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Assessment of Abrasive Containing Irritants on the Bond Strength of Gutta-Percha to Radicular Dentine

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Abstract

Background: Recently, nanoparticles have introduced in endodontic treatment as successful irrigants due to their ultra-small size and high surface area enabled them to penetrate deeply into the dentinal tubules and optimize root canal treatment.

Objective: This study aimed to evaluate the effect of silver nanoparticles and graphene oxide irrigants on apical debris extrusion and push-out bond strength of gutta-percha to radicular dentin, compared to conventional saline and EDTA.

Methods: Forty extracted human maxillary first molars with straight palatal canals were instrumented using ProTaper NEXT rotary files. Samples were randomly assigned to four groups: Group A (saline), Group B (silver nanoparticles), Group C (graphene oxide), and Group D (17% EDTA). Nano-irrigants were ultrasonically activated for five minutes. Debris extrusion was measured using precision weighing of filter discs. Push-out bond strength was assessed in apical, middle, and coronal root sections using a universal testing machine. Statistical analysis of debris extrusion was performed using one-way ANOVA and posthoc Tuckey's tests, while for the overall mean push-out bond strength Kruskal Wallis and Mann-Whitney U tests were performed ($p < 0.05$)

Results: Graphene oxide and silver nanoparticle groups exhibited significantly lower apical debris extrusion compared to EDTA ($p < 0.05$). Push-out bond strength was highest in the apical third across all groups, with graphene oxide showing the greatest overall adhesion, followed by silver nanoparticles, saline, and EDTA.

Conclusion: Silver nanoparticles and graphene oxide irrigants enhanced root canal cleanliness while limiting apical extrusion and increased bonding of gutta-percha. These findings support



the ability of nano-irrigants to effectively alternate conventional solutions in endodontic therapy.

Keywords: Debris extrusion, Endodontic irrigation, Gutta-percha adhesion, Push-out bond strength.

Introduction

Effective root canal treatment requires complete debridement and disinfection of the canal system. This objective depends on presence of the smear layer—a heterogeneous layer of organic and inorganic debris formed during mechanical instrumentation of dentin using hand or rotary instruments (1& 2). This layer may contain bacteria and their metabolic by-products, preventing the penetration of intracanal medicaments and compromising the adaptation of obturation materials to canal walls (3& 4).

The smear layer in root canals has found to be differ significantly from that found in coronal cavities (5& 6). This might attribute to variations in dentinal tubule density, presence of residual pulp tissue, and differences in instrumentation techniques (7&, 8). After instrumentation, microorganisms can penetrate dentinal tubules, and conventional irrigation with sodium hypochlorite alone is insufficient for complete removal of smear layer without adjunctive agents such as EDTA.

McComb and Smith (1975) were the first to characterize the smear layer in endodontics, composed of dentin particles, pulp remnants, odontoblastic processes, and microbial content (9&10). Lester and Boyde (1977) later described it as “trapped organic matter within translocated inorganic dentin,” which gives resistance to sodium hypochlorite (11& 12).

To improve canal cleaning, ultrasonic activation was introduced by Richman in 1957 (13). Ultrasonic activation induces acoustic streaming—a stable, unidirectional fluid motion generated by oscillating instruments—which significantly improves irrigant penetration and debris removal (14–17). Recently, the integration of laser with irrigation, especially ER:YAG provides further root canal disinfection. Published literature from 2022 and 2023 reported improve ultrasonic and laser activation over conventional irrigation, specifically with complicated canal anatomy like isthmuses, lateral canals, and deltas (18–20).

Beside chemical agents, physical abrasives as zirconia and alumina particles combined to irrigation solutions provide promising irrigation improvements due to their optimal grain size and high strength which apply scrubbing effect to canal wall and improve removal of smear layer (21). A systematic review

published in 2022 suggested further investigation for such materials to improve root canal cleaning without compromise dentin integrity (22).

However, most demineralizing irrigants pose a dual challenge: incomplete smear layer removal and reduced adhesion of gutta-percha to radicular dentin, often accompanied by increased apical extrusion of debris (23 24). This study aims to evaluate the potential of abrasive-containing irrigants to achieve effective canal wall cleaning while preserving the bond strength of gutta-percha and minimizing

2. Methods

2.1 Sample Collection and Selection

Forty extracted human maxillary first molars (age range: 18–50 years) were cleaned with distilled water, debrided using a periodontal curette, and polished with pumice. Samples were ultrasonically cleaned for 5 minutes, autoclaved, and stored in 0.1% thymol solution. Radiographs confirmed the presence of a mature apex, absence of resorption, and no prior endodontic treatment.

