

# A quantitative method for determining the antiwashout characteristics of cement-based dental materials including mineral trioxide aggregate

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## Abstract

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**Aim** To introduce and assess a novel method for measuring washout resistance of cement-based dental materials, including mineral trioxide aggregate (MTA), to qualitatively verify the results with a clinical simulation and to evaluate the washout resistance of a new root-end filling material.

**Methodology** A method for assessment of washout resistance of root-end filling materials was developed by adapting the CRD-C 661-06 (a method for evaluating the resistance of freshly mixed concrete to washout in water), to permit testing of dental cements. White Portland cement (PC), MTA-Plus mixed with either water or a polymer-based antiwashout gel (MTA-AW), MTA-Angelus, IRM and amalgam were tested with either distilled water or HBSS as washout

media. Additionally, the washout resistance was tested qualitatively by spraying the test materials at the terminus of simulated canals with a metered jet of water.

**Results** A mass loss of 2–7% for PC, 0.4–4% for MTA-Plus, –0.9% for MTA-AW, 5–10% for MTA-Angelus and 0% for IRM and amalgam was recorded with the modified CRD-C 661-06 method. No significant difference was found between using water and HBSS as washout media for the same material. The results of the modified CRD-C 661-06 method were similar to those obtained on the simulated canals.

**Conclusions** The modified CRD-C 661-06 method provided repeatable results that were comparable to the simulated clinical method. The antiwashout gel used with MTA-Plus reduced the material washout and was similar to IRM and amalgam.

**Keywords:** antiwashout, dental materials, mineral trioxide aggregate, root-end filling materials.

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## Introduction

Mineral trioxide aggregate (MTA) is a Portland-cement-based material (Camilleri *et al.* 2005) with

numerous applications in endodontics such as pulp capping, apexification, repair of root perforations, root-end filling (Torabinejad & Chivian 1999) and others (Parirokh & Torabinejad 2010). However, one of the drawbacks of MTA is washout (Bortoluzzi *et al.* 2006), which refers to the tendency of freshly prepared cement paste to 'disintegrate upon early contact with blood or other fluids' (Wang *et al.* 2007).

Following a survey of the literature, no standardized method for evaluating washout resistance of cement-based dental materials was evident. A number

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of researchers have resorted to employing diverse quantitative and qualitative methods for evaluating washout resistance. These include visual observation after immersion in water (Chen *et al.* 2010, Lin *et al.* 2010) and measuring the change in mass after injecting cement in water (Wang *et al.* 2007, Kai *et al.* 2009). Other investigators (Porter *et al.* 2010) employed water sprayed from a known distance at a specified flow rate on samples consisting of cement paste. The resultant specimens were then photographed and visually examined, and washout resistance was evaluated by allowing two independent, blinded evaluators to determine the percentage of the original margin remaining from the photographs. Further methods used for the investigation of washout resistance involved the injection of the cement into distilled water, and after immersion for 24 h, the nondecayed part of the cement was freeze-dried. Its mass, expressed as a percentage of the original mass of the cement, was used to determine the washout resistance (Kai *et al.* 2009). A similar method (Wang *et al.* 2007) involved injecting the cement into a container filled with water, shaking the container for a set number of minutes and measuring the mass of cement remaining, expressed as a percentage of the original mass of cement injected.

The materials tested were calcium silicate (Wang *et al.* 2007, Kai *et al.* 2009) and calcium phosphate (Chen *et al.* 2010, Lin *et al.* 2010) bone cements. Dental and endodontic materials (white MTA, Generex-A, Capasio and Ceramicrete-D) have also been investigated (Porter *et al.* 2010). A visual method was used to assess the loss of material caused by washout of the cement in solution.

