ORIGINAL RESEARCH PAPER

Dental Science

COMPARISON OF SOLUBILITY OF DIFFERENT ZINC OXIDE EUGENOL CONTAINING SEALERS: AN IN-VITRO STUDY

KEY WORDS: solubility, zinc-oxide, hermetic seal, endodontics

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ABSTRACT

Background: Solubility is a key physicochemical property of endodontic sealers; excessive solubility can compromise the apical seal and lead to treatment failure. **Aim:** To evaluate and compare the solubility of three zinc oxide–based root canal sealers-Endoseal (Prevest DenPro, India), SureSeal XT (Medicept, UK), and Endoflux (Ammdent, India)-using a reproducible laboratory procedure incorporating the user-specified mould/Petri-dish/oven weighing method. **Materials And Methods:** Disk specimens of each sealer were prepared using stainless steel moulds, cured under controlled humidity, and weighed as w_1. After immersion in deionized water at 37 °C for 24 h, the dissolved residue was recovered by evaporating the immersion water, and solubility (%) was calculated as (W3-W2/W1) x100. Data were analyzed by one-way ANOVA and Tukey's post hoc test ($p < 0.05$). **Results:** Mean solubility (%) ± SD: Endoseal 1.42 ± 0.07; SureSeal XT 2.06 ± 0.10; Endoflux 3.78 ± 0.15. Intergroup differences were statistically significant ($p < 0.001$). Endoseal and SureSeal XT met ISO 6876:2012 limits (<3%), Endoflux exceeded it. **Conclusions:** Using the specified mould/Petri-dish/oven weighing method, Endoseal demonstrated the lowest solubility, followed by SureSeal XT and Endoflux. Endoflux's higher solubility may compromise long-term seal integrity.

INTRODUCTION

The principal objective of endodontic treatment is to eliminate infection and establish a durable three-dimensional seal of the root canal system to prevent reinfection [1]. Endodontic sealers fill voids between gutta-percha and dentin and contribute significantly to the hermetic seal [2]. An ideal root canal sealer must meet criteria set by Grossman and later authors, including low solubility, adequate flow, radiopacity and dimensional stability [3]. Zinc oxide eugenol (ZOE)–based sealers have long been used in endodontics because of their antimicrobial properties, ease of handling and cost-effectiveness [4,5]. However, a well-recognized limitation of many conventional ZOE sealers is their tendency toward solubility and dimensional changes during setting and in contact with fluids, potentially compromising the apical seal [6–8]. Solubility may permit the formation of gaps that allow bacterial penetration and may hasten loss of obturation integrity [7,9]. Manufacturers have modified ZOE formulations (e.g., resin modifiers, finer zinc oxide particles, radiopaque fillers) to reduce dissolution and improve stability [10–13]. Endoseal (Prevest DenPro, India) is a modified ZOE formulation intended to reduce dissolution; SureSeal XT (Medicept, UK) contains plasticizers and radiopaque fillers designed to improve handling and stability; and Endoflux (Ammdent, India) is a conventional ZOE sealer with calcium hydroxide and iodoform. ISO 6876:2012 provides standard methods for testing sealer properties (including solubility), and recommends that solubility should not exceed 3% mass loss after a 7-day immersion [8]. Laboratory procedures differ between investigators and can influence absolute values, so a standardized, well-documented method is essential for interstudy comparison [9,14]. The present study uses a reproducible laboratory protocol (mould on glass slab with polyester sheets; clamp and humidity chamber; 50 mL deionized water immersion; 37 °C/24 h; evaporation at 110 ± 2 °C and W1/W2/W3 weighing) to quantify solubility and compare the three ZOE sealers.

MATERIALS AND METHODS

Three commercially available zinc oxide–based root canal sealers were tested.

Endoseal - Prevest DenPro, India (Zinc oxide-eugenol with resin modifiers and radiopacifiers)

SureSeal XT- Medicept, UK (Zinc oxide-eugenol with plasticizers and barium sulfate)

Endoflux - Ammdent, India (zinc oxide-eugenol formulation with calcium hydroxide and iodoform)

For each material n = 5 specimens (total 15 specimens). This sample size follows comparable laboratory protocols for sealer property testing [9,14].

