

Original Article

The comparative evaluation of various additives on setting time and compressive strength of MTA Plus: An *in vitro* study

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ABSTRACT

Background and Objectives: Mineral trioxide aggregate (MTA), being a near-ideal retrograde filling material, has the greatest disadvantage of longer setting time, which limits its use in a certain clinical situation. A novel MTA, known as MTA Plus, claims to have a finer particle size and is marketed with an additional anti-washout gel. This study was undertaken to compare the setting time and compressive strength (CS) of MTA Plus with various additives.

Materials and Methodology: MTA Plus powder was mixed with following additives such as 5% CaCl₂ solution, 10% CaCl₂ solution, phosphate-buffered saline, 80% distilled water/20% propylene glycol, saline, lidocaine HCl, 15% sodium phosphate monobasic, 3% sodium hypochlorite gel, and proprietary gel with MTA Plus in the ratio of 3:1 by weight. Setting time was evaluated using Vicat apparatus using a brass mold with an internal diameter of 10 mm and height of two millimeters. CS was evaluated using an Instron machine using a split brass mold with an internal diameter of Four millimeters and height of Six millimeters.

Results: The results showed that 15% sodium hydrogen phosphate and MTA Plus gel significantly decrease the setting time of MTA Plus. MTA Plus gel set cement gave the highest value of CS among all groups of additives studied.

Interpretation and Conclusion: Additives used in the given study had an influence on physical property like setting time and mechanical property like CS of MTA Plus.

Keywords: Additives, anti-washout gel, compressive strength, mineral trioxide aggregate Plus, setting time

INTRODUCTION

The prime objective of an endodontic procedure resides in obtaining hermetic seal between root canal space and periapical tissues.^[1] When it is difficult to achieve this objective by an orthograde approach, then a retrograde approach is often employed. An “ideal” retrograde filling material should provide an airtight seal. It should be nontoxic, noncarcinogenic, maintain a biological harmony with the host tissues, should be indissoluble in tissue fluids, and be dimensionally stable.^[2] Materials such as glass-ionomer

cement and gold foils were tested as potential root-end materials, but these were found to be technique sensitive and prone to moisture contamination. Newer materials such as CPC-calcium phosphate cement (hydroxyapatite cement) have demonstrated excellent biocompatibility. Mineral trioxide aggregate (MTA) has shown good sealing ability, excellent biocompatibility, and radiopacity. Biodentine has properties similar to that of MTA, but with lower dentin sealing ability

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and radiopacity compared to MTA. MTA was launched initially as a retrograde filling material^[3] and eventually was used for vital pulp therapy,^[4] apexification, apexogenesis, apical barrier formation in open apices, root perforations, and resorption repair,^[5,6] as a root canal filling material. While ProRoot MTA (Dentsply Tulsa Dental) claims to set in 2–3 h, MTA Angelus (Angelus, Londrina, Brazil) was reported to set in 15 min.^[7] Accelerated setting of MTA is often required by the clinician to place the material properly and completes the restorative procedures without worrying about its washout. Two genres of MTA, namely gray and white, are available. Due to excessive staining caused by the gray genre, the white version of MTAs was introduced into the market in 2002.^[8] MTA Plus is a new variant of MTA, marketed by Prevest Denpro, Jammu, India (for Avalon Biomed Inc., Bradenton, FL, USA) company. It proclaims to be cost-effective as compared to other MTAs and provides finer particle size 15 μm (>50% of the particles ranged < 1 μm) and an anti-washout gel^[9] along with water for dispensing the given material. The major disadvantage of MTA is its longer setting time. MTA is reported to set in three to four hours (mixed in 3:1 ratio). Some clinical situations like use as a retrograde filling material require a faster set, to protect the integrity of MTA to avoid washout with tissue fluids, during the setting period. This disadvantage can be overcome by mixing different additives with MTA Plus which can improve the setting time by altering the physical properties of the set cement. In the clinical scenario, an accelerated setting time can lead to potential clinical advantages such as reduced washout and a lower possibility of blood or serum contamination during setting while performing periapical surgeries. Considering the clinical applications of MTA, compressive strength (CS) of this material is important in some cases, for example, when MTA is used to repair furcal perforations where it must tolerate occlusal forces and the placement of restorative material on it. CS of hydraulic cement is an indicator of hydration reaction and, in fact, indirectly is a reflex of the setting process of the material.^[10] No studies have been done to evaluate the setting time and CS of MTA Plus using different additives so far. Thus, the aim of this *in vitro* study was to compare the setting time and CS of the MTA Plus with various additives. The null hypothesis of this study was that none of the additives could decrease the setting time and improve the CS of MTA Plus.