The diverse number of washout testing methods available reveals the necessity of a standardized method for measuring the washout resistance of dental materials. The ideal method would be one that gives quantitative, objective and reproducible results. One possibility is to adapt the method described in the CRD-C 661-06 specification, section 16, which provides a test method for determining the resistance of freshly mixed concrete to washing out in water, to make it applicable for small volumes of dental materials. This method is based on the older CRD-C 61-89A specification and is currently used for testing the washout resistance of concrete. Briefly, this method involved placing the test material into a perforated cylinder, allowing it to sink freely through a column of water and then raising it back up. The test cycle was repeated a number of times. The mass of material

lost following each cycle was measured. Washout was then expressed as a percentage of the initial mass of the sample. The original method uses concrete samples with a mass of 2 kg. This is prohibitively large amount for dental cements, and not representative of the small volumes of material typically employed in dentistry.

Although there are several formulations of materials based on tricalcium silicate cement, there are two main mineral trioxide aggregates namely ProRoot MTA (Dentsply, York, PA, USA) and MTA-Angelus (Angelus Soluções Odontológicas, Londrina, PR, Brazil). The materials have a similar chemical composition and are composed of Portland cement and bismuth oxide. A difference in the texture and in the particles of each material exists. MTA-Angelus does not contain the calcium sulphate phase, which results in a shorter setting time of the material (Oliveira *et al.* 2007). In addition, MTA-Angelus is less radiopaque (Camilleri & Gandolfi 2010). Recently, another MTA has been introduced on the market. According to the manufacturer, MTA-Plus (Avalon Biomed Inc., Bradenton, FL, USA) is similar in composition to ProRoot and MTA-Angelus but is ground finer. MTA-Plus is marketed accompanied by water or a hydrosoluble gel aimed at reducing washout.

The purpose of this study was to introduce and assess a method to quantitatively measure the washout resistance of cementitious dental materials and to verify these results qualitatively by comparing them with the results of a simulated clinical situation. In addition, the washout resistance of a novel root-end filling material is also assessed.

## Materials and methods

The materials used in this study included MTA-Plus (compounded by Prevest Denpro, Jammu, India for Avalon Biomed Inc.) lot #2011022801, Portland cement (PC; CEM 1, 52.5 N; LaFarge Cement, Birmingham, UK), MTA-Angelus (Angelus, Londrina, PR, Brazil), IRM (Dentsply, Konstanz, Germany) and amalgam (AB Ardent, Arlandastad, Sweden). The MTA-Plus was mixed with either water (MTA-Plus) or an antiwashout gel (MTA-AW; compounded by Prevest Denpro, Jammu, India for Avalon Biomed Inc. Bradenton, FL, USA) at a water-to-cement ratio of 350  $\mu\text{L g}^{-1}$  and gel-to-cement ratio of 350  $\mu\text{g g}^{-1}$ , respectively. White Portland cement was mixed at a water to cement ratio of 350  $\mu\text{L g}^{-1}$ . MTA-Angelus, IRM and amalgam were mixed as directed by the

manufacturer. The fluids used for washout testing included distilled water and Hank's balanced salt solution (HBSS; H6648, Sigma Aldrich, St. Louis, MO, USA).

### Drop method using an adaptation of the CRD-C 661-06 method

The test set-up (Fig. 1) consisted of a standard-sized test tube with an internal diameter of 14.5 mm, which was filled to a height of 120 mm with distilled water or HBSS at room temperature (23 °C). A cylindrical container with a 9.0 mm diameter and a height of 17 mm was constructed from two pieces of woven brass mesh (60 wires per inch) with a wire diameter 0.18 mm. The seams on the side and around the bottom had an overlap of 1.0 mm and were bonded with light-curing resin (Heliobond, Ivoclar Vivadent AG, Schaan, Liechtenstein). The empty mesh cylinder was weighed on an analytic balance with an accuracy of  $\pm 0.0001$  g (Sartorius AG, Gottingen, Germany), and a quantity of material to be tested was prepared and transferred to the cylinder. In the case of PC and MTA-based materials, approximately 1.35 g of cement paste was prepared, using 1.00 g of each cement powder, by mixing with the appropriate quantity of distilled water or antiwashout gel on a glass slab with a spatula. In the case of IRM, two scoops of powder and three drops of fluid were mixed and transferred to the basket. Amalgam was prepared by triturating one 600-mg capsule for 10 s and transferring the material immediately to the mesh cylinder. The material under study was packed

