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Adhesive performance and failure modes of hydroxyapatite coated gutta percha with different root canal sealers

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This study evaluates the adhesive performance of a novel hydroxyapatite-coated gutta-percha (HAGP) in combination with various root canal sealers, using push-out bond strength testing and failure mode analysis. Eighty human single-canal teeth were divided into four groups: three test groups obturated with HAGP using AH Plus, iRoot SP, or GuttaFlow Bioseal, and one control group obturated with conventional gutta-percha (GP) with AH Plus sealer. Push-out bond strength was assessed after 30 days with a universal testing machine, and failure modes were analysed under stereomicroscopy. Results revealed a significantly higher bond strength ($p < 0.001$) for HAGP compared to the control, regardless of the sealer used. Failure analysis indicated 62.5% mixed failures and 37.5% cohesive failures, with no adhesive failure observed. These findings suggest that HAGP significantly improves gutta-percha adhesion, as demonstrated through the higher bond strength compared to conventional gutta-percha.

Keywords Gutta-percha, Hydroxyapatite, Root canal filling materials, Push-out bond strength

The primary objective of endodontic treatment is to eradicate bacteria and maintain the tooth in a disinfected state, thereby preventing bacterial ingress[1]. Therefore, the root canal must be hermetically sealed to hinder communication between the root canal system and the periapical tissues. Gutta-percha (GP) is the standard obturation core material, with major advantages including plasticity, ease of manipulation, minimal toxicity, radiopacity, and ease of removal using solvents or heat. However, GP alone cannot fully obturate the root canal space, as it does not bond to radicular dentine and therefore requires a sealer to achieve a hermetic seal[2].

Root canal sealers should fill the gaps that may allow microleakage of fluids between the filling and the canal wall under static conditions[3]. Sealers are also required to resist dislodgement of the root filling material in dynamic conditions such as tooth flexure, post-space preparation, or operative procedures. Hence, adhesion is a desirable physical property of root canal sealers[4]. It has therefore been recommended that GP be combined with a sealer to achieve improved sealing of the root canal system[5].

Selecting an appropriate endodontic sealer is crucial for the long-term success of non-surgical root canal treatment[6]. To overcome the limitation of inadequate adhesion of GP, a novel approach was introduced by coating conventional GP cones with materials that capable of bonding to the sealer. Coating the non-bondable GP surface with such materials creates an additional circumferential interface, rendering them bondable to root canal sealers. Multiple coated GP products have been introduced to achieve the monoblock concept.

A novel apatite calcium phosphate (Hydroxyapatite)-coated GP has been developed by immersing the substrate into simulated body fluid (SBF) solution[7]. This coating is proposed to enhance surface properties of GP as a root canal filling material, due to its compositional similarity to the hydroxyapatite of radicular dentine[7]. This similarity may increase the chemical adhesion to sealers that bond chemically to radicular dentine, such as glass ionomer-based sealers[8]. Furthermore, the roughened coating surface may facilitate penetration and mechanical interlocking of root sealer particles with the coated GP surface[7]. The hydroxyapatite-coated GP has shown promising results in terms of bond strength, particularly when used with bioceramic sealers[9].

Several types of sealers are available. Resin-based sealers provide adhesion to radicular dentine and do not contain eugenol. AH Plus (Dentsply, Konstanz, Germany) is an epoxy resin sealer, developed as a modified

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formulation of AH-26 in which formaldehyde is not released[10]. Its hydrophobic nature allows it to react with exposed amino groups in collagen, forming covalent bonds when the epoxide ring opens. It has been shown to achieve high bond strength to both radicular dentine and GP, suggesting that the resin can react with both substrates[11]. Due to its excellent properties, such as low solubility, slight expansion, strong adhesion to dentine, and very good sealing ability; AH Plus is considered the benchmark "gold Standard"[12,13].

