Biodentine and Mineral Trioxide Aggregate: An Analysis of Solubility, pH Changes and Leaching Elements

Sawsan Taha Hassan Abu Zeid*; Osama S. Alothmani* Mohammed Khalil Yousef**

*Department of Endodontics, Faculty of Dentistry, King Abdulaziz University, Jeddah, Kingdom of Saudi Arabia
** Department of Operative Dentistry, Faculty of Dentistry, King Abdulaziz University, Jeddah, Kingdom of Saudi Arabia.
myousf@kau.edu.sa

Abstract: Background/Aim: Biodentine is a new restorative material used in traumatized tooth. The aim of this study was to compare the behavior of Biodentine and white ProRoot when immersed in deionized water for 28 days. **Methods**: Standard discs of freshly mixed materials (n = 10/material) were immersed in deionized water for different intervals (1, 7, 14 and 28 days). Solubility, pH changes and calcium ions release were measured at each interval whereas the released silicon, aluminum, phosphorus, iron, manganese and magnesium were quantified only at the 28th day. With significance level set at 5%, differences between both materials were analyzed using Student's *t*-test while data obtained for each material at different periods were compared using ANOVA and Tukey's *post hoc* test. **Results:** With time, solubility of Biodentine significantly increased while it maintained consistently high pH range (11.16 ± 0.52 to 11.83 ± 0.07). Weight changes recorded for ProRoot were not significant ($P > 0.05$) associated with a gradual drop in its pH from 11.53 ± 0.07 on the 1st day to 9.72 ± 0.29 after 28 days ($P = 0.00$). Biodentine released significantly more calcium ions and iron traces compared to ProRoot ($P < 0.05$). In contrast, ProRoot released significantly more silicon, aluminum, phosphorus and manganese ($P < 0.05$). Both materials released comparable quantities of magnesium ($P = 0.06$). **Conclusion**: Compared to ProRoot, Biodentine specimens exhibited higher solubility, prolonged alkalinity and increased calcium release over the 28 days. Elemental traces detected from Biodentine and ProRoot differed in type and amount.

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Keywords Calcium silicate cements, Biodentine, Solubility, pH changes, Calcium ions release, leaching elements.

1. Introduction

Mineral Trioxide Aggregate (MTA) represents an optimum option for pulp capping, perforation repair, root-end filling and apical barrier producers due its bioactive properties. However, its long setting time and difficult handling properties are among its recognized downsides (1). Biodentine (Septodont, Saint-Maur-des-Fossés, France) is another calcium silicate-based material possessing bioactive properties comparable to MTA. Its endodontic indications are similar to those of MTA with added advantages of being fast-setting and easier to manipulate. The powder of Biodentine is composed of tricalcium silicate, calcium carbonate and a radio-pacifier (zirconium oxide) whereas its liquid comprises a water reducing agent and calcium chloride accelerating the setting reaction (2, 3).

Bioactivity points to the cement's ability to produce an apatite-like layer on its surface when it comes in contact with body fluids *in vivo* (4) or with simulated tissue fluids *in vitro* (5). Physical and chemical properties of MTA have been extensively investigated (6) and its bioactivity has been confirmed (7). However, data considering the behavior of Biodentine when it comes to contact with fluid are limited. Biodentine released significantly higher amounts of calcium ions compared to MTA and BC sealer over a short observation period of 168 hours when immersed in phosphate-buffered solution (8). Upon immersion in Hank's balanced salt solution for 28 days, Biodentine samples maintained high pH levels in the range of 11.7 - 12.4 and released considerable amount of calcium ions (9). However, none of these studies quantified the release of silicon, aluminum, phosphorus, iron, manganese and magnesium from Biodentine.