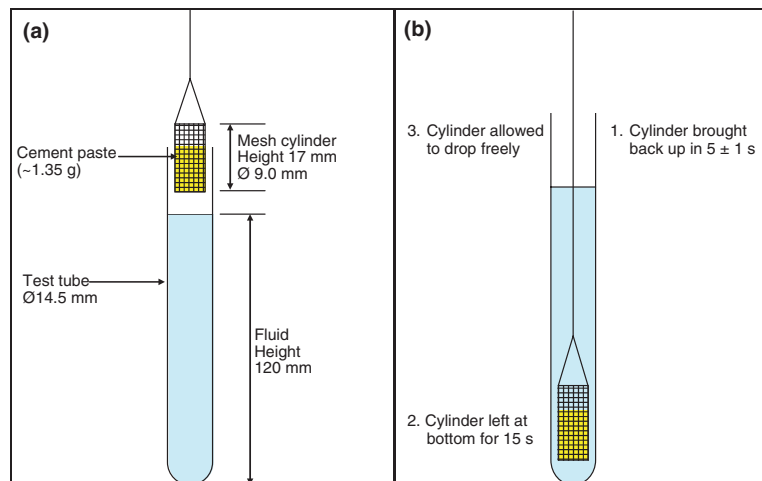
into the cylinder using a dental plugger, and the top surface of the cement was flattened (Fig. 2a). The outer part of the cylinder was lightly patted with absorbent paper to remove any extruded material. The mass of the cylinder, filled with material, was then measured, and the exact mass of cement in the cylinder was calculated. The cylinder was released just above the surface of the fluid in the test tube (Fig. 2b) and allowed to sink unhindered as specified in the standard (CRD-C 661-06). The cylinder was left at the bottom of the tube for 15 s and then brought out of the water in  $5 \pm 1$  s and allowed to drip for 2 min. The cylinder was patted dry with absorbent paper to remove any remaining water and weighed. The complete procedure (Fig. 1b,c) was repeated in the same fluid (as specified in the standard) to give a total of three drop cycles per specimen.

The materials were tested in distilled water and HBSS. Two replicate tests per material per fluid were conducted using fresh solution for each replicate. Washout (or loss of mass of the sample) was expressed as a percentage of the initial mass of the sample and calculated using Equation 1:

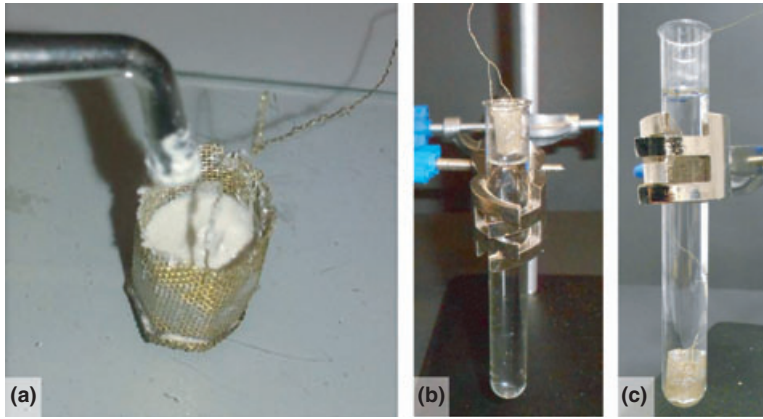
$$D = 100 \times \frac{M_i - M_f}{M_i} \quad (1)$$

where:  $D$  = washout (%);  $M_i$  = Mass of sample before initial drop;  $M_f$  = Mass of sample after each drop

Analysis of variance (ANOVA) with  $P = 0.05$  and Tukey's post hoc test were used to perform multiple comparison tests.



