# D90: The Strongest Contributor to Setting Time in Mineral Trioxide Aggregate and Portland Cement

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## Abstract

**Introduction:** The setting times of commercial mineral trioxide aggregate (MTA) and Portland cements vary. It was hypothesized that much of this variation was caused by differences in particle size distribution. **Methods:** Two gram samples from 11 MTA-type cements were analyzed by laser diffraction to determine their particle size distributions characterized by their percentile equivalent diameters (the 10th percentile, the median, and the 90th percentile [d90], respectively). Setting time data were received from manufacturers who performed indentation setting time tests as specified by the standards relevant to dentistry, ISO 6786 (9 respondents) or ISO 9917.1 (1 respondent), or not divulged to the authors (1 respondent). In a parallel experiment, 6 samples of different size graded Portland cements were produced using the same cement clinker. The measurement of setting time for Portland cement pastes was performed using American Society for Testing and Materials C 191. Cumulative heat release was measured using isothermal calorimetry to assess the reactions occurring during the setting of these pastes. In all experiments, linear correlations were assessed between setting times, heat release, and the 3 particle size parameters. **Results:** Particle size varied considerably among MTA cements. For MTA cements, d90 was the particle size characteristic showing the highest positive linear correlation with setting time ($r$ = 0.538). For Portland cement, d90 gave an even higher linear correlation for the initial setting time ($r$ = 0.804) and the final setting time ($r$ = 0.873) and exhibited a strong negative linear correlation for cumulative heat release ($r$ = 0.901). **Conclusions:** Smaller particle sizes result in faster setting times, with d90 (the largest particles) being most closely correlated with the setting times of the samples. *(J Endod 2015;■:1–5)*

## Key Words

Hydration, mineral trioxide aggregate, particle size, Portland cement, root canal filling materials, setting time

Mineral trioxide aggregate (MTA) is used widely in endodontics (1). According to the relevant patent, ProRoot MTA (Dentsply Tulsa Dental, Johnson City, TN) contains 80% Portland cement (PC) and 20% bismuth oxide (BO) by mass (2). Variants in composition exist for other MTA cements, namely 70% PC with 30% zirconium oxide (ZO) (3, 4). BO or ZO is included to make the set material radiopaque, with a greater proportion of ZO required to attain radiopacity in comparison with the original version that used BO (3, 4). There are minor variations in composition among MTA cements, such as the inclusion in some products of calcium chloride, calcium carbonate, silicon dioxide, or the removal of calcium sulfate, all of which accelerate the setting of PC (5–7). Rheologic modifiers serve to improve the flow and handling characteristics of MTA; however, their compositions have not been divulged to the authors because they are proprietary. The variation in the compositions of the MTA cements examined in this study is shown in Table 1.

The advertised setting times of commercial MTA cements range from 2.3 minutes with EndoCem Zr (Maruchi, Wonju, South Korea) to 4 hours with Trioxident (VladMiVa, Belgorod, Russia). The range of new products can be confusing to the clinicians, particularly as to why such a large range exists within products, despite all falling under the general term of MTA. These differences may be attributed to their compositions, their particle size distributions (PSDs), and the methodology used to determine the setting time.

Two international standards are used to measure the setting time of MTA; ISO 6876 for endodontic sealers (8) is more commonly used even though MTA is not used as a traditional endodontic sealer in combination with gutta-percha points. The second international standard is ISO 9917.1, which is designed for cements that set with an acid-base reaction (9) and, as such, is more tailored to glass ionomer–type cement-based materials.

Although manufacturers have a vested interest in the marketing information to claim their products are superior to others, their statements on setting time have greater reliability because the statements of conformity to an ISO standard when registering to therapeutic bodies such as the Food and Drug Administration and Conformité Européenne require consistently repeatable results over different production batches.

