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# Comparison of physico-chemical properties of zinc oxide eugenol cement and a bioceramic sealer

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**Abstract**

The aim of the present study was to compare the physico-chemical properties of EssenSeal with AH PLUS bioceramic and Pulp Canal Sealer EWT. Flow, solubility, film thickness, radiopacity and setting time were evaluated according to ISO 6876 (2012) specifications. External and cross-section surface characteristics were analysed under a scanning electron microscope. Statistical analysis was performed using Shapiro–Wilk's test, one-way ANOVA and the Tukey HSD test. All the sealers conformed to the ISO 6876 (2012) standards, except for the setting time for AH plus bioceramic, which exceeded more than 10% of the time indicated by the manufacturer. Statistically significant differences were found between the three study sealers regarding the physico-chemical properties tested ($p < 0.05$). EssenSeal demonstrated characteristics respecting the ISO 6876 (2012) standards and can be considered a predictable alternative in root canal sealing.

**KEYWORDS**

calcium silicate, physico-chemical properties, root canal sealant, scanning electron microscopy, zinc oxide eugenol cement

## INTRODUCTION

The long-term success of root canal treatments is mainly due to the chemo-mechanical disinfection of the root canal system, and the stability of both coronal restoration and apical obturation [1]. This success is provided by root canal chemo-mechanical debridement aiming to reduce bacterial load [2], although shaping and irrigation procedures are not able to completely remove bacteria from the endodontic system [3]. The persistence of microorganisms in the root canal system remains the main cause of endodontic failure [4].

For this reason, the obturation procedures are essential to obtain bacterial entombing to prevent their proliferation. One of the most common causes of failure is represented by inadequate quality sealing of the root canal system [5]. According to this, the selection of an obturation material for clinical use is a crucial choice that contributes to long-term success of root canal treatment [1, 6, 7].

For a correct selection of the most suitable endodontic sealer, a deep knowledge of its features is required [1, 8]. The physico-chemical properties of an ideal endodontic sealer are as follow: adhesiveness, dimensional stability, sufficient setting time, insolubility to oral and tissue fluids, radiopacity, absence of staining, ability to create a seal, bacteriostatic properties, fine powders for anatomical accommodation and biocompatible [1].

At present, various products are commonly utilised in clinical practice, including sealer based on resin, zinc oxide eugenol, calcium hydroxide, glass ionomer and bioceramics [9]. Among the most widely used endodontic sealers, zinc oxide eugenol sealers have a long tradition in both clinical practice and scientific research [1]. Hydraulic sealers, precisely named as calcium silicate-based sealers [10], were introduced into the market as a possible substitute for traditional endodontic sealers [9].

Therefore, the purpose of the present in vitro study was to evaluate flow properties, setting time, radiopacity, solubility and film thickness of the new endodontic sealer EssenSeal (ES) (Produits Dentaires SA) when compared to AH Plus Bioceramics (AH) (Dentsply International) and Pulp canal sealer EWT (PCS) sealers (Kerr Corporation). Furthermore, the morphologies of the external surface and the cross-section of all sealers were also quantitatively assessed under scanning electron microscopy (SEM). The null hypothesis was that the tested sealers have comparable characteristics in terms of physico-chemical tests used.

## MATERIALS AND METHODS

EssenSeal, PCS and AH were tested for flow, setting time, solubility, radiopacity and film thickness according to ISO 6876 specifications [11]. The characteristics of the sealers tested are shown in Table 1. All materials were prepared and used according to manufacturer's instructions. ES was prepared by mixing one level dosing spoon of powder and one drop of liquid and gradually incorporating the powder into the liquid, until a creamy consistency was obtained, which was fluid enough to stretch out for 2 cm on the mixing block. PCS was prepared by mixing one level dosing spoon of powder with one drop of liquid and incorporating the powder into the liquid, then mixing them in a very small area of about 1–2 cm diameter with a stainless-steel spatula. AH is available as a pre-mixed single paste and was ready for use.