**Figure 1** (a) Test set-up to determine washout resistance and (b) Procedure for one drop cycle.



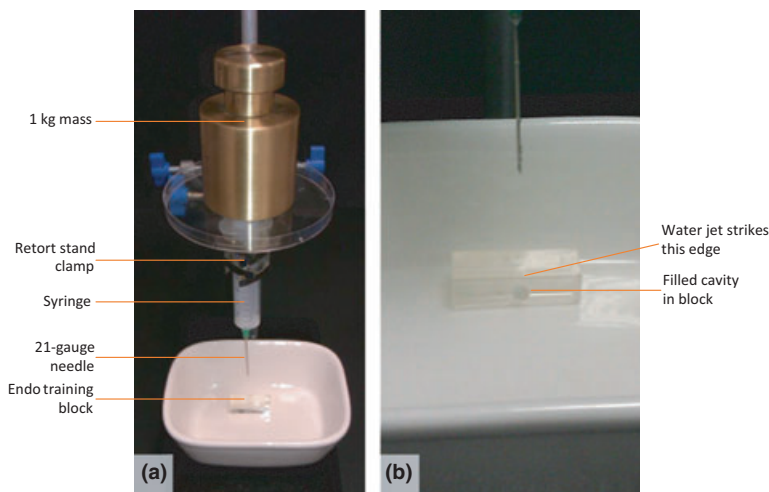
**Figure 2** (a) Preparation of cement specimen for washout testing; (b) cylinder filled with cement paste held over solution in tube; (c) cylinder allowed to free-fall in solution.

### Metered spray testing using simulated canals

The set-up for the simulated clinical situation is shown in Fig. 3a. A syringe with a 21-gauge needle was mounted in a retort stand and filled with 10 cc of distilled water. An Endovue block (Dentsply, Konstanz, Germany) with a pre-prepared root-end cavity was used. The canal was prepared to 0.5 mm short of the canal terminus using the crown-down technique with ProTaper rotary nickel–titanium instruments (Dentsply Maillefer, Ballaigues, Switzerland) under copious irrigation. The canals were dried and filled with gutta-percha and AH Plus sealer (Dentsply Maillefer, Montigny de Bretonneux, France) using the warm vertical condensation technique with System B (Sybron Endo, Orange, CA, USA).

The Endovue blocks were then weighed, filled with the material to be tested, re-weighed and placed in a

ceramic dish beneath the needle, such that the jet of water from the needle impinged on the edge of the block and flowed down the side, thus washing over the material under test but not directly spraying into it (Fig. 3b). In this way, the material is washed out by the same mechanism responsible for washout *in vivo* when the root end is irrigated with a stream of water perpendicular to the root-end cavity. The distance from the tip of the needle to the edge of the block was set to  $35 \pm 1$  mm. A 1-kg weight was placed on the syringe plunger to provide a constant force. This combination resulted in the syringe emptying entirely within  $15 \pm 1$  s, giving a mean flow rate of  $0.667 \text{ cc s}^{-1}$ . Following the test, the block was dried with filter paper, taking care not to disturb the material in the cavity, and weighed. The loss in mass of the block was then expressed as a percentage of the mass of dental material initially placed in it.



**Figure 3** (a) Experimental set-up for verification of washout results; (b) Detailed view of endo block showing orientation of filled cavity and area struck by water jet.