MATERIALS AND METHOD

This study was conducted in the Department of Oral Pathology and Microbiology at SDM College of Dental Sciences and Hospital, Dharwad. The materials used were MTA Plus powder and various additives such as 5% CaCl_2 solution, 10% CaCl_2 solution, 80% distilled water/20%

propylene glycol, phosphate-buffered saline solution, saline, lidocaine HCl, Lignox® 2%, 15% sodium phosphate, 3% sodium hypochlorite gel, and proprietary gel with MTA Plus. The samples were divided into nine groups. The groups were as follows: Group A, 5% CaCl_2 solution; Group B, 10% CaCl_2 solution; Group C, phosphate-buffered saline solution; Group D, 80% distilled water/20% propylene glycol; Group E, saline; Group F, lidocaine HCl Lignox 2%; Group G, 15% sodium phosphate; Group H, 3% sodium hypochlorite gel; and Group I, proprietary gel with MTA Plus. The armamentarium used was brass ring mold (internal diameter of 10 mm and a height of 2 mm), brass split mold (internal diameter of 4 mm and a length of 6 mm), spatulas, paper pads, glass slabs, Vicat apparatus, Instron universal testing machine (computerized, software based), and digital weighing machine.

Sample preparation and testing for setting time determination

Design of mold for setting time determination

To prepare sample for setting time, brass ring mold was used, with an inner diameter of 10 mm and height of 2 mm. A brass base plate was attached to the mold with the help of screws on contralateral sides.

Methodology for setting time determination

A paper pad was placed on the platform in the digital weighing machine in the ratio of 3:1. First additive liquid was dispensed on the paper pad, thereafter MTA Plus powder was dispensed on the paper with the help of plastic scoop in the same ratio by weight as the dispensed additive, that is, if 1.2 g of powder is taken, then 0.4 g of additive is dispensed. The paper pad was then withdrawn from the platform and was placed on the glass slab. The liquid and powder were then thoroughly mixed to ensure all the powder particles that are incorporated in the liquid to achieve a homogeneous mixture. The mixed material is then transferred into the mold and tapped to discard any air bubble formed in the mix. A total of five samples each (five holes in the mold) were prepared for each additive. The samples were tested by placing the assemblage of mixed material in a ring mold and then placed in a cell culture incubator with 95% humidity at 37°C. Setting time was calculated with the help of a Vicat apparatus. The Vicat apparatus was assembled with an indenter needle which was placed on the sample with a load of 300 g. The Vicat indenter needle had a flat end with a diameter of 1.0 ± 0.002 mm. Indentation marks were closely observed on the cement surface. The setting time was noted down when the indenter needle failed to indent in the three separate areas of the sample within the ring mold. Analysis of the samples was done at subsequent time intervals of 5 min, 10 min, 15 min, 20 min, 25 min, 30 min, 40 min, 50 min, 60 min, 1.5 h, 2 h, 2.5 h, 3 h, 3.5 h, 4 h, and so on.

Sample preparation and testing for compressive strength determination

Design of mold for compressive strength determination

For the measurement of CS, cylindrical brass split mold with an internal diameter of Four millimeters and height of Six millimeters was used. Two screws were incorporated in the design of the mold to approximate the two ends of the mold.

Methodology for compressive strength determination

The samples were dispensed and mixed as previously described in the methodology for determination of setting time. A total of 10 samples each (five holes in one mold and two of such molds were taken) were prepared with each additive. The specimens were placed in the incubator with 95% humidity at 37°C to set for seven days. The set samples were then removed from the split mold.

Testing method for compressive strength samples

Instron machine was used for the measurement of CS. Load was applied along the long axis of the specimen, while the specimen was placed with its flat ends between the plates of the apparatus. Using a crosshead speed of one mm/ min, the maximum load required to fracture each specimen was calculated. The CS was eventually determined from the maximum load and the diameter of the specimens.