### Flow test

ISO 6876 international standard version 2012 was used to conduct the flow test which requires that a sealer shall have a diameter of at least 17 mm. 0.05 ml of the material was mixed and placed on the centre of a glass plate, with a weight of 20 g and dimensions of 20 mm, using a graduated syringe (Becton Dickinson). At 180 s (±5 s) after beginning the mixing, another glass plate was placed centrally on top of the sealer, followed by a weight giving a total mass of 120 g (±2 g). Ten minutes after initiating the mixing, the weight was removed and the maximum and minimum diameters of the compressed discs of sealers were measured. Two conditions were necessary to validate the tests: the difference between the maximum and minimum diameters could not exceed 1.0 mm and the compressed disc should have a uniform shape. If these conditions were not met, the test was repeated. Three determinations were carried out and the mean value was calculated to the nearest millimetre.

### Solubility

The solubility test was conducted using the ISO 6876 (2012) standards, according to which the sealer solubility shall not exceed 3.0% by mass. Cylindrical polytetrafluoroethylene moulds with an inner diameter of 7.75 mm and a height of 1.5 mm were filled with each type of mixed sealer with the adjunct of a standardised waterproof nylon thread applied to the handling procedures. Each sample was incubated at 37 °C and 95% relative humidity for 24 h and then removed from the mould and weighed three times each (m1) with an accuracy of 0.0001 g (Gibertini 500; Gibertini elettronica SRL). After that, samples were suspended in a plastic container of 7.5 ml milli-Q using the nylon threads to avoid any contact between the sample and container. Each container was placed in the incubator

**TABLE 1** Characteristics of sealers employed

| Product name (manufacturer, city, country) | Chemical matrix | Presentation | Composition | Batch number |
|---|---|---|---|---|
| AH PLUS (Dentsply DeTrey, Konstanz, BadenWürttemberg, Germany) * | Tricalcium silicate | Single paste | Zirconium Dioxide, Dimethyl sulphide, Lithium carbonate, Thickening agent | KI211103 |
| PULP CANAL SEALER EWT (Kerr, Brea, CA, USA) | Zinc oxide eugenol | Powder | Zinc oxide, precipitated silver, oleoresin, thymol iodide | 8 593 713 |
| | | Liquid | Oil of cloves, Canada balsam | |
| ESSENSEAL (Produits dentaires, SA, Vevy, Switzerland) | Zinc oxide eugenol | Powder | Zinc oxide, barium sulphate, excipient | 97 279 726 CL |
| | | Liquid | Eugenol, tea tree essential oil (Maleleuca), excipient. | |

at 37°C and 95% relative humidity for 7 days, rinsed with deionised water and dried at 60°C. Afterwards, each sample was weighed again (m2). The solubility was measured by calculating the weight loss of each sample (m1−m2) and expressing it as the percentage of the original mass, using the following formula: (m1−m2)/m1*100%.

## Setting time

Setting time was tested using the ISO 6876 (2012) standards stating that a sealer shall be no more than 10% respect the declared value by the manufacturer. Five Plaster of Paris cast rings of 10 mm diameter and 2 mm thickness were separately filled with one of the selected sealers and then inserted in an incubator at 37°C and 95% relative humidity for 2 min. Afterwards, a needle (NEWTRY GY-3) with a weight of 100 g and a diameter of 2 mm was used by carefully lowering its flat tip in a perpendicular direction against sealer discs. The sealer setting time was determined as the difference in terms of seconds between the end of sealer mixing and the moment at which indentations ceased to be visible in the material. Three measurements were performed for each sealer.

## Radiopacity

A sealer shall have a radiopacity of less than 3 mm of aluminium in accordance with the ISO 6876 (2012) standards implemented to test radiopacity. Plaster of Paris cast rings of 5 mm diameter and 1 mm thickness were separately filled with one of the selected sealers and then incubated at 37°C and 95% relative humidity for 24 h. The filling procedures were performed with the use of a syringe in order to avoid bubbles formation.