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**TABLE 1.** Comparison and Composition of MTA Brands

| | Calcium silicates | Calcium aluminates | Calcium sulfate | Bismuth oxide | Zirconium oxide | Calcium carbonate | Silicon dioxide | Phyllosilicates | Rheologic modifier | Calcium chloride |
|---|---|---|---|---|---|---|---|---|---|---|
| Biodentine | Y | | | | Y | Y | | | Y | Y |
| EndoCem MTA | Y | Y | Y | Y | | | | Y | | |
| EndoCem Zr | Y | Y | Y | | Y | | | Y | | |
| EndoSeal | Y | Y | Y | | Y | | | Y | | |
| MM MTA | Y | Y | Y | Y | | Y | | Y | | |
| MTA Angelus | Y | Y | | Y | | | | | | |
| MTA Plus | Y | | Y | Y | | | Y | | Y | |
| Ortho MTA | Y | Y | | Y | | | | | | |
| ProRoot MTA | Y | Y | Y | Y | | | | | | |
| Retro MTA | Y | Y | | | Y | | | | | |
| Trioxident | Y | Y | Y | Y | | | | | Y | |

Some of the literature presents similar findings to the manufacturers' stated setting times, such as ProRoot MTA's stated set time of 4 hours, which corresponds with 2 articles that describe its "final set time" as 4 hours (10, 11). This shows the likely methodology that is advertised in ProRoot MTA's marketing is from ISO 9917.1. Endocem Zr's advertised setting time corresponds with the "initial set time" or ISO 6876 (10). Similarly, MTA Angelus (Angelus, Londrina, Brazil) advertises a set time of 15 minutes, which is consistent with that reported in an article testing the "final set time," which is defined by the American Dental Association's specification 57 for setting times using American Society for Testing and Materials (ASTM) C 266 at 18.33 minutes (12).

Variations between the literature and the manufacturers' testing results may exist because of the differences in the methodology as well as how the samples are processed. Furthermore, the composition of dental materials is subject to change at the discretion of the manufacturer.

Standard test method ASTM C 191 is commonly used for testing the initial and final setting time of PC pastes using a Vicat needle (13). The differences between this Vicat needle test and the Gillmore needle test (ASTM C 266) are compared in Table 2. The 2 ISO standards are somewhat similar to ASTM C 266 (Gillmore needle); ISO 9917.1 uses parameters similar to the defined initial setting time of ASTM C 266, and the parameters used in ISO 6786 are similar to the defined final setting time of ASTM C 266. The American Dental Association's specification 57 for setting time uses ASTM C 266 but with the key difference of the temperature requirement being 37°C.

Another way to assess the ongoing reactions within a material that contribute to setting is to measure their cumulative heat release whereby the thermal energy released by a sample is measured over an arbitrary period of time. In this study, isothermal calorimetry was applied to the PCs to assess their cumulative heat release during the first 4 hours of hydration. For a given set of reactions occurring in the same proportions, the sample with the most energy released on a per mass basis shows the greatest amount of reaction.

A fundamental concept in reaction kinetics is that any reduction of particle size of a powder reactant will result in a higher surface area per unit mass and, therefore, is generally expected to increase the rate of reaction. This increased rate of reaction with a reduced particle size has been observed with PC when used in industrial applications (14). Therefore, an MTA cement with a lower (average) particle size is anticipated to have an accelerated reaction and a reduced setting time. Particle sizes within a given sample of PC typically vary over 3 orders of magnitude (15), and the PSD is typically described using 3 reference points: the 10th percentile (d10), 10% of the estimated particle diameters fall under this size; the median (d50), 50% of the particles fall under this size; and the 90th percentile (d90), 90% of the particles fall under this size.

The particle size distribution of MTA has been assessed in studies by Komabayashi and Spangberg (16, 17) and Ha et al (15). The studies by Komabayashi and Spangberg used flow particle image analysis in which MTA particles flow past a camera that rapidly determines the characteristics of the particles. Within 1 study, Komabayashi and Spangberg (17) used 2 flow particle methods to analyze the PSD, with a lower-power field showing half or more of the particles being between 6 and 10 $\mu$m, whereas another method using a high-power field showed three quarters of the particles to be within 1.5 and 3 $\mu$m (17). Within this study, these 2 methods appear to provide results that are at odds with each other; however, the 2 methods are restricted by the limitations of the machine. A low-power field can only effectively assess 6- to 160-$\mu$m particles; however, through the results of this study, it is evident that there are MTA particles that are unable to be measured because their sizes are smaller than 6 $\mu$m and larger than 40 $\mu$m. Similarly, a high-power field can only measure particles between 1.5 and 40 $\mu$m (17). The subsequent study by Ha et al (15), using laser diffraction for particle analysis on a machine that can effectively measure between 0.05 and 550 $\mu$m, showed that ProRoot MTA has 80% of its particles falling between 1.13 and 4.30 $\mu$m, which matches the high-power field findings of Komabayashi and Spangberg. MTA Angelus exhibited 80% of its particles falling between 4.15 and