Tissue response to filling materials could be affected by the material's solubility (10) and pH controlling its antibacterial activity (11). Further, material bioactivity depends mainly on the elements leaching into surrounding tissues (7, 12). Since the available information on Biodentine is scarce, the aim of this study was to investigate the solubility, pH changes and the release of calcium and other leaching elements from Biodentine and to compare that with ProRoot white-MTA (Dentsply Tulsa Dental, Tulsa, OK, USA) when these materials were immersed in deionized water. The null hypothesis was that there was no significant difference between both materials regarding their solubility, pH levels and leaching elements.

2. Materials and Methods

The procedures were approved by the institutional ethical committee (# 092-13). Standard discs of freshly prepared Biodentine and white-MTA (wMTA) (n = 10 for each material), mixed according to their manufacturer's recommendations, were prepared. The discs were 15 mm in diameter and 3 mm in thickness (13). After complete setting, each disc was tied with impermeable nylon thread and its initial weight (W_0) was recorded using an analytical balance machine (Balance and Scale Model AW-220, Shimadzu Corporation, Kyoto, Japan). The discs were then immersed in polypropylene tubes containing 20 mL deionized water for different intervals (1, 7, 14 and 28 days) at 37°C/100% humidity.

Solubility testing:

At each interval, the discs were removed from their tubes, dried with blotted paper and left 24 hour to completely dry. Final weights were recorded (Wf_1, Wf_7, Wf_{14} and Wf_{28}). Each weight measurement was done thrice and its average was calculated. Changes in the weight of the discs reflected the extent of their solubility. The Solubility (%) was calculated following the equation (14):

$$\text{Solubility (\%)} = \frac{W_0 - W_f}{W_0} \times 100$$

pH changes:

At each interval, the pH of the deionized water was recorded using pH meter (HANNA pH 211, Hanna Instruments, Woonsocket, RI, USA) pre-calibrated against standard solutions with pH= 4.0 and 7.0 at 25°C.

Calcium ions and other leaching elements analysis:

At each interval, the amount of calcium ions released into the deionized water was measured using Inductively Coupled Plasma (ICP) spectrometer (Optical Emission Spectrometer, JY-Ultima 2, HORIBA Science, USA). After 28 days, the concentrations of six leaching elements including silicon, aluminum, phosphorus, iron, manganese and magnesium were also quantified using the ICP spectrometer.

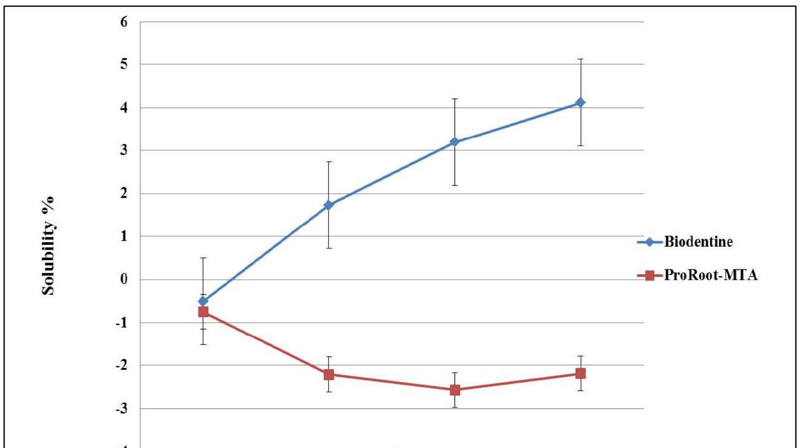
Statistical analysis:

Utilizing the SPSS software (Version 16.0; SPSS, Inc, Chicago, IL), and with level of significance set at $P < 0.05$, Biodentine and wMTA data were statistically compared using Student's *t*-test. Values of each material obtained at different intervals were compared using ANOVA and Tuckey's *post hoc* test.

3. Results

Solubility evaluation:

Biodentine exhibited significantly higher solubility than wMTA on Day 7, 14 and 28 ($P < 0.05$). Despite an initial weight gain, Biodentine's Solubility (%) increased overtime approaching 4.11 ± 1.03% on Day 28. The recorded weight changes associated with wMTA specimens over the 28 days of observation were all not significant ($P > 0.05$) (Figure 1).