Bioceramic-based sealers have favourable physicochemical and biological properties, including bioactivity and biomineralization. In addition, they have been found to increase the in vitro fracture resistance of endodontically treated roots, particularly when used in conjunction with Activ GP cones[14]. ROEKO GuttaFlow Bioseal (Coltene/Whaledent, Altstatten, Switzerland) is a novel formulation consisting of polydimethylsiloxane-based GP incorporated with calcium silicate particles. It demonstrated higher biocompatibility compared to AH Plus, as it promotes cementoblast differentiation of human periodontal ligament stem cells even in the absence of growth factors[15]. iRoot SP (Innovative BioCeramix Inc, Canada) is a calcium phosphate silicate-based cement whose major inorganic components include tricalcium silicate, dicalcium silicate, calcium phosphates, colloidal silica, and calcium hydroxide. It uses zirconium oxide as the radiopacifier and contains water-free thickening vehicles, enabling delivery as a premixed paste.

The aim of this study is to evaluate the HAGP adhesion by assessing its push-out bond strength to radicular dentine in the presence of different sealers. The null hypothesis is that there is no significant difference in the push-out bond strength of HAGP to radicular dentine when used with three types of sealer (AH Plus, iRoot SP, and GuttaFlow Bioseal), compared to uncoated GP and AH Plus.

Materials and methods

This research was approved by the Medical Ethics Committee, Faculty of Dentistry, University of Malaya (Reference No. DF RD 2001/0001). Accordingly, the use of human extracted teeth, and all related methods were conducted in full accordance with our institutional guidelines and regulations. Sample size calculation was performed using G Power version 3.1.9.7. Eighty extracted teeth were collected from the Unit Pakar Ortodontik, Klinik Pergigian Cahaya Suria, Kuala Lumpur, Malaysia, following informed consent. The teeth were disinfected using a 0.5% chloramine-T trihydrate solution for one week.

Inclusion criteria for the study were as follows: fully formed root canals measuring at least 16 mm, relatively straight single-canal with curvatures less than 15°, a patent foramen, and a first binding file ≤ #20. Radiographs were used to confirm the root canal curvature.

The anatomical crowns were removed with a separating disc at the level of the cemento-enamel junction perpendicular to the long axis of the root canal to the standard root canal length for all specimens (16 mm). Following that, each tooth was mounted in an impression compound to facilitate handling during root canal preparation and root canal obturation. Root canal preparation was performed using rotary ProTaper Next files (PTN; Dentsply Tulsa Dental, Tulsa, OK) until size X3, along with intermittent irrigation using 5.25% sodium hypochlorite (NaOCl). After preparation, the root canals were irrigated with 17% ethylenediaminetetraacetic acid (EDTA) for 1 min, followed by rinsing with 10 mL of distilled water. Samples were randomly divided into 4 groups with 20 samples each.

- Group 1: Conventional GP + AH Plus (Dentsply, Konstanz, Germany).
- Group 2: HAGP + AH Plus.
- Group 3: HAGP + iRoot SP (Innovative BioCeramix Inc, Canada).
- Group 4: HAGP + GuttaFlow Bioseal (Coltene, Switzerland).

The single cone obturation technique was performed using ProTaper Next GP cone size #30/0.07 (X3, ProTaper NEXT, Dentsply, Maillefer, USA) for the first group. For the other three groups, HAGP was used for obturation after coating ProTaper Next GP cone size #30/0.07 (X3, ProTaper Next, Dentsply, Maillefer, USA) with hydroxyapatite[7].

Each root was mounted using epoxy resin (Mirapox 950 – 230 A/B; Miracon Sdn Bhd, Malaysia), and sectioned perpendicular to the root canal with a sintered diamond wafering blade (Struers, Ballerup, Denmark) at low speed under constant water cooling. A 1 mm-thick mid-root dentine section was obtained at a level ensuring a main cone diameter greater than 0.5 mm. As ProTaper Next GP cone size X3 has a tip diameter of 0.3 mm and 0.07 taper, its cross-sectional diameter increases by 0.07 mm per 1 mm from the tip. Therefore, to yield a sample with more than 0.5 mm of apical diameter, 3 mm of the apical portion was sectioned and discarded before sectioning 1 mm thick of the sample. One sample was taken from each tooth.