**TABLE 2.** Summary of Setting Time Standards Commonly Encountered in Dentistry and for Portland Cement

| Standard | Setting time term used | Mass (kg) | Diameter (mm) | Indentation pressure (MPa) | Indentation amount for setting | Test temperature (°C) | Needle type |
|---|---|---|---|---|---|---|---|
| ISO 6876 | Setting time | 0.100 | 2.00 | 0.312 | No visible penetration | 37 | Gillmore |
| ISO 9917.1 | Net setting time | 0.400 | 1.00 | 4.991 | No visible penetration | 37 | Gillmore |
| ASTM C 266 | Initial setting time | 0.113 | 2.12 | 0.315 | No visible penetration | 23 | Gillmore |
| ASTM C 266 | Final setting time | 0.454 | 1.06 | 5.037 | No visible penetration | 23 | Gillmore |
| ASTM C 191 | Initial setting time | 0.300 | 1.00 | 3.743 | <25 mm penetration | 23 | Vicat |
| ASTM C 191 | Final setting time | 0.300 | 1.00 | 3.743 | No visible penetration | 23 | Vicat |

ASTM, American Society for Testing and Materials.

**TABLE 3.** Summary of Particle Size Distributions of Portland Cement, Mineral Trioxide Aggregate, Their Setting Times, and Cumulative Heat Release

| Material used | d10 ($\mu$m) | d50 ($\mu$m) | d90 ($\mu$m) | Initial setting time (h) | Final setting time (h) | 4-h heat release (J/g cem) |
|---|---|---|---|---|---|---|
| Types of Portland cement | | | | | | |
| Type III PC | 0.975 | 6.768 | 17.441 | 1.92 | 2.67 | 36.56 |
| Type II/V PC | 1.245 | 11.237 | 32.912 | 2.17 | 3.18 | 33.03 |
| 25:75 blend of type I/II and III PC | 1.079 | 7.875 | 24.180 | 2.89 | 3.70 | 29.44 |
| 50:50 blend of type I/II and III PC | 1.152 | 9.072 | 29.034 | 3.23 | 4.11 | 28.96 |
| 75:25 blend of type I/II and III PC | 1.473 | 12.460 | 45.086 | 3.75 | 4.64 | 19.50 |
| Type I/II coarse PC | 1.850 | 17.780 | 49.870 | 3.76 | 4.97 | 20.71 |
| Correlation with initial setting time: $r$ = | 0.748 | 0.674 | 0.804 | | | |
| Correlation with final setting time: $r$ = | 0.837 | 0.781 | 0.873 | | | |
| Correlation with 4-h heat release: $r$ = | −0.835 | −0.769 | −0.901 | | | |
| Brand of mineral trioxide aggregate | | | | | | |
| Biodentine | 1.170 | 3.481 | 7.510 | | 0.20 | |
| EndoCem MTA | 1.086 | 3.247 | 8.328 | | 0.08 | |
| EndoCem Zr | 0.967 | 2.490 | 6.035 | | 0.04 | |
| EndoSeal MTA | 0.838 | 2.060 | 5.394 | | 0.10 | |
| MM MTA | 1.844 | 7.286 | 16.511 | | 0.33 | |
| MTA Angelus | 2.606 | 8.493 | 23.739 | | 0.25 | |
| MTA Plus | 1.259 | 4.740 | 10.267 | | 1.25 | |
| OrthoMTA | 0.966 | 3.384 | 21.117 | | 5.50 | |
| ProRoot MTA | 0.980 | 5.070 | 19.386 | | 4.00 | |
| RetroMTA | 1.225 | 8.218 | 24.948 | | 0.04 | |
| Trioxident | 2.969 | 12.789 | 32.259 | | 4.00 | |
| Correlation with setting time: $r$ = | 0.110 | 0.212 | 0.538 | | | |

42.55 $\mu$m. However, the study by Ha et al used water as the dispersant (15), which can react with MTA, resulting in the dissolution of some larger particles into smaller particles as well as the formation of calcium silicate hydrate structures forming larger particles.

Laser diffraction involves the cement sample passing through a laser light source whereby the scattering of the beam, the angular dependence, and the intensity of the light are measured by detectors (18). The particle size, shape, and optical properties of the particles influence the scattering of the beam, requiring the use of mathematical models to estimate the PSD (18). The 2 most common mathematical methods used are the Mie and Fraunhofer models, with little difference between them when particle sizes are above 50 $\mu$m; the Mie model is recommended for particles below 50 $\mu$m in size.