	Day 1	Day 7	Day 14	Day 28
Biodentine	-0.51	1.73	3.19	4.11
ProRoot-MTA	-0.75	-2.21	-2.57	-2.19

Figure 1: Mean values of Solubility (%) for Biodentine and wMTA over the 28 days. (-) indicates an increase in wieght

pH changes:

Biodentine's pH was consistently and significantly higher than wMTA over the entire observation period ($P < 0.05$). Biodentine maintained strong alkalinity over the 28 days (pH range = 11.16 ± 0.52 to 11.83 ± 0.07). Although Biodentine's pH peaked on Day 14, this escalation was statistically not significant compared with its pH recorded at Day 1 (*P* = 0.56) or Day 7 (*P* = 0.89). Interestingly, this rise was followed by a significant drop at Day 28 (*P* = 0.00). The wMTA's pH gradually dropped from 11.53 ± 0.07 on Day 1 to 9.72 ± 0.29 after 28 days (*P* = 0.00) (Figure 2).

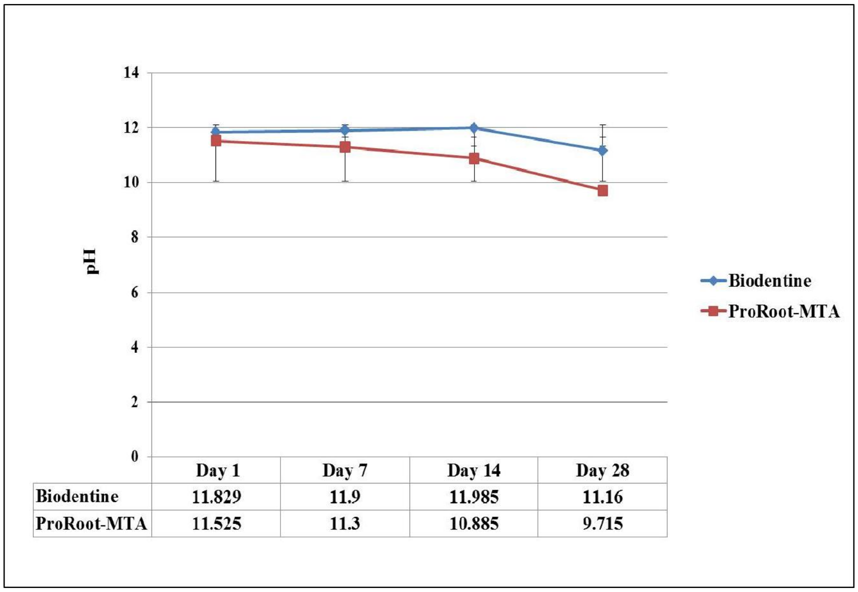


Figure 2: Mean pH values for Biodentine and wMTA over the 28 days

Calcium ions release:

Biodentine released significantly more calcium ions compared to wMTA throughout the experiment (*P* < 0.05). As shown in Figure 3, the greatest calcium ions release from both materials was recorded on Day 1 consistently declining afterwards. For Biodentine, this drop was significant only after one week (*P* = 0.00) whereas differences between Day 7, 14 and 28 were not significant (*P* > 0.05). For wMTA, the reduction was significant for every interval (*P* < 0.05).