RESULTS

Data were entered into Microsoft Excel spreadsheet and were analyzed using the Statistical Package for the Social Sciences (SPSS) version 21 (IBM SPSS Statistics for Windows, version 21.0, Armonk, NY:IBM Corp.). The analysis was done using the one way ANOVA and Tukey's multiple *post hoc* test. A comparison of the nine groups with compressive strength was done using one way ANOVA. As $P < 0.05$, the comparison between the groups was statistically significant. Table 1 shows the setting time of MTA Plus with the different additives.

Table 1: Setting time of mineral trioxide aggregate Plus mixed with various additives

Additives	Setting time
5% CaCl ₂	1 h 20 min
10% CaCl ₂	45 min
Phosphate-buffered saline solution	1 h 45 min
80%DW/20%PG	2 h 40 min
Saline	2 h 15 min
Lignocaine HC	2 h 20 min
15% sodium phosphate	25 min
3% sodium hypochlorite gel	50 min
Proprietary gel with MTA Plus	40 min

MTA: Mineral trioxide aggregate; CaCl₂: Calcium chloride; PG: Propylene glycol; HC: Hydrochloride

Results were compared with distilled water. Setting time with distilled water (control group) was known to be approximately 45 min \pm 20 min when mixed in the ratio of 3:1 by weight. As shown in Graph 1, while 5% CaCl₂ was found to slightly increase the setting time, 10% CaCl₂ reported to have an approximately similar setting time. Additives such as lignocaine HCl, propylene glycol, and saline retarded the setting time significantly. 3% sodium hypochlorite gel remained nearly unaffected. 15% sodium phosphate significantly decreased the setting time to 20 min. MTA Plus gel decreased the setting time by 5 min.

Handling properties with different additives

Additives such as 3% sodium hypochlorite gel, MTA Plus gel, and 15% sodium phosphate significantly improved the handling properties of MTA Plus powder, while the gel form of additives gave a thick putty-like mix and 15% sodium phosphate and propylene glycol gave a lesser grainy and smoother mix.

Compressive strength with different additives

Testing of the MTA samples for CS was carried out in Praj Metallurgical Laboratory, Pune, Maharashtra, India. Ten samples were tested in each additive group. The value of CS for each group is given in Table 2.

The highest value of CS was given by the MTA Plus group (Group I), followed by 10% CaCl₂ (Group B) and 15% sodium phosphate (Group G), while the least value was given by 80% DW/20% PG (Group D), as shown in Graph 2.

DISCUSSION

MTA has become one of the most popular endodontic materials over the past quarter of a century because of its wide applications in dentistry. Calcium, selenium, and aluminum form trioxide aggregate in MTA. First introduced as a potential root-end filling material, it is proven to be effective in procedures such as apexification, pulpotomy, direct and indirect pulp capping, apexification, apexogenesis, apical barrier formation in teeth with open apices, repair of root perforations, external root resorption repair, obturation of retained primary teeth, and prophylactic treatment of dens evaginatus.^[1] High biocompatibility and radiopacity, excellent sealing ability, low diffusion, and high hydrophilicity make it an "ideal" root-end filling material.^[2] On contact with moisture, calcium oxide in MTA converts into calcium hydroxide raising the pH and thus provides the added antibacterial effects. It delivers high resistance to marginal leakage and bacterial penetration by forming a steady barrier due to its expansion and contraction properties which are quite similar to dentin. MTA Plus is a new variant

Table 2: Compressive strength of various additives with mineral trioxide aggregate Plus

Specimen number	CS (5%CaCl ₂)	CS (10%CaCl ₂)	CS (phosphate-buffered saline solution)	CS (80%DW/20%PG)	CS (saline)	CS (lidocaine HCl)	CS (15%Na ₂ HPO ₄)	CS (3% sodium hypochlorite gel)	CS (proprietary gel with MTA Plus)
1	0.19	1.24	0.10	0.10	0.35	1.10	1.00	0.24	1.28
2	0.21	1.19	0.11	0.11	0.29	1.22	0.97	0.15	1.99
3	0.20	1.27	0.13	0.11	0.38	0.20	0.8	0.24	1.63
4	0.42	1.51	0.45	0.10	0.28	0.36	0.23	0.21	3.19
5	0.97	0.72	0.31	0.13	0.34	0.29	1.21	0.30	1.85
6	1.19	1.81	0.34	0.21	0.39	1.26	0.88	0.24	2.87
7	1.22	1.48	0.38	0.18	0.34	1.19	1.36	0.24	2.45
8	0.79	1.61	0.15	0.13	0.19	0.77	1.16	0.32	1.42
9	0.52	0.54	0.21	0.16	0.22	0.85	0.72	0.27	1.89
10	0.28	0.99	0.17	0.12	0.35	0.93	0.57	0.27	2.04