Clinicians are now seeing multiple variations of MTA that set faster than the original formulations, with their respective marketing attributing their properties to unique differences in their formulations. This study explores whether a correlation exists between the setting time of MTA and some measure of the powder's PSD, irrespective of the formulation. The inclusion of PC in this study serves to confirm whether any trends identified with MTA are replicable with PC, particularly when the cements all originate from the same clinker, albeit ground to different particle sizes.

## Materials and Methods

### PSD

The standard method of laser diffraction was used to measure the PSD of MTA cement powders, as performed by Ha et al (15), with a difference being that methanol was used as the solvent instead of water to prevent hydration of the particles during analysis. One gram of each MTA sample was suspended in 1 L methanol within a Mastersizer 2000 (Malvern Instruments, Worcestershire, UK), with the analysis being completed within 4 seconds. The Mastersizer 2000 can assess particle sizes from 0.02–2000 $\mu$m.

The methanol solution was under continuous ultrasonic agitation to prevent particle agglomeration. Based on the equipment manufacturer's recommendations, the refractive index (RI) used for MTA cements nominally containing 80% PC and 20% BO was 1.844 (calculated from the weighted average of the 2 components, respectively [1.68 and 2.5]). Likewise, the RI used for MTA products nominally containing 70% PC and 30% ZO was 1.82 (calculated from the weighted average of the 2 components, respectively [1.68 and 2.25]). For both variants, the particle absorption index used was 0.1. The ability to classify each of the 11 materials used according to their composition was based on data provided by the manufacturers although exact compositions were not disclosed to the investigators.

### Materials

The commercial MTA products that were analyzed using an RI of 1.842 were EndoCem MTA (Lot no. C2304160610; Maruchi, Wonju, Republic of Korea), MM MTA (Lot no. 7302238; MicroMega, Besancon, France), MTA Angelus (Lot no. 21934), MTA Plus (Lot no. 85001; Avalon Biomed, Bradenton, FL), OrthoMTA (Lot no. OM1305D02; BioMTA, Seoul, Republic of Korea), ProRoot MTA (Lot no. 9001766), and Trioxident (Lot no. 0509; VladMiVa, Belgorod, Russia). The commercial brands of MTA or related hydraulic cements that were analyzed using an RI of 1.82 were Biodentine (Lot no. B01564; Septodont, Saint Maur des Faussés, France), EndoCem Zr (Lot no. ZC2403120516), EndoSeal MTA (Lot no. SC402080525, Maruchi), and RetroMTA (Lot no. RM1304D05, BioMTA).

Three cements, having different fineness but manufactured from the same cement clinker, were supplied by Lehigh Cement Corporation (Redding, CA). These 3 cements from fine to coarse are classified according to ASTM C150 as being type III, type II/V, and type I/II PC. From these 3 cements, 3 more cements of intermediate levels of fineness were created by blending the coarsest and finest cements at 3 different ratios as seen in Table 3. Similar to the determination of MTA's PSDs, the PC was also assessed using wet laser diffraction, as described by Bentz (19).

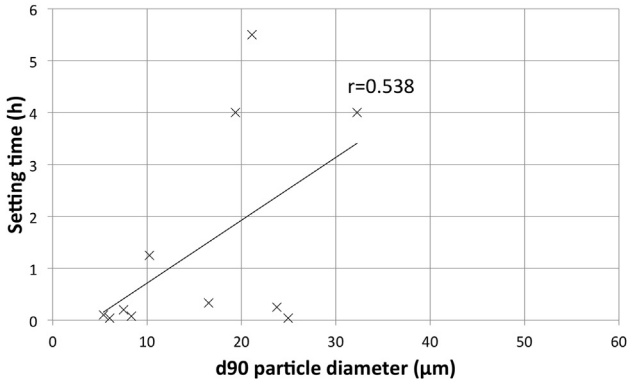


**Figure 1.** Setting time versus d90 of MTA.

## Setting Time

Data for the setting time of MTA were compiled through direct contact with the manufacturers. With 2 exceptions, all confirmed the testing methodology used was ISO 6876. The manufacturers of Biodentine used ISO 9917.1, whereas the manufacturers of ProRoot did not divulge their methodology. The correlation of d10, d50, and d90 of the PSD with the setting time was assessed for strength of correlation via determination of $r$, the Pearson correlation coefficient.