**Results**

**Drop method using an adaptation of the CRD-C 661-06 method**

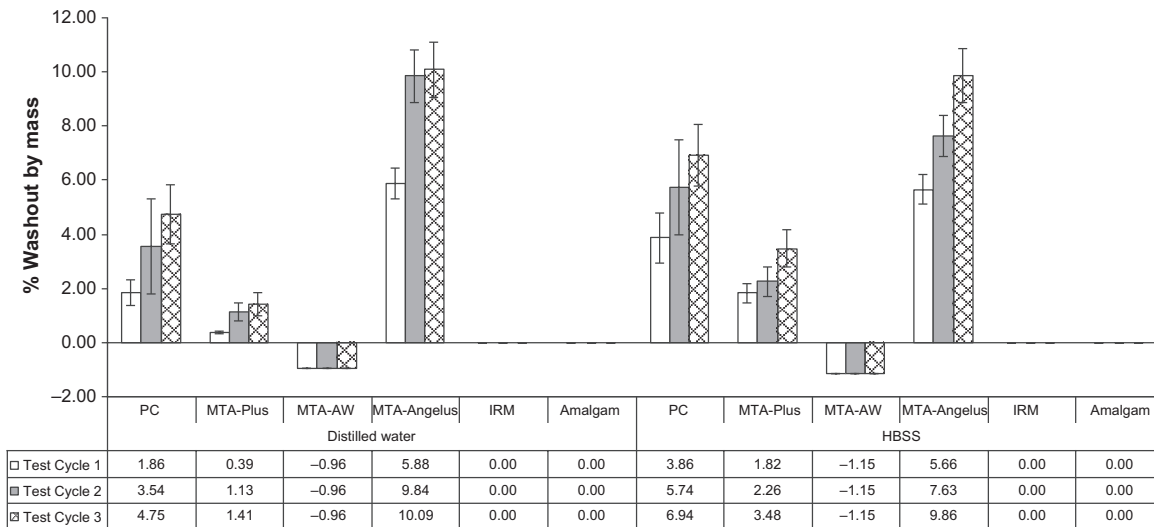
The results of the drop tests are presented in Fig. 4. Three of the materials tested exhibited washout. In order of increasing washout resistance, these included MTA-Angelus, Portland cement and MTA-Plus. MTA-AW displayed mass gain after the first washout test cycle, but then its mass remained unaltered after subsequent test cycles. IRM and amalgam did not change mass after any of the three drops. The standard deviation was relatively small compared with the mean values, indicating an acceptable confidence level and repeatability between samples. Thus, the number of repeats conducted was deemed satisfactory.

No statistically significant differences were observed between the washout percentage in distilled water and HBSS for the same material at the same test cycle number ( $P > 0.05$ ) in all cases. This indicates that washout was not affected by the different constitution of the two media tested. No statistically significant differences were observed between IRM and amalgam for all tests. For the first test cycle in water, no statistically significant differences were observed amongst PC, MTA-Plus and MTA-AW, whilst statistically significant differences were observed between MTA-Angelus and MTA-Plus ( $P = 0.001$ ), amalgam and MTA-Angelus ( $P < 0.001$ ) and similarly between IRM

and MTA-Angelus ( $P > 0.001$ ). For the first cycle in HBSS, more material was washed out from PC than from MTA-AW ( $P < 0.001$ ), and more was washed out from MTA-Angelus than MTA-Plus ( $P = 0.003$ ). For the second test cycle, more material was washed out from PC than from MTA-AW in water, whilst in HBSS the loss in weight was observed with the numerical order being MTA-Angelus > PC > MTA-Plus > amalgam/IRM > MTA-AW. There was a statistically significant difference between the PC and MTA groups ( $P = 0.022$ ,  $P < 0.001$  respectively), between MTA-Plus and MTA-Angelus ( $P < 0.01$  in all cases) and between the MTA-Plus groups with and without antiwashout gel ( $P = 0.027$ ). In the third test cycle, the pattern of weight loss was similar to that of the second test cycle.

**Metered spray testing using simulated canals**

The results of the simulated canal tests are presented in Fig. 5. All the water-based materials (PC, MTA-Plus and MTA-Angelus) were washed out of the cavity in their entirety. In the case of the other materials (MTA-AW, IRM and amalgam), the mass did not change following spraying with water and drying, indicating that none of the material was washed out. These results indicate that the antiwashout gel successfully inhibited washout even in a stream of water. The results from this test concur with the trend seen in the plunge tests, where a positive washout percentage was recorded for PC and the two MTA



**Figure 4** Washout percentage after each test cycle for the materials and washout media tested.

preparations, and zero or a negative washout percentage was recorded for MTA-AW, IRM and amalgam.