CS: Compressive strength; MTA: Mineral trioxide aggregate; CaCl₂: Calcium chloride; PG: Propylene glycol

of MTA, marketed by Prevest Denpro, Jammu, India (for Avalon Biomed Inc., Bradenton, FL, USA) company. MTA Plus showed improved reactivity and prolonged capability to release calcium and increase the local pH to alkaline values in comparison with ProRoot MTA (a commercial form of MTA).

According to a study by Funteas *et al.*, a comparative analysis of 15 different elements by inductively coupled plasma emission spectrometry revealed that there was no notable difference between the composition of MTA and Portland cement.^[11] Contrary to this study, Dammaschke *et al.* compared the chemical composition of white ProRoot MTA with two common Portland cement and it was found that ProRoot MTA contains fewer toxic heavy metals which reduce inflammation and other allergic reactions and lead to reduced cytotoxicity and more acceptances by the patients. Furthermore, the presence of a few aluminum particles in ProRoot leads to the formation of few reactive by-products as tricalcium aluminate and hence prolonged setting time.^[12]

These pronounced ion-releasing properties are interlinked with its noticeable porosity, water sorption, and solubility and with the formation of calcium-phosphorus minerals. The finer calcium silicate powder may explain the higher ion release, water sorption, porosity, and solubility of MTA Plus compared with conventional MTA. For clinicians, MTA Plus represents a lower-cost bioactive tricalcium silicate material with interesting chemical-physical properties that could be a convenient alternative to the conventional calcium silicate MTA-like cement.^[13] It is marketed with an anti-washout gel^[9] along with water for dispensing the material. MTA is known for its poor handling property and longer setting time and has a consistency which makes its use limited in some clinical situations. It is also said to lose its cohesiveness post drying, making its overall use as very technique sensitive

and unpredictable. Furthermore, common additives and solutions used in a clinical scenario such as saline, lignocaine, phosphate-buffered saline, sodium phosphate monobasic, propylene glycol, and sodium hypochlorite have been tried and tested in the past studies with former MTAs in an attempt to improve its handling as well other physical and mechanical properties.

Frough Reyhani *et al.*^[14] evaluated the effect on setting time on mixing chlorhexidine or sodium hypochlorite with calcium-enriched mixture cement. It was observed that while chlorhexidine did not adversely affect the setting time, sodium hypochlorite significantly increased the setting time of the cement. This result is opposite to the effect of chlorhexidine on MTA which does not allow MTA to set even after several days. This was in contradiction to the study by de Andrade *et al.*^[15] and Kogan *et al.*^[16] which proved that MTA mixed with chlorhexidine did not set even for days and drastically increased the setting time. This forms the basis for exclusion of this additive in our study.

The composition of MTA Plus was studied and compared with the former commercial MTAs. Camilleri *et al.* proved that MTA Plus had a higher specific surface area of 1.5366 m²/g compared to ProRoot MTA with 0.9822 m²/g while having a similar chemical composition. It was discovered that MTA Plus constituted portlandite, dicalcium silicate, and tricalcium silicate as major constituents and bismuth oxide as a radiopacifier. The setting time of MTA Plus was effected by the environmental conditions. Wet curing retarded the setting time. This is in accordance with previous research investigating the setting time of Portland cement and tricalcium silicate-based cement cured in different environmental conditions.^[17]

Root-end filling material may wash off while rinsing an osteotomy site resulting in compromised root-end seal and its unintended consequences. Formosa *et al.*^[9] first studied the chemical composition of proprietary gel or anti-washout gel provided with MTA Plus and the properties of the corresponding set cement. The anti-washout gel reduced the setting time of MTA Plus by 65 min when taken in the water/powder ratio of 0.35 g/g. The density of pure water is around 1 g/ml, but as 8% of the gel is composed of dissolved solids, the effective water/powder ratio was around 0.32 with the gel, which explains its lower setting time. Energy-dispersive X-ray fluorescence analysis of MTA gel identified silicon dioxide, potassium oxide, calcium oxide, chlorine, calcium oxide, and organic polymer in its composition. The anti-washout additives are said to function by increasing the viscosity of the water, which increases the resistance to segregation of the cement paste under washout environmental conditions by forming a branched polymer network when it comes in contact with water.