The initial and final setting times of PC were determined using a Vicat needle as described in ASTM C 191 (13) for pastes with a water-to-cement mass ratio of 0.40 prepared in a high shear blender (19).

## Heat of Hydration

Heat of hydration was measured over a period of 4 hours using isothermal calorimetry of sealed 5-g samples of premixed pastes of cement and water (same water-to-cement mass ratio of 0.4 as the setting time specimens) (19).

# Results

## MTA

The PSDs of MTA products are summarized in Table 3. When data from all 11 materials were combined, the highest positive correlation with setting times was found for d90, which gave a positive Pearson correlation of $r = 0.538$. This is illustrated in Figure 1. In contrast, the correlations for d50 and d10 were significantly less (ie, 0.212 and 0.110, respectively). The equation describing the correlation for setting time ($t$) to d90 was as follows:

$$t(h) = 0.1211 \times (d_{90}) - 0.4965 \qquad \text{(Equation 1)}$$

The plot of Equation 1 with the actual results is shown in Figure 1 in which a modest fit of the experimental data to this function can be seen. Some of the MTA cements likely contain chemical accelerators or are based on different chemistries, confounding the observed relationship with particle size.

## PC

A summary of the PSD characteristics of the PCs and their measured setting times and heat release is provided in Table 3, whereas the results are shown graphically in Figure 2 (14). For the initial setting time, positive correlations exist with d10 ($r = 0.748$) and d50 ($r = 0.674$), but the highest correlation exists with d90 ($r = 0.804$). The final setting time is positively correlated with d50 ($r = 0.781$) and is more strongly correlated with d10 and d90 although the d90 strengths of correlation ($r = 0.872$) are greater than that of d10 ($r = 0.837$). Heat release is negatively correlated with all 3 PSD