2.2 Inclusion Criteria

Teeth with a straight palatal root, single canal, mature closed apex, no internal resorption, intact root structure (≥ 15 mm length, apical diameter \leq ISO #15), and no visible cracks were included.

2.3 Sample Preparation

Palatal roots were standardized to 13 mm using a digital caliper and sectioned perpendicular to the long axis with a diamond disc under water coolant. Using a dental surveyor to ensure axial parallelism, each sample was embedded in silicone putty within a plastic tube. A reference line was marked 3 mm apical to the coronal boundary. After setting, tubes were stabilized in a bench vice.

2.4 Root Canal Instrumentation

Gates-Glidden drills (#1 and #2) used first for canal orifices flaring. Working length was set by subtracting 0.5 mm from the length at which a K-file #10 exited the apex. After ensuring a loose K-file #15, canals were prepared using ProTaper NEXT rotary NiTi files (X1–X4) at 300 rpm and 2.0 Ncm torque. Each file set was used for five canals and discarded. Irrigation with 1 ml normal saline followed each file, and apical patency was verified with a K-file #10. Irrigation needles were placed no deeper than 2 mm short of working length.



2.5 Irrigation Protocol and Grouping

Samples were randomly divided into four groups for final irrigation:

- **Group A:** Normal saline only
- **Group B:** Silver nanoparticles (100 ppm in saline) + ultrasonic activation (5 min)
- **Group C:** Graphene oxide solution (0.1% w/v in saline) + ultrasonic activation (5 min)
- **Group D:** 17% EDTA

One minute ultrasonic flush with normal saline for all nano-irrigant groups after activation was performed to remove residual particles.

2.6 Debris Extrusion Test

Each tooth was mounted in a two-sided filter disc and weighed (Sartorius, Germany; precision: 0.0001 g) before and after instrumentation. Weight was recorded at three stages: pre-irrigation, post-irrigation, and post-evaporation. Debris extrusion was assessed by the difference in filter weight.

2.7 Push-Out Bond Strength

After irrigation, canals were obturated using a single gutta-percha cone with bioceramic sealer. Each root was sectioned into three 4-mm cylinders (apical, middle, coronal). Push-out test was performed using a plugger slightly smaller than the apical diameter. Force at bond failure (N) was recorded, and bond strength (MPa) was calculated using the following formula:

$$\text{Adhesive Surface Area} = \frac{(D_1 + D_2)}{2} \times \pi \times h$$

With D_1 = apical diameter, D_2 = coronal diameter, and h = specimen thickness (25).

2.8 Statistical Analysis

Data were analyzed using SPSS v26. Normality was assessed via Shapiro-Wilk test. Debris extrusion was compared using one-way ANOVA and Tukey's post hoc test. Push-out bond strength was evaluated using Kruskal-Wallis and Mann-Whitney U tests. Significance was set at $p < 0.05$

3. Results

3.1 Debris Extrusion

Quantitative analysis of debris extrusion revealed statistically significant differences among the four groups ($p < 0.05$). Group A (saline only) showed the highest mean of debris extrusion, followed by Group D (EDTA). Groups B and C, which received nano-irrigants with ultrasonic activation, showed markedly less debris extrusion.

- Group A (Saline): 0.0123 ± 0.0021 g^{a, b}
- Group B (Silver nanoparticles): 0.0065 ± 0.0014 g^{a, c}
- Group C (Graphene oxide): 0.0059 ± 0.0012 g^{b, d}
- Group D (EDTA): 0.0098 ± 0.0017 g^{c, d}

Identical superscript lowercase letters indicate statistically significant differences between the related groups.

Post hoc Tukey test confirmed that Groups B and C had statistically significant lower extrusion compared to Groups A and D ($p < 0.01$).

3.2 Push-Out Bond Strength

Push-out bond strength values were interpreted in megapascals (MPa) and analyzed across apical, middle, and coronal sections. Group C (graphene oxide) showed the highest mean overall bond strength, followed closely by Group B (silver nanoparticles). Group D (EDTA) illustrated reduced bond strength, while Group A (saline) had the lowest mean values as shown in table 1.