Both the apical and coronal aspects of each sample were photographed and examined before testing to confirm a circular canal shape and that the GP filled the entire canal space. By using 2.5x magnification dental loupes, a 0.5-mm cylindrical stainless-steel plunger was positioned to cover the GP cone without contacting canal walls. The plunger was mounted in the upper part of a Universal Testing Machine (Shimadzu Corporation, Kyoto, Japan). Subsequently, samples were aligned over a 1 mm-diameter circular opening in a 10 mm-thick Perspex plate and mounted in an apical to coronal direction to avoid any constriction interference due to root canal taper during push-out testing. The tests were conducted at a cross-head speed of 0.5 mm/min using a 100 N load cell set at 50 N maximum loads. The highest value recorded was taken as the force in Newtons. Photographs of both sides of the samples were taken to check for anomalies. The thickness of the specimens was measured using a digital calliper (Mitutoyo Corporation, Kawasaki, Japan) to within 0.01 mm. Push-out bond strength (MPa) was calculated from force (N) divided by area (mm^2) according to the following formula:

Group	Mean bond strength (MPa) ± SD	*p*-value
GP/AH Plus [a]	2.02 ± 0.63	< 0.001*
HAGP/AH Plus [b]	4.55 ± 0.56	
HAGP/iRoot SP [b]	4.90 ± 0.66	
HAGP/GuttaFlow Bioseal [b]	4.49 ± 0.58	

Table 1. Mean bond strength of different groups to radicular dentine in MPa. *Significant difference $p < 0.05$. Different small letters indicate significant difference between groups.

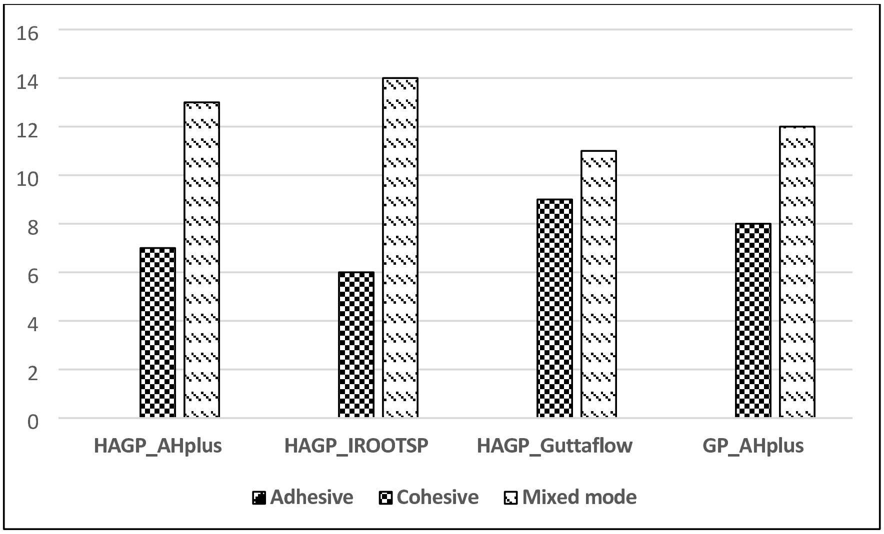

Fig. 1. Distribution of failure modes in the different HAGP groups.

$$\text{Debonded Area}\left(\text{mm}^2\right) = 1/2 \times [\text{Apical } 2\pi r_1\,(\text{mm}) + \text{Coronal } 2\pi r_1\,(\text{mm})] \times \text{Thickness}\,(\text{mm})$$

$$\text{Bond Strength}\ \ (\text{Mpa}) = \text{Force}\,(\text{N}) \div \text{Area}\left(\text{mm}^2\right)$$

Failure modes were assessed under a stereomicroscope at 56X magnification and classified as adhesive, cohesive, or mixed. Assessments were conducted twice by the same investigator at one-month intervals. Intra-observer reliability was analysed using Cohen's kappa coefficient.

Statistical analyses were performed with SPSS version 24 (SPSS Inc., USA). Data normality was verified using the Shapiro-Wilk test. One-way ANOVA and Tukey's post hoc tests were applied for bond strength comparisons. Associations between failure mode and filling material were examined with chi-square analysis. Statistical significance was set at $p < 0.05$.

Results

Table 1 presents the mean push-out bond strength for different groups. The highest mean bond strength (4.90 ± 0.66 MPa) was observed in Group 3 (HAGP/iRoot SP), while the lowest was in Group 1 (GP/AH Plus) (2.02 ± 0.63 MPa).

A one-way ANOVA revealed a significant difference among groups ($p < 0.001$). Post hoc comparisons using Tukey's test showed that Group 1 exhibited significantly lower bond strength than all other groups ($p < 0.001$). No statistically significant differences were detected among Groups 2, 3, and 4 ($p > 0.05$).

