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# Influence of composition on setting kinetics of new injectable and/or fast setting tricalcium silicate cements

H.M. Setbon<sup>a,d,\*</sup>, J. Devaux<sup>b,d</sup>, A. Iserentant<sup>e</sup>, G. Leloup<sup>a,c,d</sup>,  
J.G. Leprince<sup>a,c,d</sup>

<sup>a</sup> Advanced Drug Delivery and Biomaterials, Louvain Drug Research Institute, Université catholique de Louvain, Brussels, Belgium

<sup>b</sup> Institute of Condensed Matter and Nanosciences – Bio- and Soft-Matter, Université catholique de Louvain, Louvain-la-Neuve, Belgium

<sup>c</sup> School of Dentistry and Stomatology, Université catholique de Louvain, Brussels, Belgium

<sup>d</sup> Center for Research and Engineering on Biomaterials (CRIBIO), Université catholique de Louvain, Brussels, Belgium

<sup>e</sup> Earth and Life Institute – Environmental Sciences, Université catholique de Louvain, Louvain-la-Neuve, Belgium

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## ABSTRACT

**Objectives.** New commercial tricalcium silicate based cements were elaborated to improve handling properties and setting time. The goals of the present work were: (i) to determine the composition of the new injectable and/or fast setting calcium silicate based cements, and (ii) to investigate the impact of the differences in composition on their setting kinetics. **Methods.** The materials considered were Angelus MTA<sup>TM</sup>, Biodentine<sup>TM</sup>, MM-MTA<sup>TM</sup>, MTA-Caps<sup>TM</sup>, and ProRoot MTA<sup>TM</sup> as control.

Elemental composition of materials was studied by Inductively Coupled Plasma-Atomic Emission Spectroscopy and X-ray Energy Dispersive analysis, whereas phases in presence were analyzed by Micro-Raman spectroscopy and X-ray Diffraction analysis and cement surface by Scanning Electron Microscope. Setting kinetics was evaluated using rheometry. **Results.** Elemental analysis revealed, for all cements, the presence of three major components: calcium, silicon and oxygen. Chlorine was detected in MM-MTA, MTA-Caps and Biodentine. Different radio-opacifiers were identified: bismuth oxide in ProRoot MTA, Angelus MTA and MM-MTA, zirconium oxide in Biodentine and calcium tungstate (CaWO<sub>4</sub>) in MTA-Caps. All cements were composed of di- and tri-calcium silicate, except Biodentine for which only the latter was detected. Major differences in setting kinetics were observed: a modulus of  $8 \times 10^8$  Pa is reached after 12 min for Biodentine, 150 min for MM-MTA, 230 min for Angelus MTA and 320 min for ProRoot MTA. The maximum modulus reached by MTA-Caps was  $7 \times 10^8$  Pa after 150 min.

**Significance.** Even if these cements possess some common compounds, major differences in their composition were observed between them, which directly influence their setting kinetics.

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\* Corresponding author at: Université catholique de Louvain, Louvain Drug Research Institute, Avenue E. Mounier 73, B-1200 Brussels, Belgium. Tel.: +32 2 764 57 50.

E-mail address: [hugosetbon@gmail.com](mailto:hugosetbon@gmail.com) (H.M. Setbon).