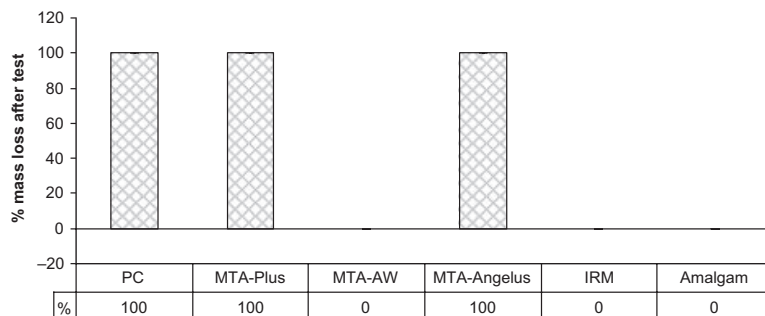
**Discussion**

Washout poses a clinical problem during root-end surgery. Prior to closing an apical flap, it is necessary to irrigate the area well to avoid postoperative complications. In addition, the flow of blood in the surgical site once the suction keeping the area dry is ceased will also cause washout to a certain degree. Washout resistance of root-end filling materials is thus important. Enhanced washout resistance avoids loss of the material placed at the root end.

The Specification CRD-C 661-06 (2006) suggests the test method for testing washout resistance of concrete in the construction industry. Portland cement, which is the main constituent compound in MTA (Torabinejad & White 1995), is also used as a binder in concrete. The washout test determines the relative amount of cement paste lost when the concrete is exposed to a large volume of water. Washout resistance in concrete can be determined by the stream, drop, pH factor, plunge and the spray tests (Sonebi et al. 1999). The stream and drop tests are based on visual inspection of loss of material when exposed to a liquid normally water and thus are operator dependent. The pH factor test measures the rise in pH of the storage liquid when fresh concrete is dropped. This method is relevant for Portland cement type materials, which leach out calcium hydroxide thus causing a rise in pH of the surrounding media, but may have limited use when testing other materials. The plunge test is specified by CRD-C 661-06, and both this test and the spray test measure the change in mass of concrete when subjected to water. The conditions set by these tests are standard and reproducible. In addition, the measurements undertaken are not subjective.

In this study, the plunge test was selected to measure the washout resistance of a variety of dental materials, including newly introduced variety of mineral trioxide aggregate (MTA-Plus) mixed with water or an antiwashout gel, MTA-Angelus, intermediate restorative material (IRM) and amalgam. All these materials are used as root-end filling materials. Portland cement was tested as a control material. The plunge test developed was based on the CRD-C 661-06 specifications using smaller dimensions of the basket and water container to be able to accommodate the testing of a dental material. The results obtained indicate that the method exhibits good levels of repeatability and precision. As the result is a value, and does not rely on personal judgment as visual inspection methods do, it is quantitative and objective. Because the method involves multiple drops of the same sample, it gives an insight into the behaviour of the material.

The materials were tested both in distilled water and HBSS as previous studies have shown a link between physical properties and curing conditions of MTA-like systems (Formosa et al. 2012). The Portland cement lost a relatively large percentage of mass after the first test cycle and continued losing mass in subsequent cycles. The MTA-Plus lost a smaller amount of mass in the first test cycle, and the rate of mass loss started to taper off by the third cycle. This tapering-off effect was also observed for MTA-Angelus in distilled water, but not in HBSS. The increased resistance of MTA-Plus compared with PC may be due to its finer particle size. This would give MTA-Plus particles a greater surface area and thus greater cohesive force of attraction between adjacent particles; hence, the slightly greater resistance to washing out as this is the only force keeping the freshly mixed paste intact in the minutes immediately after mixing, as the C-S-H network has not had time to develop any strength. With regards to washout, MTA-Angelus



**Figure 5** Mass loss after testing with Endovue blocks, expressed as a percentage of the initial mass of material placed in the block.