Intra-observer agreement on failure mode classification was high ($\kappa = 0.95$). Figure 1 illustrates the distribution of failure modes across groups. Mixed failures predominated: 60% in Group 1, 65% in Group 2, 70% in Group 3, and 55% in Group 4. The remaining specimens exhibited cohesive failures. No adhesive failures were observed. Chi-square analysis indicated no significant association between filling material and failure mode ($p > 0.05$).

Discussion

The current study assessed the push-out bond strength of HAGP with three sealers and compared it to a control group of uncoated conventional GP with AH Plus sealer. The mean push-out bond strength of the control group was significantly lower when compared to the various HAGP groups. Therefore, the null hypothesis that there is no significant difference between the groups was rejected.

The AH Plus sealer served as a positive control. It is an epoxy-resin-based sealer known for its high bond strength when used with conventional GP[16–20]. This is attributed to its ability to form a covalent bond by opening its epoxide ring, which reacts with the exposed amino groups of the collagen matrix of radicular dentine[11]. Additionally, its flowability promotes deeper penetration into dentinal tubules, enhancing mechanical interlocking between the sealer and dentine[21]. Its low shrinkage upon setting and long-term dimensional stability also contribute to its superior bond strength.

In the current study, the HAGP/AH Plus group demonstrated significantly higher push-out bond strength than GP/AH Plus, suggesting potential bonding between the sealer and the hydroxyapatite coating of HAGP. By contrast, the lower push-out bond strength in the GP/AH Plus group can be explained by the fact that conventional GP does not bond to either radicular dentine or sealers[6,9].

The HAGP groups with iRoot SP and GuttaFlow Bioseal showed comparable push-out bond strength to HAGP/AH Plus. These findings are partially consistent with previous studies using conventional GP, where iRoot SP demonstrated bond strength comparable to AH Plus[22]. However, in line with earlier reports, GuttaFlow generally exhibits lower bond strength than AH Plus[23].

The overall improvement in push-out bond strength among all HAGP groups compared to conventional GP can be attributed to the hydroxyapatite coating of HAGP, confirmed by the presence of hydroxyl groups in earlier research[7]. Furthermore, the surface roughness of the apatite-calcium phosphate coating increases the bonding surface area, thereby enhancing adhesion. These irregularities might allow penetration of the sealer particles into the coating layer of the GP, leading to micromechanical retention of the sealers and hence a high bond strength[9].

The push-out strength test was selected because it is widely recognized for evaluating adhesion between obturation materials and root canal walls, due to its simplicity, reproducibility, and reliability[15,19–21,24,25]. In the push-out test, fracture occur parallel to the dentine-sealer interface, making it a true shear test for parallel-sided samples and a better measure of bond strength than conventional shear tests[26,27]. Push-out strength reflects the combined effects of friction between materials and canal walls, molecular bonding forces, and chemical adhesion to radicular dentine[23]. It is also influenced by factors such as friction[28]C factor[29,30] and root canal treatment protocols[31].

The thin-slice push-out method is a reliable technique for evaluating the bond strength of root canal filling materials to radicular dentine[29] and to evaluate 1 mm-thick samples[32]. It was chosen in the current study over tensile and shear strength tests because it is less sensitive to sample variation and to the variations in stress distribution during load application. Additionally, it allows easy alignment of specimens during testing[20].

Root sections in the current study were taken from the mid-root, 3 mm from the apical portion, ensuring a cone diameter greater than 0.5 mm to match the plunger size (0.5 mm) and reduce variables affecting bond strength. Earlier studies reported that the different sizes of the plunger used to push out the obturation material from different levels of the roots (apical, middle, and coronal) can influence the bond strength of the root sealer[19,33]. Nevertheless, when the same plunger size is used for that purpose, the bond strength does not significantly vary between the root levels[34].

The predominance of mixed failure modes in HAGP groups was consistent with findings by Al-Haddad et al.[9], where HAGP with bioceramic sealer had similar results. This suggests an equivalent bond strength of the sealer to radicular dentine as well as to HAGP due to the similarity of the components in both the hydroxyapatite coating and the components of the inorganic radicular dentine (70% minerals by weight and a Ca/P molar ratio of 1.53 compared to the 1.67 Ca/P molar ratio of pure hydroxyapatite)[35].