Each sample was positioned alongside an aluminium wedge with the thickness varying from 0.5 to 6 mm in order to uniform steps of 1 mm each and three digital radiographs per each sealer were taken (KAVO, Dental Imaging Technologies Corporation) at 60 kV and 2.5 mA with a focus-film distance of 30 cm and exposure time set to 0.16 s. Grey pixel values of each sealer and aluminium wedge on the images were measured using ImageJ software (National Institutes of Health). The radiopacity values were then converted into millimetres of aluminium (mm Al) as previously described [12].

## Film thickness

Film thickness was assessed using ISO 6876 (2012) which requires a film thickness lower than 50 μm. Each mixed sealer was placed between two glass plates characterised by a thickness of 5 mm and surface of 200 ± 25 mm$^2$. Before sealer placement, the thickness of the two glass plates was measured with a calliper (Dongguan Kuaijie Measuring Tool Instrument Co., Ltd.) to obtain the reference thickness starting point. After 180 ± 10 s from the start of mixing, a loading device was utilised to uniformly load 150 N vertically on the top plate allowing the sealer to entirely fill the space between the glass plates. After 10 min from the start of the mixing, the final thickness of the two glass plates with the sealer layer was measured (mm) using a micrometre, and the difference (in terms of mm) between the reference thickness starting point and the final thickness was determined as the film thickness of the sealer. Three measurements were performed for each sealer.

## SEM examination

In order to perform SEM examination, freshly mixed sealers were poured into cylindrical polytetrafluoroethylene moulds. A glass plate covered with a cellophane sheet was used to sustain the moulds and placed in a chamber (37°C, 95% relative humidity) for a period equivalent to three times the setting time. Through the use of a size 15 disposable surgical scalpel blade, fixed on a metallic stub (10 x 5 mm), samples were consequently sectioned and spatter coated with gold–palladium (Bal-Tec AG) at 20 mA. Quantitative analysis of the sample's external surface characteristics and cross-section was achieved under a field-emission SEM (GEOL JSM-6060). The examination was carried out at diverse magnifications, at a working distance from 6 to 10 mm and an accelerating voltage of 15 kV.

## Statistical analysis

The normality of data distribution was checked using the Shapiro–Wilk test. After verification of data distribution, a one-way ANOVA test was used to compare all the sealers. Tukey's HSD test was used for pairwise comparisons. The level of significance was set at alpha = 0.05; all *p*-values were two sided.

# RESULTS

The ISO 6876 (2012) requirement was satisfied by ES and PCS sealers. AH setting time exceeded 10% of that stated by the manufacturer. The physico-chemical property results of the sealers tested are shown in Table 2. According to flow test, all the sealers tested exceeded the minimum

**TABLE 2** Physico-chemical properties of the tested sealers

| | Tested sealers | | |
|---|---|---|---|
| | **I** | **II** | **III** |
| | **AH** | **PCS** | **ES** |
| Flow (mm)** | 25.46 ± 0.99 | 30.10 ± 2.17a | 27.64 ± 0.85 |
| Solubility (%)* | 0.137 ± 0.006 | 0.120 ± 0.003 | 0.114 ± 0.003a |
| Setting time (s)** | **408** ± 15.37B | 590 ± 7.90 ac | 119.8 ± 0.78ab |
| Radiopacity (mmAl)* | 9.83 ± 1.28 | 5.92 ± 2.49a | 9.52 ± 0.89b |
| Film thickness (μm)** | 10.24 ± 0.11b | 9.8 ± 0.07a | 10.78 ± 0.13ab |

*Note*: Values are mean ± standard deviation.

Values in bold letters do not comply with ISO 6876 standards. Values followed by different superscript letters in each row differ significantly. One-way ANOVA test between sealers: *$p < 0.05$, **$p < 0.001$.

value of 17 mm requested. PCS showed a greater flow followed by ES and AH. Statistically significant differences were present when AH was compared to PCS ($p = 0.000$), whereas no difference was observed between ES and PCS ($p > 0.05$). In terms of solubility, all sealers were respectful of the 3% of the mass established by ISO 6876 (2012) standards.

