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Letter to the Editor

Water content of ampoule packaged with ProRoot MTA

Dear Editor

We are writing to draw attention to an inconsistency in the amount of water in the packages of both white and grey MTA which are commercially available as ProRoot MTA® Original (Dentsply International Inc., Johnson City, TN, USA) and ProRoot MTA® (Dentsply International Inc.).

In each package, there are five sachets (pouches) containing 1 g of ProRoot MTA powder and six ampoules of water. The manufacturer claims that the amount of water inside each ampoule is equal to 0.35 g. This information is written on the sides of each package. However, unfortunately, we have determined that the amounts of water in the ampoules are inconsistent.

Our first reaction to this finding was to recheck the accuracy of our measurements. The accuracy of the laboratory digital scale was confirmed by using predetermined standard weights. Afterwards, in an attempt to evaluate the amount of water in each ampoule; 58 ampoules from various packages of ProRoot MTA® original (grey) and ProRoot MTA® (white) were chosen randomly. The samples were kept at a controlled laboratory temperature (20 °C) for 24 h before measurement. The weight of each ampoule with the water inside was measured using a Precisa 80A-200M device (Zürich, Switzerland), an analytical laboratory balance that measures weight to a high degree of accuracy. The analytical balance was stabilized at zero then the weight of each ampoule with the water inside was measured and recorded to an accuracy of three decimal places at 20 °C. Then the tip of each ampoule was removed using a surgical scalpel and water was released carefully into a glass container, which had also been kept at 20 °C for 24 h. Then, the weight of water was measured (MW) to an accuracy of three decimal places at 20 °C.

Subsequently the weight of each empty plastic container and the removed tip section were also determined. To calculate the definitive amount of water inside the ampoules, the weight of each empty plastic container and the cut tip were subtracted from the weight of ampoules before opening. The value obtained was considered as the definitive amount of water inside each ampoule (DW). The mean difference of the definitive water (DW) and measured water (MW) was 0.011 g (range: '0.30 g, 0.000 g' with a SD: 0.007). This is the amount of water that could not be released from the plastic ampoule. However, in the clinical situation this amount of water would probably remain in the ampoules. Both the definitive and measured values were compared with the amount of water that the manufacturer claims is inside the ampoules.

The results are demonstrated in Figs 1 and 2. The average measured (MW) and definitive weight (DW) of water in the ampoules were 0.211 g (range: '0.140 g, 0.270 g'; SD: 0.40) and 0.223 g (range: '0.149 g, 0.290 g'; SD: 0.40) respectively. These values are 0.139 g (range: '0.210 g, 0.800 g') and 0.198 g (range: '0.201 g, 0.060 g') less than the amount of water claimed to be inside the ampoules.

This lack of consistency in the amount of water inside ProRoot MTA packages is of concern and may explain the uncontrolled and undesirable characteristics of the material in certain clinical and laboratory situations. MTA consists of hydraulic powders that set and harden in the presence of water through the hydration process. According to the US patent number 5 415 547, the principle component of MTA is Portland cement (Torabinejad & Dean 1995). Complete hydration of Portland cement cannot occur if the water/cement ratio is below a certain value (Taylor 1997). The ratio of 0.33 is recommended for MTA (Fridland & Rosado 2003) and this is reflected in the nominal 0.35 g of water that is provided alongside each 1 g of MTA powder by the manufacturer.