Strong calcium hydroxide peaks were observed with MTA Plus and water mixture, whereas weak peaks were observed with anti-washout gel. This indicates that the gel influenced the reaction of the cement with environmental fluid rather than the water introduced during mixing. The setting time of MTA Plus with the anti-washout gel was significantly low. The high viscosity of MTA Plus gel set samples contributes to lesser porosities compared to MTA Plus water sample, and also, the CS of set gel samples was significantly higher than water set samples.

MTA Plus formed a thick calcium phosphate layer (good bioactivity) after soaking in simulated body fluid forming large calcium phosphate-based spherules. This material has shown dentin demineralization in the presence of SBF. The high calcium and OH ion releases are connected with the formation of calcium phosphate deposits. MTA Plus had a higher ion release than ProRoot MTA and Dycal; the use of the MTA Plus gel enhanced the initial calcium release and raised the pH. The finer calcium silicate powder may explain the higher ion release, water sorption, porosity, and solubility of MTA Plus compared with ProRoot MTA. Both MTA products were more water soluble and water sorptive than Dycal and more bioactive.^[12]

Zapf *et al.*^[18] studied the repercussion of additives such as 5% calcium chloride, phosphate-buffered saline, 3% sodium hypochlorite, or lidocaine on setting time and setting reaction using differential scanning calorimetry.

While this test did not give the absolute values of setting time, relative prediction between the groups was done based

on calcium hydroxide formation. Calcium chloride gave the maximum reaction rate, followed by phosphate-buffered saline, and the least or detrimental reaction rate was observed with lidocaine. These results are quite synergistic with our study.

Lee *et al.*^[19] compared the setting time and CS of ProRoot MTA mixed with hydration accelerators such as 10% CaCl₂, 0.1% citric acid, and calcium lactate gluconate keeping distilled water as a control group. The setting time was reported to be 108 ± 1.6 min for distilled water, 74 ± 0.6 min for 10% calcium chloride, 72.7 ± 1.4 min for 0.1% citric acid, and 13.9 ± 0.3 min for calcium lactate gluconate solution, while the corresponding values of CS were 39.08 MPa, 35.30 MPa, 27.23 MPa, and 8.94 MPa after 1 week when MTA was mixed in the cement/water ratio of 0.8 g/0.3. It was thus concluded from the following study that although calcium lactate gluconate decreased the setting time of MTA remarkably, it also caused a tremendous decrease in the CS values.

According to Machado *et al.*, 10% CaCl₂ decreased the setting time of Portland cement showing in accordance with our study. According to Al Anezi *et al.*, NaOCl significantly decreased the setting time, which was not in accordance with our study where NaOCl did not affect the setting time. Ber *et al.*^[20] used the combination of 1% methylcellulose and 2% CaCl₂ resulting in a mixture of chemically modified MTA, which did not significantly affect the CS but markedly decreased the setting time. The setting time of 1% MC/CaCl₂ with MTA was 57 ± 3 min, and 2% MC/CaCl₂ with MTA was 105 ± 5 min, while it was 202 ± 3 min of MTA without additional additives. Monteiro *et al.* compared the setting time of 2% chlorhexidine alone or in combination with 5% CaCl₂. In the given study, the 5% CaCl₂ + 2% CHX group had the shortest final setting time (23 min for gray MTA), which was also significantly shorter than that of MTA with water (63 min for gray MTA).

The formation of chlorhexidine chloride, which increases the ionizing capacity of the chlorhexidine molecule, could be the likely cause of faster reaction with this combination which is not seen when chlorhexidine is mixed alone.