Statistically significant differences were present when ES was compared to AH ($p < 0.001$).

AH sealer had a stated setting time between 2 (180′) and 4 (240′) hours, thus did not meet the standards indicated by the manufacturer (Mean = 408, SD ± 15.37). ES and PCS sealers respected the setting time established, respectively, in 2 to 4 and more than 6 h. Statistically significant differences were present between all the sealers tested ($p = 0.000$).

All sealers showed radiopacity values above that requested by ISO 6876 (2012) standards (3 mm Al). Similar values were observed between AH and ES, whereas the PCS sealer showed a lower radiopacity. Statistically significant differences were present when PCS was compared to both AH ($p < 0.001$) and ES ($p < 0.05$).

All the sealers presented a film thickness lower than 50 μm, although a statistically significant difference was present between sealers ($p = 0.000$). PCS showed a lower film thickness followed by AH and ES. Statistically significant differences were present when AH was compared to both PCS ($p = 0.000$) and ES sealers ($p = 0.000$), whereas no difference was observed between ES and PCS ($p > 0.05$).

## SEM examination

Scanning electron microscopy revealed sphere-shaped polymers of different sizes homogeneously spread on the external surface in both PCS and ES samples. In the PCS sample, a more uniform and organised layer with a higher number of polymers in a reduced resin matrix was appreciated, in contrast to ES. AH samples revealed a regular surface with homogenous, smaller and globular-like particles of different sizes. The phases were uniformly distributed, and at 2000× magnification, shrinkage lines can be noted, probably arising from cutting procedures (Figure 1).

# DISCUSSION

The aim of the present study was to evaluate flow, solubility, radiopacity, film thickness and setting time properties of a zinc oxide eugenol-based sealer ES compared to both PCS and AH according to ISO (2012) 6876 standards. The results of this study showed that all sealers tested differ in the physico-chemical properties analysed. Thus, the null hypothesis was rejected.

All the sealers tested satisfied the ISO 6876 (2012) standard for the physico-chemical properties analysed except for the setting time of AH, which exceeded more than 10% of the time indicated by the manufacturer. This could be partly explained by the fact that calcium silicate-based cement needs moisture present in the dentinal walls to improve the setting reaction [13].

Recently, it was demonstrated how the properties of calcium silicate-based sealers are strongly influenced by the surrounding environment, for this reason in vivo and ex vivo studies are more accurate compared to in vitro studies in the evaluation of their physico-chemical properties [14, 15].

AH showed a higher value in terms of solubility with respect to both ES and PCS. Since there are no other articles evaluating the physico-chemical properties of AH and ES, it is only possible to indirectly compare different calcium silicate-based and zinc oxide eugenol sealers. Other authors demonstrated greater solubility for the calcium silicate-based sealers tested compared to zinc oxide eugenol sealers, indeed [16, 17].