In the manufacturer's instructions for use, which can be found inside the package, it is also clearly stated that: '*Note: 1: Adding too much, or too little liquid will reduce the ultimate strength of the material.*'

The setting of Portland cement and thus MTA takes place in two stages. After mixing with water the hydration reaction of calcium silicates begins and results in the formation of a gel consisting of calcium silicate hydrates, which liberates calcium hydroxide. The calcium hydroxide then gradually reacts with the minerals to form other hydrated compounds. The

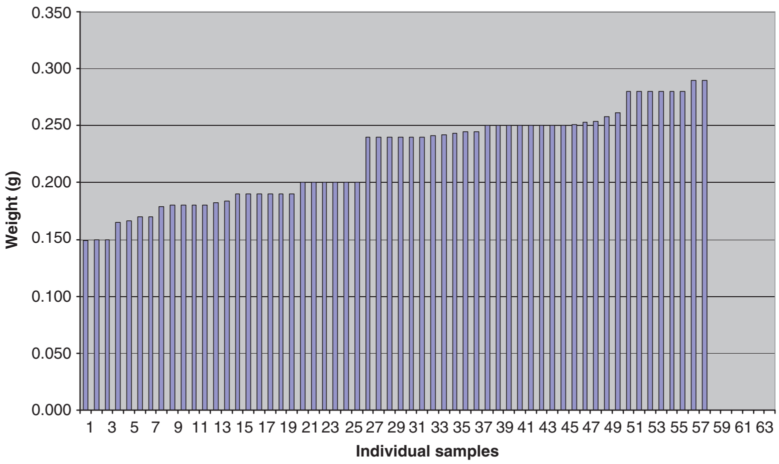

Figure 1 Definitive amount of water (g) in each ampoule (DW).

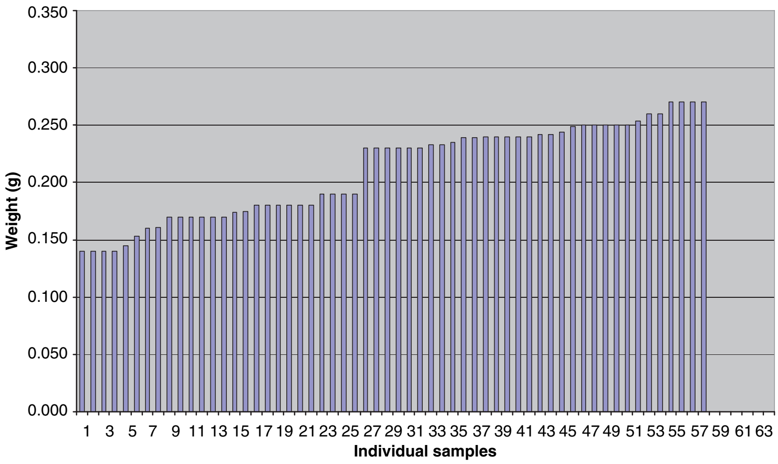

Figure 2 Measured amount of water (g) in each ampoule (MW).

calcium silicates contribute most to the binding power and strength of the material. It is also the main binding agent of crystalline calcium hydroxide that leaches most readily from the gel (Eglinton 1987). The bioactive hydration product of MTA is calcium hydroxide (Camilleri 2007) which is released during and after completion of hydration process.

The characteristic of the resultant set material is likely to be dependent on various factors including water to powder ratio, temperature, environmental humidity and pH, entrapped air and water, the rate of packing and the condensation pressure applied (Roy & Gouda 1975, Ishikawa *et al.* 1994, Torabinejad *et al.* 1995, Fridland & Rosado 2003, Lawley *et al.* 2004, Lee *et al.* 2004, Felekoglu *et al.* 2007, Nekoofar *et al.* 2007, Namazikhah *et al.* 2008).

The compressive strength of Portland cement is affected directly by the water/cement ratio (Papadakis *et al.* 2002). Walker *et al.* (2006) evaluated the effect of setting conditions (time and hydration) on MTA flexural strength and showed that a sufficient amount of water during the setting of MTA is essential to obtain optimized flexural strength of the hardened material. Therefore, mixing the inconsistent and underweight amount of water that is supplied inside ProRoot MTA packages with 1 g of MTA powder may result in an incomplete hydration process and in unwanted, uncontrolled and undesirable mechanical, chemical and biological properties of the material. It may also be one of the reasons that the material does not set or solidify occasionally after its placement at the first appointment, which is the indicator of an incomplete hydration process (Torabinejad & Chivian 1999). In addition, in most of the laboratory and clinical research studies on MTA, specimens were prepared by mixing all the water in an ampoule with 1 g of MTA. As a result, the water to cement ratio, that is one of the most significant variables, has been uncontrolled in most experimental studies reported to date.