Table 1: apical, middle, coronal, and overall mean push-out bond strength of the tested groups measured in MPa along with statistical tests.

Group	Apical (MPa)	Middle (MPa)	Coronal (MPa)	Overall Mean (MPa)
A (Saline)	2.31 ± 0.42	3.12 ± 0.51	3.45 ± 0.48	2.96 ± 0.47 ^{a, b}
B (Silver NP)	3.45 ± 0.39	4.12 ± 0.44	4.38 ± 0.41	3.98 ± 0.41 ^a
C (Graphene Oxide)	3.67 ± 0.36	4.35 ± 0.40	4.62 ± 0.38	4.21 ± 0.38 ^{b, c}
D (EDTA)	2.89 ± 0.40	3.65 ± 0.45	3.92 ± 0.43	3.49 ± 0.43 ^c

Identical superscript lowercase letters indicate statistically significant overall mean differences between groups using Mann-Whitney U test.

Kruskal-Wallis and Mann-Whitney U tests resulted statistically significant differences between groups ($p < 0.05$), with Group C showed highest bond strength.

4. Discussion

Post-endodontic complications as apical periodontitis, periapical tissue inflammation, and post-operative pain are directly related to apical extruded debris; therefore, the focus is to minimize extrusion of debris to reduce these complications. Apical extruded debris are influenced by different factors as variations of canal anatomy, canal preparation technique, enlargement of apical foramen, type of irrigants, and mode of activation (15& 24). In this study, straight palatal roots of maxillary molars were used trying to control these variables and reduce bias.

Recently, there are interests in combine endodontic irrigations and nanoparticle technology because of its unique properties in enhancement smear layer removal, disinfection, and compatibility with obturation materials. (26& 27). This study used irrigation solutions containing silver nanoparticles and graphene oxide to evaluate their effects on apical debris extrusion and push-out bond strength compared to normal saline and EDTA irrigants.

As shown by the results, both groups B and C (nanoparticle irrigant groups) are associated with lower apical debris extrusion than groups A and D with the least debris related to graphene oxide group (group C) followed closely by silver nanoparticles group (group B). This could be due to high surface tension and viscosity of graphene oxide that limits apical flow of this irrigation solution. On the opposite, EDTA group (group D) showed the highest mean of debris extrusion which could be related to chelation action and low surface tension keeping

patency of apical foramen and facilitates debris passage (21). The present findings agreed with previous study observations on irrigation surface tension and penetration of canal dentin (28), and supported by other previous observations regarding greater extrusion with EDTA over gel formulations (29).

The results of push-out bond strength of all groups showed variations across root sections with the higher values reported in apical third of within all groups which could be explained by smaller canal diameter apically and good sealer adaptation that increase frictional resistance (30). The highest overall mean push-out bond strength was resulted for graphene oxide containing group (group C) followed by silver nanoparticle, saline, and EDTA respectively (27& 31). Push-out bond strength was also improved within silver nanoparticles containing group that could be attributed to their antimicrobial action and surface modifying-effects which facilitates sealer adhesion (32).

On the other hand, EDTA group was associated with lowest mean bond strength which could be due to its aggressive chelation behavior and depletion of calcium from dentinal tubules (12). The present findings are opposing with Braz et al. (2018), who stated that there are no difference among different irrigation solutions, likely because of the use of EndoVac and ultrasonic activation (33).

Bond strength was highest in the apical third across all groups, followed by the middle and coronal sections. This gradient may reflect variations in dentinal tubule density, sealer thickness, and instrumentation effects (21). Ultrasonic activation, while enhancing smear layer removal, may reduce coronal bond



strength by overexposing dentin and reducing mechanical retention (34& 35).

This study and its promising results have encountered some limitations. As an in vitro study, this study cannot simulate the oral cavity and its complicated conditions as periapical pressure and long term stability of nanoparticles under function; therefore, additional clinical trials and studies should be conducted to determine the long term biocompatibility and antimicrobial effects of these nanoparticles containing irrigation solutions.

5. Conclusion

Within the limitations of this in vitro study, it can be concluded that the outcome of root canal treatment could be enhanced by using nanoparticles combined to irrigation solution as they could reduce apical debris extrusion and improve obturation material adaptation to root canal dentin. These nano-irrigants provide effective cleaning with minimum extrusion of debris reduce the risk of flare-ups and post-operative complications.

6. Ethical approval

An *in-vitro* study.

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