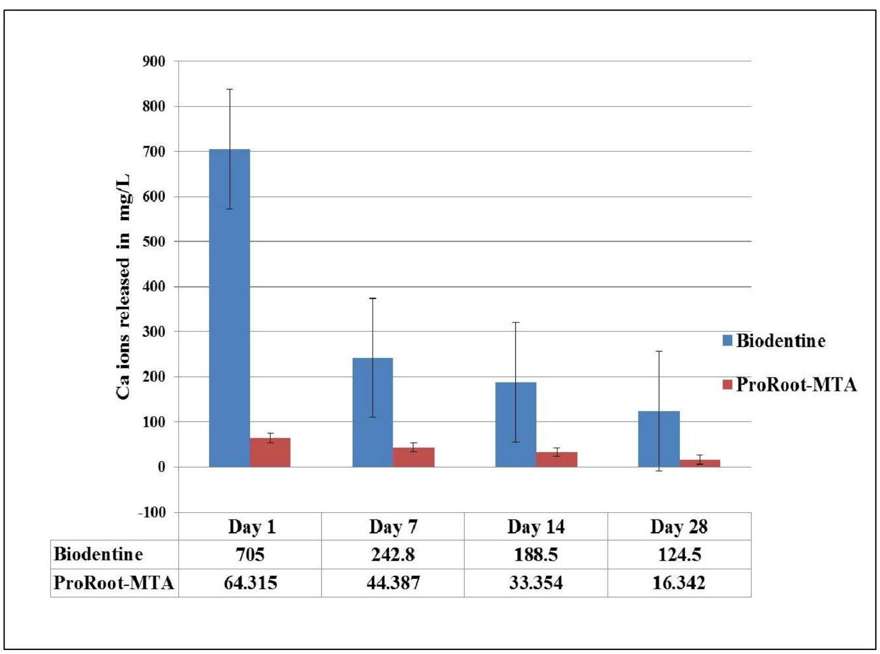
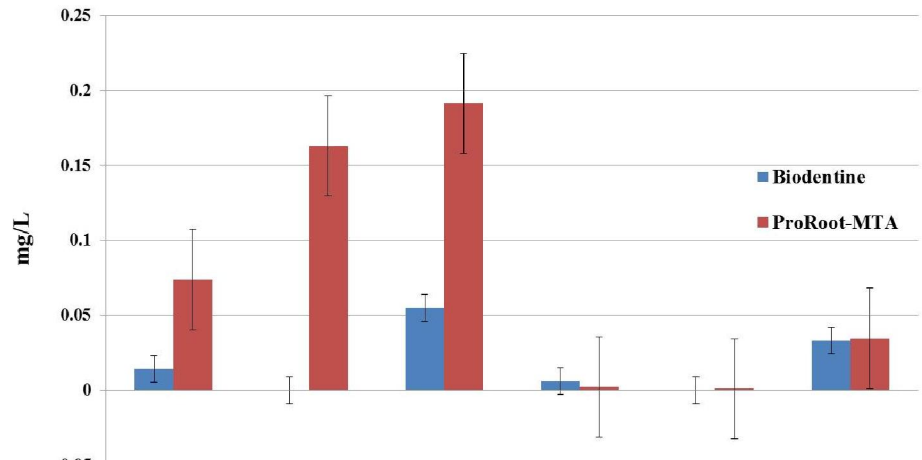


Figure 3: Mean values of calcium ions release (mg/L) from Biodentine and wMTA over the 28 days

Cumulative leaching elements after 28 days:

Mean quantities of elements which leached from both materials are represented in Figure 4. wMTA released significantly higher amounts of silicon, aluminum, phosphorus and manganese ($P < 0.05$) while Biodentine samples leached significantly higher iron traces ($P = 0.01$). Both materials released comparable quantities of magnesium ($P = 0.06$).



	Si	Al	P	Fe	Mn	Mg
Biodentine	0.014	0	0.0549	0.006	0	0.0331
ProRoot-MTA	0.0738	0.163	0.1913	0.002	0.001	0.0347

Figure 4: Mean cumulative values of leaching elements (mg/L) from Biodentine and wMTA after 28 days

4. Discussion

The current study assessed the solubility, pH changes and the release of several elements when standard discs of Biodentine and wMTA were immersed in deionized water for a period of 28 days. Based on the findings, the null hypothesis was rejected. Progressive Biodentine dissoultion was noted while weight changes noted for wMTA specimens were not significant. In comparison to wMTA samples, higher alkalinity and noticeable calcium ions release were sustained from Biodentine samples. Minimal amounts of variety of leaching elements were detected from the tested materials.