fared worst amongst the materials tested. The MTA-Plus mixed with antiwashout gel exhibited a gain in mass after the first cycle. The enhanced antiwashout resistance may be due to the water-soluble polymer present in the gel as claimed by the manufacturer, having the capacity to absorb water to some degree when the surface of the sample was placed in contact with the water. The washout resistance of the samples incorporating antiwashout gel was confirmed visually as the liquid in the test tube remained clear after each test. In contrast, the fluid in the test tube was visibly turbid after testing with PC, MTA-Plus and MTA-Angelus, both with water and HBSS. IRM and amalgam did not change mass in any of the tests. This was a predictable result as neither material is water based, and in fact, the eugenol in the liquid component of IRM is hydrophobic, as are the metals in the amalgam.

The results of the drop tests were confirmed with the simulated canal tests. Preliminary tests using root filled teeth were performed, but the results obtained were not reliable because of the tooth absorbing a portion of the water and thus distorting mass measurements. For this reason, simulated canals in resin blocks were substituted for the teeth as they are made of a material that does not absorb water and thus provided a means of eliminating water uptake as a source of error. The simulated canal results were consistent with the plunge test results. In particular, MTA-AW resisted washing out in both tests, but did not gain any appreciable mass. This may be due to the much smaller surface area exposed, compared with the drop test, and the very small mass of material that could be placed into the root-end cavity (around 0.04 g in the tests conducted). Comparing the two methods, more material loss was observed with the simulated canals than with the plunge test. The reason for this may be that material in the simulated canal had its surface directly exposed to the water, whilst in the plunge test, the mesh cylinder partially isolated the cement from the water. In the simulated canal, the water jet was able to mechanically dislodge large quantities of cement, whilst in the plunge test a mass loss could only be achieved as a result of cement particles migrating into the washout medium and being washed out through the spaces in the mesh. In addition, the 5 mm diameter of the exposed cement placed in the cavity prepared in the blocks was significantly larger than the space between wires in the mesh (approximately 0.4 mm).

This method appears to address the main shortcomings of the other methods used in literature. Visual observation of degree of washout (Chen *et al.* 2010, Lin *et al.* 2010, Porter *et al.* 2010) is subjective and does not give a quantitative measure of washout resistance. Measurement of mass change after injecting cement in water (Wang *et al.* 2007, Kai *et al.* 2009) requires that the nondecayed part be taken out of the fluid and weighed. This introduces a potential source of variation as there may be some subjectivity in picking what constitutes the 'nondecayed' part and it is difficult to pick out the nondecayed part without losing further mass in the process. In contrast, in the plunge method, the mesh cylinder conveniently isolates the nondecayed part of the cement and makes mass measurement unambiguous. Finally, injection of the cement into distilled water, immersing for 24 h, freeze-drying and weighing (Kai *et al.* 2009) is more time-consuming and requires additional specialized equipment (and its associated costs) compared with the plunge method.

The main shortcoming of the plunge method is that the immersion speed was only controlled by the fluid resistance encountered by the falling cylinder. The immersion speed is dependent on the mass and geometry of the mesh cylinder. The latter is negated by using the same cylinder dimensions for each test. In the case of the fluid resistance, a greater mass of cement would result in a higher terminal velocity of the cylinder through the water. However, this is offset by the greater volume of cement present, which thus exposes more area to the washout in the test tube and results in a greater mass of cement being washed out. In practice, this gave comparable washout percentages between replicates of the same material, for the range of sample masses tested. However, testing of materials with large differences in density is not catered for and might result in the denser cements having a higher apparent washout percentage because of their higher falling speed.

## Conclusions

The method presented has been verified as a quantitative, objective way in which the washout resistance of cementitious dental materials may be investigated and compared. The standard deviation in percentage washout between replicate runs with the same material was found to be relatively small, on the order of 10% of the average value, and thus the method exhibits acceptable repeatability. The results

were found comparable to the simulated clinical method. Portland cement, MTA-Plus mixed with water and MTA-Angelus exhibited significant washout, whilst the antiwashout gel used with MTA-Plus reduced the material washout and made its washout resistance similar to IRM and amalgam.

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