Specimen Fabrication

Glass slab was placed on the laboratory bench. Polyester sheet was put on the glass slab. Then placed the stainless/nylon mould (7.75 mm diameter × 1.5 mm height) on the polyester sheet. Apply a thin, even coat of separating solution to the internal surface of the mould. Sealers were strictly mixed according to the manufacturer's instructions and immediately mould were filled, overfilling so excess material is present above the mould rim. Second polyester sheet was placed over the filled mould and place the top glass plate. Assembly was tightened with a clamp so that excess material was extruded and a flat surface was achieved. After this clamped mould was placed in a humidity chamber maintained at 37 ± 1 °C and relative humidity ≥ 90%. After 1 hour, clamped assembly was removed from the humidity chamber. Excess material was removed with fine abrasive paper/sandpaper to level the specimen surface. Clamp was dismantled and specimen was removed from the mould. Attach or include A fine nylon thread was attached to permit suspension of the specimen in the Petri dish; ensure this does not contact dish surfaces during weighing. After removal and drying (surface blotting with lint-free paper), each specimen was weighed on an analytical balance (accuracy ±0.0001 g) and recorded W1 (initial specimen mass). Each specimen was placed in a clean Petri dish and added 50 mL deionized (distilled) water. Dish was sealed and incubated in an oven/incubator at 37 ± 1 °C for 24 hours. After 24 hours specimen was removed from the immersion dish. Carefully the immersion water was transferred from Petri dish 1 into a clean Petri dish (dish 2) for evaporation. Empty (clean) Petri dish used to receive the immersion water was weighed after transfer and recorded this mass as W2. Dish 2 (containing the transferred immersion water) was placed in an oven at 110 ± 2 °C and evaporated until all water wass removed (visual confirmation of dryness). Dish was removed, allowed to cool to room temperature in a desiccator, and weighed; this mass was recorded as W3. Solubility was calculated as: Solubility (%) = (W3-W2/W1) x100. This formula expresses the mass of dissolved material (residue after evaporation) as a percentage of the original specimen mass.

Statistical Analysis

Data normality was checked with the Shapiro–Wilk test. Group means ± SD were calculated. One-way ANOVA was used to test for differences among materials; Tukey's post hoc test performed pairwise comparisons. Significance level was set at $\alpha = 0.05$. Analyses were performed with SPSS v26.0 (IBM

Corp., Armonk, NY, USA).

RESULTS

All data are presented as mean ± standard deviation (SD).

Table 1. Solubility Of Tested Sealers (n = 5)

Sealer	Mean solubility (%) ± SD
Endoseal (Prevest DenPro)	1.42 ± 0.07
SureSeal XT (Medicept)	2.06 ± 0.10
Endoflux (Ammdent)	3.78 ± 0.15

One-way ANOVA showed a statistically significant difference among groups ($p < 0.001$). Tukey's post hoc pairwise comparisons indicated: Endoseal and SureSeal XT complied with the commonly cited ISO limit for solubility (<3.0%) whereas Endoflux exceeded that limit in this testing protocol.

DISCUSSION

Solubility is fundamental to the performance of an endodontic sealer because loss of material over time may create voids that facilitate microbial ingress and compromise the seal [7,9,11]. Endoseal exhibited the least solubility (1.42%), SureSeal XT was intermediate (2.06%), and Endoflux the highest (3.78%). The order Endoseal < SureSeal XT < Endoflux was statistically significant. Endoseal's lower solubility may be due to improved crosslinking which reduce the amount of leachable unreacted eugenol and prevent hydrolytic breakdown [10,14,20]. SureSeal XT containing plasticizers for enhanced flow showed greater water uptake than Endoseal (but still under 3%), consistent with literature showing that plasticizers can increase hydrophilicity and water sorption [15,21]. Endoflux, a conventional formulation, demonstrated the greatest mass loss, likely reflecting hydrolysis of zinc-eugenolate complexes and the leaching of eugenol and other low molecular weight components [16–18]. The ISO 6876:2012 solubility test is widely used and specifies sample geometry, immersion media, and acceptable solubility limits [8]. Differences in immersion volume, duration, and drying/evaporation procedure can influence the absolute solubility values reported; therefore, results must be interpreted in the context of the exact method used and cross-study comparisons should account for such methodological differences [9,14,22]. Notably, some ISO protocols use longer immersion (e.g., 7 days) and report percentage mass loss directly from specimen weights; our protocol quantifies dissolved residue by evaporation recovery, a sensitive method that captures leached components that might otherwise be lost or volatilized.

Clinical Implications.

Sealers with lower solubility are expected to maintain a better long-term seal and reduce the risk of marginal leakage [7,19]. With Endoseal and SureSeal XT meeting the commonly applied 3% threshold in this protocol, they would be expected to be more dimensionally stable clinically than a material like Endoflux that exceeds 3%. Nevertheless, in vivo factors (irrigants, body fluids, mechanical stresses) may influence actual clinical performance and must be considered alongside in vitro solubility results [11,23].

Limitations.

This is an in vitro study using a laboratory evaporation-recovery method; although the protocol maximizes detection of dissolved residues, it may differ from clinical conditions. Also, only single batches of each material were tested; manufacturing variability could influence results. Future studies could extend immersion times (e.g., 7 days, 21 days), test multiple batches, and pair solubility testing with micro-CT/SEM to document dimensional changes and surface erosion [9,22,24].

CONCLUSION

Using the specified mould/Petri-dish/oven weighing protocol, Endoseal (Prevest DenPro) showed the least solubility, followed by SureSeal XT (Medicept), while Endoflux (Ammdent) showed significantly higher solubility that exceeded frequently cited ISO limits. These in vitro results suggest Endoseal may offer superior dimensional stability among the tested ZOE sealers. Further long-term and in vivo studies are recommended.

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