Huang *et al.* claimed that the setting time of WMTA reduced to 26 min compared to 151 min (~3 h) with additional 15% Na₂HPO₄ with MTA powder. It also increased the diametric tensile strength of the set cement. Strong ionic interactions between phosphate and calcium and/or silicate cations, thereby enhancing the formation of calcium silicate hydrates or other phases, could be the possible mechanism of this accelerated reaction.^[21]

Singh *et al.*^[22] compared the push-out bond strength of Biodentine with MTA when mixed with sodium hypochlorite or chlorhexidine gluconate. The push-out bond strength of Biodentine was proven to be higher than MTA.

While there was no effect of these additives on the push-out bond strength of Biodentine, chlorhexidine gluconate adversely affected the property when mixed with MTA. Thus, it was proven that chlorhexidine gluconate has other deteriorating properties other than increasing the setting time of MTA.

In the present study, nine groups were studied, where MTA was mixed with the following additives: Group A – 5% CaCl₂ solution, Group B – 10% CaCl₂ solution, Group C – phosphate-buffered saline solution, Group D – 80% distilled water/20% propylene glycol, Group E – saline, Group F – lidocaine HCl, Group G – 15% sodium phosphate, Group H – 3% sodium hypochlorite gel, and Group I – proprietary gel with MTA Plus. While 15% sodium phosphate gave the lowest value of setting time that is 25 min, 20% propylene glycol/80% distilled water retarded the setting time to almost 2 h 40 min. MTA Plus gel showed an insignificant decrease in setting time by 5 min compared to the lowest setting time of 25 min as shown by 15% sodium phosphate among the study groups. MTA Plus gel showed the highest average CS value of 2.061 MPa, compared to CS value of 0.89 MPa as shown by 15% sodium phosphate, which makes MTA Plus gel a potential additive. 20% propylene glycol/80% distilled water decreased the average CS value to 0.135 MPa. Proprietary gel with MTA Plus gave the highest value of CS, followed by 10% CaCl₂, and 15% sodium phosphate correlates with the previous studies as these are proven to be potential additives for MTA decreasing the setting time without much compromising on the CS. On the other hand, additives such as saline, phosphate-buffered saline, and 20% propylene glycol/distilled water led to decrease CS and are also not proven to be better accelerants though they improved the handling characteristics of MTA Plus.

According to an article by Prasad *et al.*, 10% CaCl₂ significantly reduced the setting time of white MTA and also maintained the pH at a high value. However, there was no improvement in the CS of the material which is similar to the results obtained in this study.^[23]

A recent investigation of the anti-washout characteristics of MTA Plus mixed with anti-washout gel indicated that the gel drastically increased the washout resistance of the MTA. In underwater concrete, anti-washout additives function by increasing the viscosity of the water mixed with

the cement powder. This increased viscosity increases the resistance to the segregation of the cement paste under the washing action of external water solutions. When in contact with water, the anti-washout admixtures produce a branched polymer network which controls the movement of water and reduces the tendency for dilution with external water during and after placing of the cement in the surgical site. Increased paste viscosity may also be the result of increased adhesion between the grains. This is consistent with the mechanisms of action for anti-washout additives proposed by other researchers. An increase in CS of MTA-anti-washout gel was observed compared to the MTA-water group.^[24]

Various studies show a great deal of discrepancy among values of setting time with the same additive. While Camilleri *et al.*^[17] showed the setting time of ProRoot MTA to be 3 h 22 min with Vicat needle, Lee *et al.*^[19] proved the value to be 2 h 45 min with the use of Gillmore needle. Many such studies contradict their values for the same set cement. This difference is probably attributed to different machinery used in each study, also the time for which needle rest on the cement is different. No standardization of procedure is available for accuracy in calculating the setting time; it is just an objective approximation when needle fails to indent given cement.

CONCLUSION

Within the limitations of our study, it was found that 15% sodium phosphate significantly decreased the setting time to 20 min, though 15% sodium phosphate reduced the CS when compared to the anti-washout proprietary gel provided with MTA Plus. This could be considered to be used in situations where a slight reduction of CS would not affect the treatment outcome. Additives such as phosphate-buffered saline and 20% propylene glycol/distilled water led to decrease CS and also acted as retarders though they have proven to improve the handling characteristics of MTA Plus. Further studies in this direction and biocompatibility studies yet need to be done.

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Conflicts of interest

There are no conflicts of interest.

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