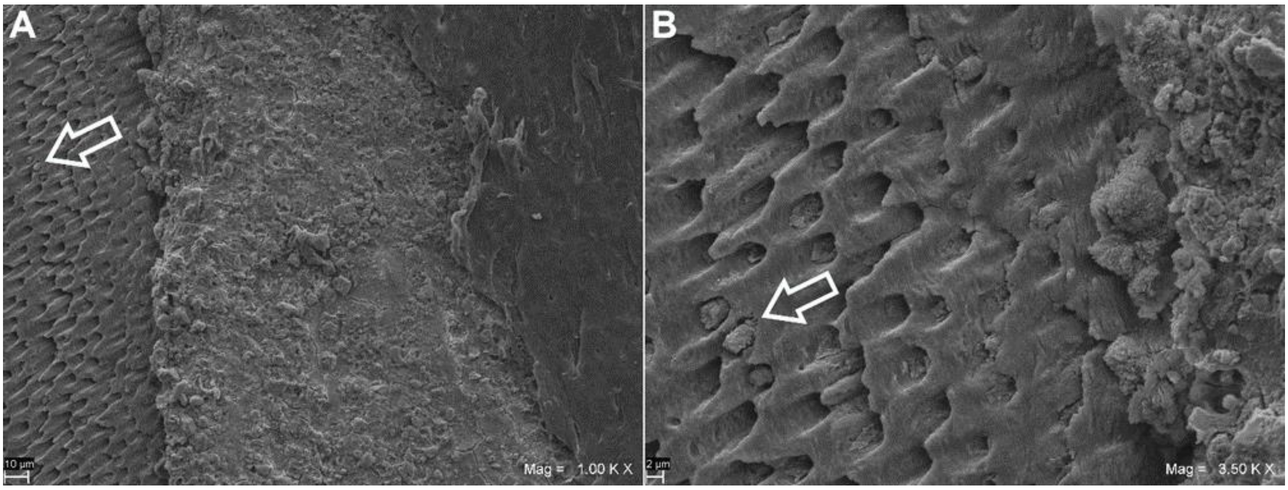


**Figure 3.** Scanning electron microphotograph ((**A**), original magnification 1000×; (**B**), original magnification 3500×) of a sample obturated with a bioceramic-based root canal sealer and warm vertical condensation. Sealer penetration into the dentinal tubules may be observed (arrows).

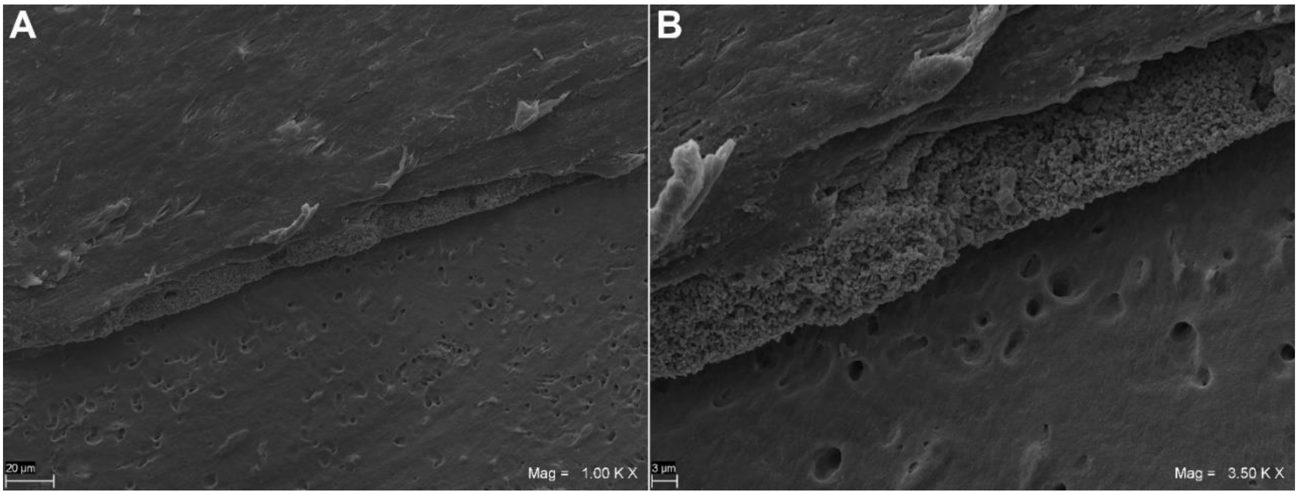


**Figure 4.** Scanning electron microphotograph ((**A**), original magnification 1000×; (**B**), original magnification 3500×) of a sample obturated with zinc-oxide-eugenol-based root canal sealer and warm vertical condensation. Dentinal tubules appear empty.