The present study has limitations, including the fact that it is an in vitro study with strict inclusion criteria. Further research should investigate a wider range of variables, such as testing HAGP in curved canals, and employ advanced analytical methods; including Fourier Transform Infrared Spectroscopy (FTIR) and X-ray diffraction (XRD); to better elucidate the bonding mechanism.

Conclusions

Within the limitations of the current in vitro study, it can be concluded that HAGP, regardless of the sealer used (AH Plus, iRoot SP, or GuttaFlow Bioseal), demonstrated significantly higher bond strength than conventional GP. The predominant failure modes were mixed and cohesive, with no adhesive failures observed.

Data availability

The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

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References

1. Schilder, H. Cleaning and shaping the root canal. *Dent. Clin. North Am.* **18**, 269 (1974).
2. Skinner, R. L. & Himel, V. T. The sealing ability of injection-molded thermoplasticized gutta-percha with and without the use of sealers. *J. Endod.* **13**, 315 (1987).
3. Ørstavik, D., Eriksen, H. M. & Beyer-Olsen, E. M. Adhesive properties and leakage of root canal sealers in vitro. *Int. Endod. J.* **16**, 59 (1983).
4. Grossman, L. I. Physical properties of root canal cements. *J. Endod.* **2**, 166 (1976).
5. Pascon, E. A. & Spngberg, L. S. W. In vitro cytotoxicity of root canal filling materials: 1. Gutta-percha. *J. Endod.* **16**, 429 (1990).
6. Lee, M., Winkler, J., Hartwell, G., Stewart, J. & Caine, R. Current trends in endodontic practice: Emergency treatments and technological armamentarium. *J. Endod.* **35**, 35 (2009).
7. Al-Haddad, A., Kutty, M. G., Abu Kasim, N. H. & Che Ab Aziz, Z. A. Physicochemical properties of calcium phosphate based coating on gutta-percha root canal filling. *Int. J. Polym. Sci.* **2015**, 414521 (2015).
8. Weiger, R., Heuchert, T., Hahn, R. & Löst, C. Adhesion of a glass ionomer cement to human radicular dentine. *Dent. Traumatol.* **11**, 214 (1995).