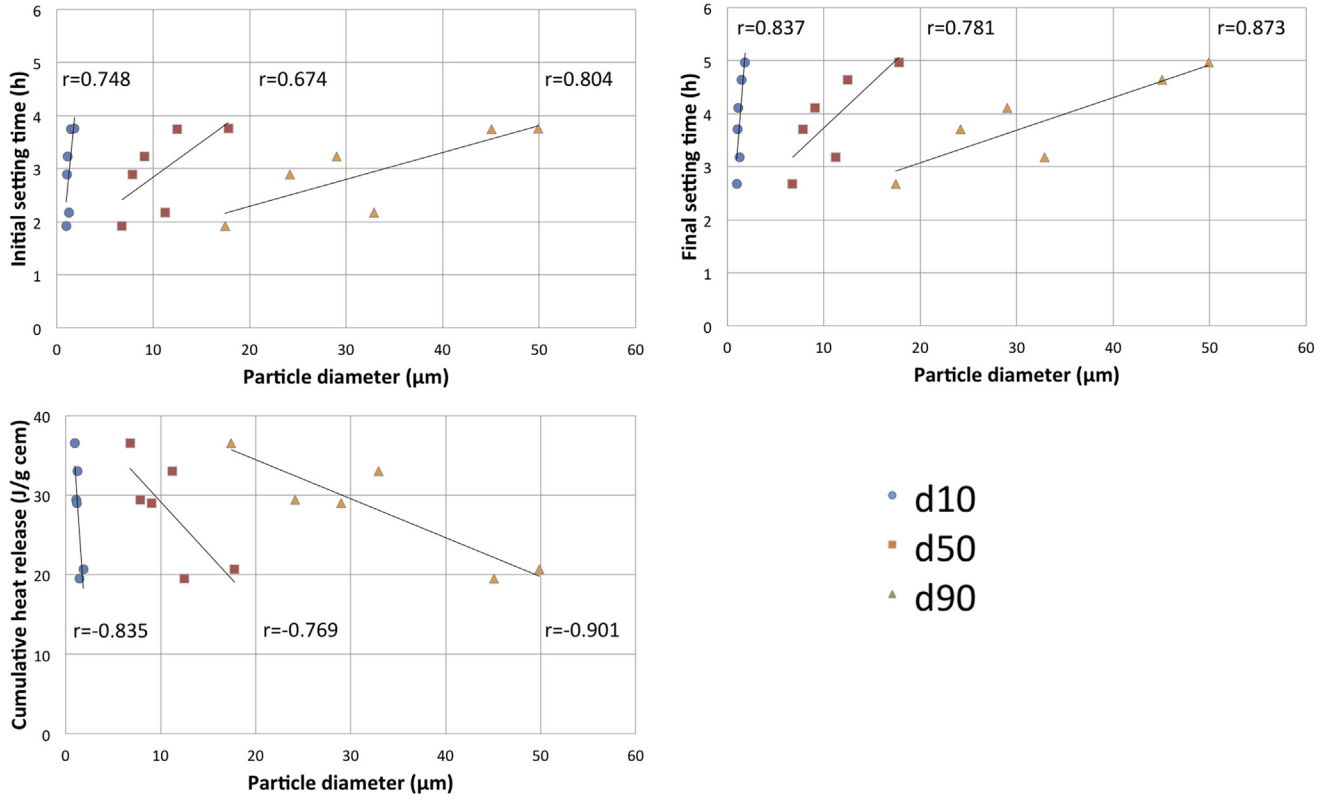


**Figure 2.** Setting times and cumulative heat release versus particle size of Portland cement.

parameters, with once again the highest magnitude correlation being found for d90 ($r = -0.901$). The equations describing the correlation for the initial setting time ($_it$), the final setting time ($_ft$), and cumulative heat ($h$) to d90 were as follows:

$$_it(h) = 0.0507 \times (d_{90}) + 1.276 \quad \text{(Equation 2)}$$

$$_ft(h) = 0.0615 \times (d_{90}) + 1.8428 \quad \text{(Equation 3)}$$

$$h\left(\frac{J}{g}\right) = -0.4906 \times (d_{90}) + 44.264 \quad \text{(Equation 4)}$$

The heat released after 4 hours is greatest for samples with the smaller particle sizes, illustrating a greater exothermic reaction has occurred within that timeframe. This shows smaller particles result in a greater exothermic reaction within that period of time, which produces a quicker setting time.

## Discussion

Equation 1 describes a modest trend line with 4 notable outliers, OrthoMTA, ProRoot MTA, MTA Angelus, and RetroMTA. If the assumption is made that ProRoot's advertised set time is the "final set time" of ASTM C 266, or ISO 9917.1, then the preferred value to include within this study is the initial set time, or ISO 6876, which would be approximately 78 minutes (10), with the setting time of ProRoot MTA falling much closer to the trend line. MTA Angelus and RetroMTA both have setting times substantially quicker than the trend line, with both MTA products being absent of calcium sulfate, a common setting retarder found in PC.

The highest correlation indicated by this study is between the setting time and the d90 reference point in particle size. This correlation was observed in both PC and MTA materials although the magnitude of the correlation in the MTA studies was smaller, perhaps because of the presence of accelerants or other modifiers in some of those formulations.

The largest particles play a significant role in setting because they typically react the slowest because they provide less surface area per unit volume, slowing the nucleation and growth of hydration products (20). In other words, the testing method for setting reflects the characteristics and proportions of the largest-sized particles that are consumed at a slower rate, not those of the smallest particles that are consumed in less time. Setting requires the formation of a percolated framework (scaffolding) of connected partially hydrated cement particles. Some of the smallest particles (eg, 1 $\mu$m or less in diameter) will dissolve quickly in the mix water and therefore will not be available to participate in the construction of this percolated particle network. This will not be the case for the larger particles that only partially hydrate, even after a curing duration of several days, and can therefore contribute to the connected structure that will produce a set cement paste.

Aside from using indentation tests, such as the Gillmore and Vicat needles, there are other ways to infer the setting during hydration of cement. This study features the use of isothermal calorimetry where it is shown that smaller particle sizes result in greater heat release over a period of 4 hours. In the case of PC, halving the d90 size almost doubled the heat release. Because the d90 of the different brands of MTAs range from 7.5–32.3 $\mu$m, it is possible that the heat release between 2 different samples could almost be several times different. Further research is required to determine if this could potentially affect pulpal repair and osteogenesis.

Many manufacturers alter the setting time of MTA by adjusting PSD. There are limits to this approach because smaller particles of PC may produce hydration products with greater porosity (because of increased water demand), an elevated risk of cracking, or greater shrinkage during setting (14). 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 (21). More research is required to determine if such practical advantages outweigh possible long-term structural concerns when the material sets too quickly.

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