We strongly believe that supplying the precise and accurate amount of water is essential for a product such as ProRoot MTA. As less water results in a reduced water to cement ratio and incomplete hydration, unwanted chemical, mechanical and biological consequences can occur. According to the optimized water to cement ratio, which is 1 to 3, it is recommended that users calculate the amount of water themselves rather than relying on the water supplied in an ampoule, which we have demonstrated to be unreliable.

In the short term, the manufacturer should change the batch weighing system in order to supply the correct amount of water. In the long term, the development of a different delivery system is suggested.

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References

Camilleri J (2007) Hydration mechanisms of mineral trioxide aggregate. *International Endodontic Journal* **40**, 462–70.

Eglinton MS (1987) *Concrete and Its Chemical Behavior*, 1st edn. London: Thomas Telford.

Felekoglu B, Türkel S, Baradan B (2007) Effect of water/cement ratio on the fresh and hardened properties of self-compacting concrete. *Building and Environment* **42**, 1795–802.

Fridland M, Rosado R (2003) Mineral trioxide aggregate (MTA) solubility and porosity with different water-to-powder ratios. *Journal of Endodontics* **29**, 814–7.

Ishikawa K, Takagi S, Chow LC, Ishikawa Y, Eanes ED, Asaoka K (1994) Behavior of a calcium phosphate cement in simulated blood plasma in vitro. *Dental Materials* **10**, 26–32.

Lawley GR, Schindler WG, Walker WA III, Kolodrubetz D (2004) Evaluation of ultrasonically placed MTA and fracture resistance with intracanal composite resin in a model of apexification. *Journal of Endodontics* **30**, 167–72.

Lee YL, Lee BS, Lin FH, Yun Lin A, Lan WH, Lin CP (2004) Effects of physiological environments on the hydration behavior of mineral trioxide aggregate. *Biomaterials* **25**, 787–93.

Namazikhah MS, Nekoofar MH, Sheykhrezae MS *et al.* (2008) The effect of pH on surface hardness and microstructure of mineral trioxide aggregate. *International Endodontic Journal* **41**, 108–16.

Nekoofar MH, Adusei G, Sheykhrezae MS, Hayes SJ, Bryant ST, Dummer PMH (2007) The effect of condensation pressure on selected physical properties of mineral trioxide aggregate. *International Endodontic Journal* **40**, 453–61.

Papadakis VG, Antiohos S, Tsimas S (2002) Supplementary cementing materials in concrete Part II: A fundamental estimation of the efficiency factor. *Cement and Concrete Research* **32**, 1533–8.

Roy DM, Gouda GR (1975) Optimization of strength in cement pastes. *Cement and Concrete Research* **5**, 153–62.

Taylor HFW (1997) *Cement Chemistry, 2nd edn*. London, UK: Thomas Telford.

Torabinejad M, Chivian N (1999) Clinical applications of mineral trioxide aggregate. *Journal of Endodontics* **25**, 197–205.

Torabinejad M, Dean J (1995) *Tooth Filling Material and Method of Use*. United State Patent No. 5,415,547 USA: Loma Linda University.

Torabinejad M, Hong CU, McDonald F, Pitt Ford TR (1995) Physical and chemical properties of a new root-end filling material. *Journal of Endodontics* **21**, 349–53.

Walker MP, Diliberto A, Lee C (2006) Effect of setting conditions on mineral trioxide aggregate flexural strength. *Journal of Endodontics* **32**, 334–6.

A follow-on from the response from Tulsa Dental specialities

Dr Nekoofar and colleagues have confirmed that the samples of ProRoot MTA evaluated above were from kits well within their expiry date.