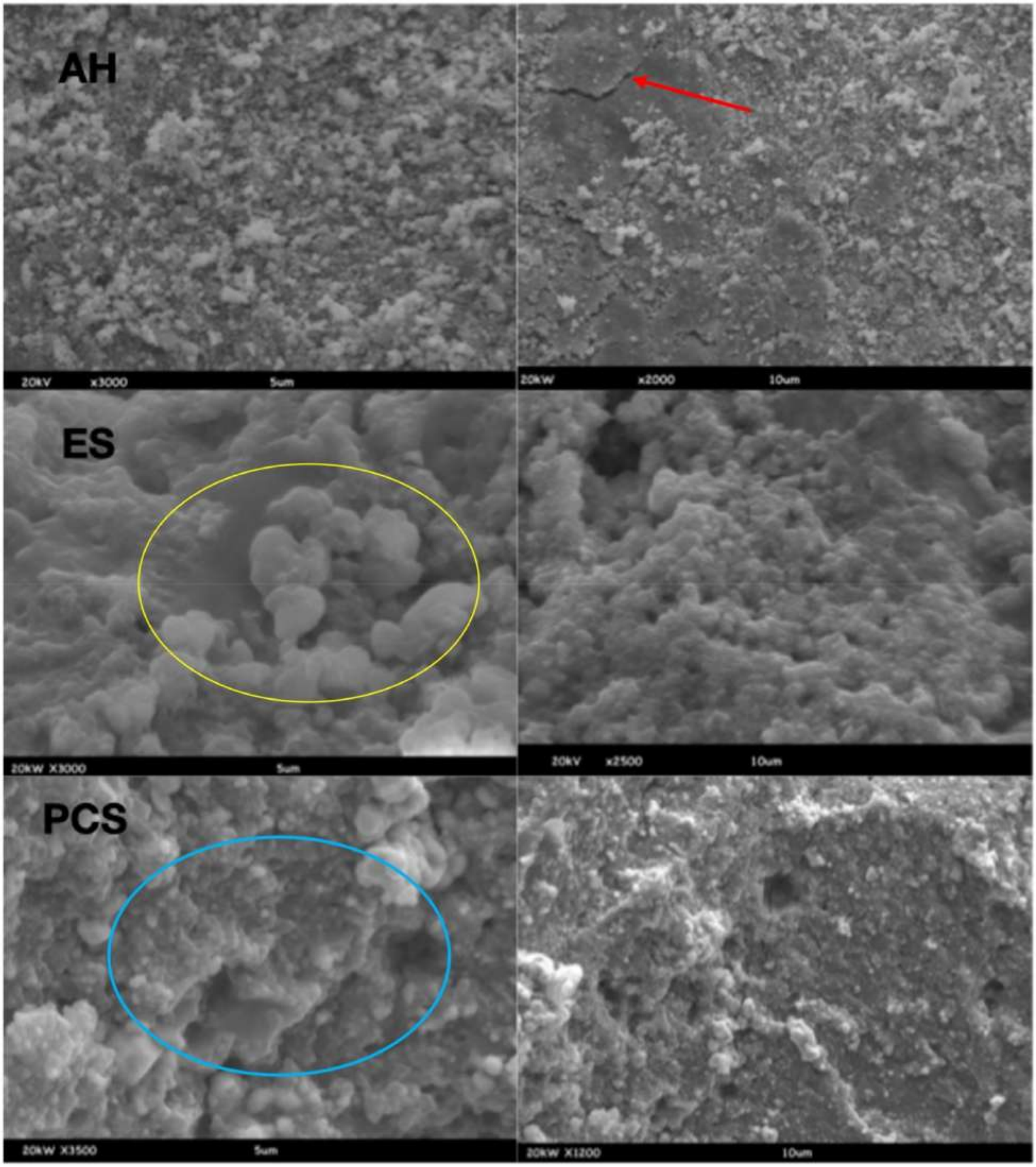


**FIGURE 1** Qualitative analysis of the internal and external surface characteristics. AH, AH plus bioceramic; ES, EssenSeal; PCS, Pulp canal sealer. The micrographs were acquired at different magnifications ranging from x1200 to x3000 in order to highlight surface characteristics (respectively, AH: Right image ×2000 magnification, left image ×3000 magnification, ES: Right image ×2500 magnification, left image ×3000 magnification, PCS: Right image ×1200 magnification, left image ×3500 magnification). In the second image of AH plus bioceramic, a shrinkage line arising from cutting procedures during sample preparation (red arrow) can be noted. PCS presents more uniform and organised layers with higher polymer numbers in the resin matrix (blue circle) compared to ES (yellow circle).