## 4. Discussion

The null hypotheses tested in the present study had to be rejected: there were statistically significant differences in terms of microleakage between the tested sealers and obturation techniques.

Several methods have been proposed in the literature to investigate microleakage: Fluid filtration or transportation method, which consists in measuring an air bubble movement within a capillary tube [41,42]; dye extraction or dissolution method, in which the teeth are dissolved in acids to release the dye from the interfacial areas and obtain a solution whose optical density is determined with a spectrophotometer [41]; bacteria and toxin infiltration method, a qualitative evaluation whereby penetration of bacterial organisms is estimated [43]; glucose penetration model, which relies on the assessment of the filtration rate of glucose along the root canal obturation material [44,45]; protein microleakage test, that is performed using bovine serum albumin in a dual-chamber apparatus and estimated using a spectrophotometer [46,47]; electrochemical microleakage method, which consists in the diffusion of ions through narrow spaces [48]; three-dimensional methods carried out using cone-beam computed tomography (CBCT) [49] and SEM evaluations [50]; dye penetration methods, in which teeth are immersed in various types of dyes and then sectioned (longitudinally or transversely) or cleared to record linear penetration of the dye [43,51]. To date, there is still some debate about the most reliable method for investigating the apical microleakage in endodontics. In this study, the dye penetration method was preferred because of its relative simplicity and easy replicability.

In the present research, the highest dye infiltration values were observed in samples from Group 1, probably because they combined the intrinsic limits of both a conventional sealer and a cold compaction technique. Among the main disadvantages of a single-cone technique, the lack of a sufficiently homogeneous obturation has been often underlined, together with the reduced chance of filling every void, which ultimately provide an apical seal whose predictability ends up relying just on the specific properties of the endodontic sealer. Clinically, this could represent a limit in case of resorbable sealers [7,8]. In Group 1, a conventional zinc-oxide-eugenol-based canal sealer was used: based on previous research, such a material shows a certain degree of shrinkage after setting [19], which might lead to the generation of voids. In Group 2, a tricalcium-silicate-based sealer was used in combination with the single-cone technique and the dye penetration turned out to be significantly lower than in Group 1. Tricalcium-silicate sealers have the ability to undergo a slight expansion after setting [27], which probably helped increasing the tightness of the apical seal even if combined with a particularly inefficient obturation strategy. The present results would support the wide-spread habit of employing bioceramic sealers together with a single cold gutta-percha cone [52], as the many limitations of such an extremely simplified obturation technique seem to be overcome owing to the advantageous properties of those innovative materials.