9. Al-Haddad, A. Y., Kutty, M. G. & Che Ab Aziz, Z. A. Push-out bond strength of experimental apatite calcium phosphate based coated gutta-percha. *Int. J. Biomater.* **2018**, 1731857 (2018).
10. Leonardo, M. R., Da Silva, L. A. B., Filho, M. T. & Da Silva, R. S. Release of formaldehyde by 4 endodontic sealers. *Oral Surg. Oral Med. Oral Pathol. Oral Radiol. Endod.* **88**, 221 (1999).
11. Tagger, M., Tagger, E., Tjan, A. H. L. & Bakland, L. K. Measurement of adhesion of endodontic sealers to dentin. *J. Endod.* **28**, 351 (2002).
12. Donnelly, A. et al. Water sorption and solubility of methacrylate resin-based root canal sealers. *J. Endod.* **33**, 990 (2007).
13. Schäfer, E., Bering, N. & Bürklein, S. Selected physicochemical properties of AH Plus, EndoREZ and RealSeal SE root canal sealers. *Odontology* **103**, 61 (2015).
14. Ghoneim, A. G., Lutfy, R. A., Sabet, N. E. & Fayyad, D. M. Resistance to fracture of roots obturated with novel canal-filling systems. *J. Endod.* **37**, 1590 (2011).
15. Rodríguez-Lozano, F. J. et al. GuttaFlow Bioseal promotes spontaneous differentiation of human periodontal ligament stem cells into cementoblast-like cells. *Dental Mater.* **35**, 114 (2019).
16. Abada, H. M., Farag, A. M., Alhadainy, H. A. & Darrag, A. M. Push-out bond strength of different root canal obturation systems to root canal dentin. *Tanta Dent. J.* **12**, 185 (2015).
17. Fisher, M. A., Berzins, D. W. & Bahcall, J. K. An in vitro comparison of bond strength of various obturation materials to root canal dentin using a push-out test design. *J. Endod.* **33**, 856 (2007).
18. Gesi, A. et al. Interfacial strength of Resilon and gutta-percha to intraradicular dentin. *J. Endod.* **31**, 809 (2005).
19. Sagsen, B., Ustün, Y., Demirbuga, S. & Pala, K. Push-out bond strength of two new calcium silicate-based endodontic sealers to root canal dentine. *Int. Endod. J.* **44**, 1088 (2011).
20. Ungor, M., Onay, E. O. & Orucoglu, H. Push-out bond strengths: The epiphany-resilon endodontic obturation system compared with different pairings of epiphany, resilon, AH plus and gutta-percha. *Int. Endod. J.* **39**, 643 (2006).
21. Carneiro, S. M. B. S. et al. Push-out strength of root fillings with or without thermomechanical compaction. *Int. Endod. J.* **45**, 821 (2012).
22. Ersahan, S. & Aydin, C. Dislocation resistance of iRoot SP, a calcium silicate-based sealer, from radicular dentine. *J. Endod.* **36**, 2000 (2010).
23. Dem, K. et al. The push out bond strength of polydimethylsiloxane endodontic sealers to dentin. *BMC Oral Health* **19**, 181 (2019).
24. Yap, W. Y., Che Ab Aziz, Z. A., Azami, N. H., Al-Haddad, A. Y. & Khan, A. A. An in vitro comparison of bond strength of different sealers/obturation systems to root dentin using the push-out test at 2 weeks and 3 months after obturation. *Med. Princ. Pract.* **26**, 464 (2017).
25. Drs, A., Koch, K., Brave, D. & Nasseh, A. A. A review of bioceramic technology in endodontics. *CE Artic. Bioceram. Technol.* **10**, 6 (2013).
26. Üreyen Kaya, B., Keçeci, A. D., Orhan, H. & Belli, S. Micropush-out bond strengths of gutta-percha versus thermoplastic synthetic polymer-based systems—An ex vivo study. *Int. Endod. J.* **41**, 211 (2008).
27. Drummond, J. L., Sakaguchi, R. L., Racean, D. C., Wozny, J. & Steinberg, A. D. Testing mode and surface treatment effects on dentin bonding. *J. Biomed. Mater. Res.* **32**, 533 (1996).
28. Soares, C. J. et al. Finite element analysis and bond strength of a glass post to intraradicular dentin: Comparison between microtensile and push-out tests. *Dent. Mater.* **24**, 1405 (2008).
29. Goracci, C. et al. The adhesion between fiber posts and root canal walls: Comparison between microtensile and push-out bond strength measurements. *Eur. J. Oral Sci.* **112**, 353 (2004).
30. Pane, E. S., Palamara, J. E. A. & Messer, H. H. Critical evaluation of the push-out test for root canal filling materials. *J. Endod.* **39**, 669 (2013).
31. Patil, A. S., Dodwad, K. P. & Patil, A. A. An in vitro comparison of bond strengths of Gutta-percha/AH Plus, Resilon/Epiphany self-etch and EndoREZ obturation system to intraradicular dentin using a push-out test design. *J. Conserv. Dent.* **16**, 238 (2013).
32. Sönmez, I. Ş, Sönmez, D. & Almaz, M. E. Evaluation of push-out bond strength of a new MTA-based sealer. *Eur. Arch. Paediatr. Dent.* **14**, 161 (2013).
33. Nagas, E., Uyanik, O., Durmaz, V. & Cehreli, Z. C. Effect of plunger diameter on the push-out bond values of different root filling materials. *Int. Endod. J.* **44**, 950 (2011).
34. Bouillaguet, S. et al. Alternative adhesive strategies to optimize bonding to radicular dentin. *J. Endod.* **33**, 1227 (2007).
35. De Dios Teruel, J., Alcolea, A., Hernández, A. & Ruiz, A. J. O. Comparison of chemical composition of enamel and dentine in human, bovine, porcine and ovine teeth. *Arch. Oral Biol.* **60**, 768 (2015).

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Author contributions

N.A: Supervision, Methodology, Conceptualization. N.M: Supervision, Formal analysis, Conceptualization. A.A: Writing – review & editing, Conceptualization. R.D.: Writing – original draft, review & editing. F.M.: Investigation, Data curation, Writing – original draft. Z.C: Supervision, Funding acquisition, Conceptualization.

Declarations

Competing interests

The authors declare no competing interests.

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