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## 1. Introduction

Mineral Trioxide Aggregate (MTA) is a tricalcium silicate based cement for dental applications, which was initially developed as a root-end filling material. The first formulation, commercialized during the 90s under the name ProRoot MTA (Dentsply, Tulsa, OK, USA), was a 4:1 Portland cement:bismuth oxide mixture [1]. This cement was composed of hydrophilic particles of di- and tri-calcium silicate. During the hydration reaction, these particles react and form a calcium silicate hydrated gel (C-S-H) that hardens with time, and calcium hydroxide. Besides the application as a root-end filling material, ProRoot MTA possesses many qualities needed for other applications in endodontics: setting in wet conditions, good marginal adaptation [2,3], dimensional stability, anti-bacterial activity due to its high alkalinity, biocompatibility [4–6] and bioactivity [7]. Recently, it has been shown that ProRoot MTA can interact with phosphate containing fluids and generate a mineral precipitate on its surface [8]. An interfacial layer can also be created between the tooth and the cement, along with intra-tubular mineral precipitation, which was described as biomineralization [9]. Biocompatibility and bioactivity of ProRoot MTA have largely been demonstrated by previous studies [4–7,10], as well as its capacity to promote dental pulp cell proliferation [11] and to induce cell differentiation [12], without affecting cellular viability [12,13]. It is therefore used in numerous clinical applications, such as pulp capping procedures, repair of perforation, management of root resorptions, apexification of immature necrotic tooth, root-end filling or in the more recently introduced regenerative endodontic procedures [14–17]. ProRoot MTA (Dentsply, Tulsa, OK, USA) is the most widely studied material among calcium silicate based cements but it has two major drawbacks: poor handling properties and long setting time (165 min) [18]. Therefore, several new silicate-based cements were developed and introduced to the market, claiming improved setting and/or handling characteristics: Angelus MTA (Angelus, Londrina, PR, Brazil), Biodentine (Septodont, Saint Maur des Fosses, France), MM-MTA (Micromega, Besançon, France), and MTA-Caps (Acteon, Merignac, France). Like ProRoot MTA, Angelus MTA promotes the biomineralization process and is considered as a biocompatible and bioactive material [19–22]. Biodentine was commercialized in 2010 [23] and was proposed for similar applications as ProRoot MTA (i.e. pulp capping, perforation repair, apexification and root-end filling) but also as a dentin substitute. Beside its good biocompatibility [24] and reduced setting time, Biodentine showed improved mechanical properties compared to ProRoot MTA and Angelus MTA [25,26], but comparable efficacy for dental pulp capping and induction of dentin bridge formation [5,27]. Recently, Biodentine was demonstrated to possess the ability to stimulate biomineralization and induce odontoblastic differentiation of dental pulp cells [28]. Finally, a recent clinical study evaluating Biodentine as posterior restoration has reported satisfactory results for up to 6 months [29]. MM-MTA and MTA-Caps, which are presented in injectable capsules, were also proposed for similar applications as ProRoot MTA. There is currently no literature available on their mechanical properties but a recent study demonstrated a comparable biocompatibility and

odontogenic potential between MM-MTA and ProRoot MTA [30]. According to the manufacturers, the setting time of these alternative materials are reduced to 15 min for Angelus MTA, 9 min for Biodentine, and about 20 min for MM-MTA and MTA-Caps. These reduced setting times could allow one-visit treatments, which is not possible with the ProRoot MTA. Regarding handling characteristics, Biodentine is presented in mixing capsules, but needs to be placed in the cavity using hand instruments. MM-MTA and MTA-Caps are dispensed in capsules with a tip, enabling mixing and direct injection into the cavity (Fig. 1). The improvements put forward for these new products are potentially very valuable for clinicians. However, little or no information is currently available regarding the composition of these new materials. The importance of material composition is especially important at the surface of the cements, as it influences their interaction with the restorative materials on the one hand (glass ionomer cements, resin-based adhesives and composites, etc.), and with the living tissues on the other hand (pulp, periodontal ligament, or periapical bone). Material composition is also crucial in terms of setting kinetics. For example, the presence of elements like chlorine and sulfur, or the increased proportion of tricalcium instead of dicalcium silicate, were shown to impact the setting reaction significantly [31,32]. Hence, the aim of the present work was to first to determine the composition of the new injectable and/or fast setting tricalcium silicate cements, and second to investigate the impact of the differences in composition on their setting kinetics.

## 2. Materials and methods

The five cements considered in this work (ProRoot MTA, Angelus MTA, MM-MTA, Biodentine, and MTA-Caps) were prepared according to the manufacturers recommendations. ProRoot MTA (batch number 10003598) was mixed at a 3:1 powder:liquid ratio and Angelus MTA (batch number 21013) at a ratio of one powder spoon for one liquid drop, both on a non-absorbent paper. Biodentine (batch number B03844) was mixed by adding five liquid drops to the powder, MM-MTA (batch number 7111803) and MTA-Caps (batch number 7203285) were activated, and all three cements were then mixed in an amalgamator at 4300 oscillations per minute during 30 s. Cements were allowed to set during three days at 37.5 °C and saturated humidity in an incubator (INE400, Memmert GmbH, Germany), as described by Camilleri et al. [33].

### 2.1. Elemental composition

Cements were prepared in stainless steel molds (1 mm thick, 10 mm diameter) and powder was spread on double-sided tape. Semi-quantitative analysis of both powders and hydrated cements were obtained by X-ray energy dispersive analysis (EDX) (Jeol JSM 7600f, Jeol Ltd., Tokyo, Japan) under a 20 kV accelerating voltage, during 400 s per sample ( $n=3$ ) at 750 $\times$  magnification.

Quantitative analysis of powders were performed by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) using a iCAP 6500 ICP Spectrometer (Thermo Scientific, Waltham, MA, USA).

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