Tricalcium-silicate-based sealers seem to positively interact with dentin fluid, potentially inducing biomineralization with the formation of mineral tags within the dentinal tubules, thus enhancing the biological activity within the root canal [53], due to the presence of calcium phosphate in their composition that enhances the setting mechanism [28] and improves the chemical bonding between the sealer and the root dentin [26]. Calcium phosphate is also the main reason for the outstanding bioceramic's biocompatibility, that is their ability to not trigger adverse reaction when in contact with tissues [28]. Calcium phosphate is, in fact, one of the major inorganic constituents of hard tissues. Indeed, it has been shown that these materials can even promote bone regeneration [54] when accidentally extruded through the apical foramen during root canal filling procedures [55,56]. These materials have also the ability to establish a micromechanical retention by tag sealer penetration in dentinal tubules [57]. In the present study, the scanning electron microscope analysis confirmed a visible penetration of the bioceramic root canal sealer into the dentinal tubules: this behavior may help establishing a stronger physical barrier, improving retention of the root filling, entombing possible residual bacteria into dentinal tubules and, probably, enhancing the ultimate quality of the apical seal [35]. Among the other interesting properties of bioceramic-based sealers, the following should be underlined: An adequate setting time [26,58,59]; a reduced risk of discoloration of tooth structures when compared with other conventional root canal sealers [26]; a high radiopacity due to the presence of bismuth trioxide [60,61]; an antimicrobial activity, owing to the high pH values and the high tendency to release calcium ions [62,63]. Of course, these materials are not completely free from possible shortcomings: a major drawback is their complex removal in case of orthograde endodontic retreatments [64]. Moreover, endodontically treated teeth may often benefit from the use of adhesively luted fiber-post to effectively support the coronal restoration [65–70] but a thorough cleaning of the root canal walls, which is required for an appropriate post-space preparation, might become definitely challenging when a bioceramic sealer is used [64]. Finally, some doubts seem to come from a few studies that showed how certain properties of bioceramics, such as flow [62,71,72] or solubility [73,74], do not steadily fulfil the ISO standard requirements.

In Group 3, the zinc-oxide-eugenol-based canal sealer was used together with the continuous wave of condensation technique and the microleakage results were comparable to Group 2, again significantly enhanced compared to Group 1. In this case, the improved performances can be explained owing to the advantages of the continuous wave of condensation, which typically lead to a denser and more homogeneous filling and allow a better sealing [7,75], in presence of less voids, when compared to cold obturation techniques [76]. The use of warm gutta-percha allows to fill even the endodontic areas of challenging access, due to the reduced viscosity and owing to the vertical compaction loads.

In Group 4 (tricalcium-silicate-based sealer and continuous wave of condensation technique), the dye penetration values were the lowest among all the experimental groups. According to these authors, such a finding could be explained taking into account the combined benefits coming from both the warm obturation technique and the advantageous properties of the bioceramic-based sealer.

## 5. Conclusions

As previously mentioned, the current literature is not unanimous when dealing with the association of a bioceramic-based sealer to a warm obturation techniques. Qu et al. [37] showed that after heat application, bioceramic-based root canal sealer exhibited a reduction in setting time and flow, which could negatively influence the performance of obturation and the procedure outcome. Camilleri et al. [36] observed how the material chemistry and the physical properties were not affected by the heat, but still suggested to follow the guidelines of the manufacturers, which recommend the use of a single cone technique. Other studies [33–35] affirmed that the obturation technique does not affect the efficacy of the bioceramic sealers. Hadis et al. [39] compared a new bioceramic-based root canal sealer, designed to be used with a warm obturation technique, with one recommended for the single cone technique; the results showed no differences between the two sealers, when they were used following a warm filling technique. Viapiana et al. [38] observed bioceramic sealers to be stable even when used with a warm vertical compaction, but they also underlined the need for a better investigation about the effect of heat on sealer properties. Atmeh et al. [77] claimed that even if heat does not cause changes in the bioceramic chemical structure, it seems to produce some microstructural changes due to water loss. Specifically, a Fourier-transformed infrared spectroscopy (FT-IR) analysis of an heated bioceramic sealer revealed a drop in the peaks representing vibrational modes of the OH group in water, thus proving an irreversible water loss above 100 °C by means of evaporation [78].

Within the limits of the present study, it seems that in terms of microleakage of recently sealed samples a warm obturation technique is promising even when used in association with a tricalcium-silicate root canal sealer. However, it must be underlined that no attempt was made herein to artificially simulate any effect of specimen aging. Thus, the long-term stability of the apical seal obtained with tricalcium-silicate-based sealers subjected to heat sources should be still verified, and it could represent an interesting subject for further research.

Based on the results of the present study, the following conclusions can be drawn:

- The obturation technique is a factor able to significantly affect the quality of the apical seal in terms of microleakage and the dye penetration is reduced with a warm wave of condensation technique, compared to a cold single-cone obturation technique;
- Obturation technique being equal, tricalcium-silicate-based root canal sealers appear to be more performing than conventional zinc-eugenol-based sealers;
- In terms of microleakage, warm obturation techniques seem promising even when used in combination with a tricalcium-silicate-based root canal sealer.

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