Although Biodentine and MTA are derivatives of Portland cement (3, 15), the current study identified some differences in their physical and chemical behaviors. Biodentine's solubility eventually exceeded acceptable limits stated by the ANSI/ADA standards (13). This could be attributed to the early washout of Biodentine upon contact with fluids. Further, Biodentine's low water sorption could have also affected its solubility (16). Althought statistically insignificant, the increase in wMTA weight in the first 14 days observed in the present study could be explained by its hydration reaction (1). The weight loss recorded on Day 28 for wMTA samples is in line with a previous report demonstrating increased MTA solubility over extended observation (17).

Results of the current study showed that both tested materials initially exhibited high alkalinity. Biodentine's pH peaked on Day 14 followed by a significant reduction at the end of observation period recording 11.16 ± 0.52. The pH of wMTA samples progressively dropped over the 28 days to reach 9.72 ± 0.29 (Figure 2). Regarding calcium ions release, Biodentine released significantly higher amounts compared to wMTA throughout the experiment. Nevertheless, calcium ions release from both materials dropped with time (Figure 3). Biodentine's sustained high alkalinity and calcium ions release have been observed (9). The setting reaction of calcium silicate cements, including Biodentine and MTA, results in the formation of calcium silicate hydrate and calcium hydroxide (6, 9). In the presence of moisture, calcium hydroxide dissociates to hydroxyl ions, responsible for the increased alkalinity and antibacterial activity, and calcium ions that promote material bioactivity and apatite layer formation (1). Increased solubility has been correlated with increased dissolution of calcium hydroxide (18). Thus, the higher solubility of Biodentine in comparison to wMTA (Figure 1) could explain the prolonged alkaline pH (Figure 2) and the larger calcium ions release (Figure 3) observed in the current study. High alkalinity could induce dentinal collagen denaturation facilitating the penetration of calcium ions and mineral exchange establishing the “Mineral Infiltration Zone” at the dentin-Biodentine interface (19).

The amounts of released silicon, aluminum, phosphorus, iron, manganese and magnesium were minimal from both materials although their quantification was done after 28 days. Despite the higher solubility of Biodentine, it released silicon and phosphorus in significantly lower quantities than wMTA (Figure 4). In general, lower amounts of leaching elements from Biodentine could be related to its fast setting reaction and its water reducing agent (20). It seems that solubility does not deteriorate the silicon bulk of hydrated Biodentine (12). Silicon and phosphorus were stronger inducers of dentin remineralization and new bone growth compared to calcium (21, 22). Both materials released nearly equal traces of magnesium (Figure 4). Iron traces released from Biodentine were significantly higher than wMTA. Different results could have been obtained if gray-MTA, known for its higher iron content compared to the white variant (23), was used. Meanwhile, aluminum and manganese were only released from wMTA samples. Addition of impurities in form of oxides such as Al_2O_3, were essential to improve the crystal structure. The minor aluminum constituent detected in wMTA was considered essential for the hydration reaction and improves the strength of the material (24, 25). Manganese traces in wMTA may reduce its cytotoxicity, rejection, inflammatory and allergic reactions (24). Variations in the types and quantities of all these elemental traces seem not to interfere with the bioactivity of Biodentine and wMTA (8).

Conclusion

Under the conditions of current study, Biodentine specimens exhibited higher solubility, prolonged alkalinity and increased calcium release over 28 days compared to wMTA. Elemental traces detected from Biodentine and wMTA differed in type and amount.

Corresponding author:
Dr. Mohammed Khalil **Yousef**, B.D.S, MSD, PhD
Associate Professor, Department of Operative Dentistry, Faculty of Dentistry, King Abdulaziz University. Email: myousf@kau.edu.sa
P.O. Box 80209 Jeddah, 21589
Kingdom of Saudi Arabia

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