According to our result, AH respected the standards and disagreed with recent studies in which different calcium silicate-based sealers did not satisfy all the ISO 6876 (2012) requirements [18–20]. Flow property plays a crucial role because it allows proper sealer penetration into the endodontic system [21]. According to the standard requirements, the sealer during the flow test should have a diameter of no less than 17 mm with a standard deviation of 1 mm. All the sealers tested respected the minimum demanded flow value; PCS showed the highest results with respect to ES and AH. The flowability results of the PCS of this research are slightly different in comparison to those of Donnermeyer et al. [22].

Regarding this, the authors tested the physico-chemical properties of different endodontics sealers at different temperatures, stating that there were no differences in terms of physico-chemical properties when heating did not exceed 60 s. Despite this, considering their PCS results obtained at 20°C, they found lower flowability values than that obtained in this study. The flow characteristics seem to be influenced by particle size in the powder part of the sealers, as well as the setting time.

In 1982, Grossman showed better plasticity of zinc eugenol resin-based sealer and demonstrated improvement in the flow test of this sealer [23], despite the flow values being sensibly inferior to that obtained by Siqueira [24]. In any case, the rate of shear, time setting and temperature could sensibly affect the flow value justifying, in part, the different values obtained.

Radiopacity represents a physical property that allows the clinician to better evaluate radiographically the sealing quality of endodontic treatment. Despite the constitution of AH presenting some radiopacifier, such as zirconium oxide and calcium tungstate, ES showed the same value in terms of radiopacity, determining both a significant difference when compared to PCS. The calcium silicate sealer used in this study seems to have better radiopacity properties in comparison with the one tested in a recent study [25]. This could be explained by the fact that the presence, absence and amounts of some radiopacifier agents might produce a more or less radiopaque cement [12]. The PCS had a lower value, whereas the previous study reported a higher value in terms of radiopacity [26]. Silver is the principal radiopacifier in zinc oxide eugenol-based sealers and thus the variation in powder/liquid ratio could sensibly modify the amounts of these agents affecting the radiopacity value.

The film thickness standards formulated by ISO 6876 (2012) requirements were widely satisfied from all the sealers tested, showing greater values for AH and ES with respect to PCS. For several years, epoxy resin and zinc oxide eugenol sealers have been considered the gold standard in endodontics [27]. Recently, calcium silicate-based endodontic sealers were proposed as a valid alternative to the traditional widely used sealers [14]. However, all the sealers tested confirmed the data of the manufacturer, respecting the minimum standards required. This in vitro study does not take into account some variables related to the real clinical environment that could alter the physico-chemical performance of the tested sealer. There are some questions indeed about the actual use of the sealers in specific clinical procedures that should be addressed with further research.

Due to the lack of reliable data on clinical outcomes, ex vivo and in vivo studies are required to better evaluate the real performance in long-term clinical outcomes.

However, in vitro studies are the best predictable and repeatable way to evaluate sealer properties. On the contrary, in vivo studies could lead to altered results due to the underestimation of uncontrolled factors. In accordance with this, in vitro studies can be considered as the first level of evidence, essential for an initial evaluation, that should be implemented with ex vivo and in vivo studies.

## CONCLUSION

All tested sealers meet the minimum values required by ISO 6876 (2012) standards except for setting time in the case of AH plus bioceramics. As a rule, a fast-setting time renders technical difficulty during the application, whereas a slow setting time or an incomplete set can result in higher solubility, possibly leading to sealing failure. Future studies are needed to better understand the behaviour of calcium silicate-based sealers under various clinical conditions, focusing on the setting time properties.

### AUTHOR CONTRIBUTIONS

Gaeta Carlo: study concept and design and writing of the original manuscript. Marruganti Crystal: performed statistical analysis, study concept and design. Mignosa Emanuele: laboratory experiment and generation of figures. Malvicini Giulia: data analysis and review of manuscript. Verniani Giulia: performed in vitro experimental data analysis and review of the manuscript. Tonini Riccardo: review of the manuscript. Grandini Simone: study concept and design, data analysis, editing and review of the manuscript.

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### CONFLICT OF INTEREST

The Authors have no fundings and no conflict of interest with the companies present in the current manuscript.

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