Comparison of Compressive Strength of Five Hydraulic Cements

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Abstract
***Objectives*:** Maintaining pulp vitality is a main goal in restorative dentistry. Introduction of pulp capping agents paved the way to eliminate the shortcomings of these materials and obtain successful restorations. On the other hand, nanotechnology is an emerging field of science with increasing use in dental materials. This study sought to assess the effect of addition of nano-TiO_2, nano-SiO_2 and nano-Al_2O_3 on compressive strength of five hydraulic cements.
***Methods*:** In this in vitro, experimental study, three experimental formulations of nano hybrid MTA, MTA Angelus and MTA Angelus+ nano-oxide particles cements were placed in molds measuring 4±0.1mm in internal diameter and 6±0.1mm in height made of stainless steel (ISO9917-1). Ten samples were fabricated for each of the five groups of materials. Sound samples were stored at 37°C and 95±5% humidity and were subjected to compressive strength testing in a universal testing machine at a crosshead speed of 0.5mm/minute after 24 hours and one month. Two-way ANOVA, one-way ANOVA and independent samples t-test were used for comparison of compressive strength of groups at different time points.
***Results*:** The highest compressive strength belonged to MTA Angelus+ nanohydroxyapatite and nano-hybrid MTA C at 24 hours and 30 days, respectively. The lowest compressive strength belonged to nano-hybrid MTA B and MTA Angelus at 24 hours and 30 days, respectively ($P<0.05$).
***Conclusion*:** Addition of nanoparticles affected the compressive strength of cements. Compressive strength significantly increased over time in all groups.
***Key Words*:** Compressive Strength; Dental Cements; Mineral Trioxide Aggregate.
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Introduction

Maintaining pulp vitality is a main goal in restorative dentistry. Introduction of pulp capping agents paved the way to eliminate the shortcomings of these materials and achieve successful restorations. On the other hand, nanotechnology is an emerging field of science with increasing use in dental materials. Nanotechnology has been successfully used in food manufacturing and pharmaceutical industries, medicine, electronics and aerospace. Considering the four main functions of dental pulp namely regeneration, nutrition, protection and sensation (1), maintaining pulp vitality is a priority in both developing and mature teeth. A successful vital pulp treatment depends on optimal seal, quality of dentinal barrier, sterility of the exposure site, bleeding control, absence of inflammation, duration of exposure and many other factors (2). Vital primary or permanent teeth with complete or incomplete apices and mechanical, traumatic or carious pulp exposure may be suitable candidates for vital pulp therapy (3). Several methods have been suggested to improve the strength of dental cements. Improving the biological properties of cements has also been the topic of many investigations. Compressive strength of dental materials is important in functional loading of restorations. One novel idea for increasing the compressive strength and mechanical properties of dental materials is to add nanoparticles to cement matrix(4,5). Saghiri *et al*. (6) in 2013 compared the compressive strength of ProRoot MTA, nano-white MTA (Kamal Asgar Research Center, Iran), white MTA (Dentsply) and bioaggregate (Innovative Bioceramix) under different acidic conditions at the time of hydration. The results showed that the highest compressive strength belonged to nano-white MTA at a pH of 10.4 (133.19±11.14MPa). Reduction in pH caused a significant reduction in compressive strength of all samples (6).

Akbari *et al*. (7) showed that addition of nano-silica to MTA decreased the setting time by accelerating the process of hydration but had no significant effect on compressive strength. The present study aimed to assess the effect of addition of nano-TiO_2, nano-SiO_2 and nano-Al_2O_3 on compressive strength of three experimental formulations of MTA, MTA Angelus and MTA Angelus+nano-oxide particles.

Methods

This in vitro, experimental study was conducted on experimental formulations of nano-hybrid MTA (A, B and C), MTA Angelus and MTA Angelus plus nano-oxide particles. Compressive strength of MTA Angelus cement has been reported to be 15-50MPa at 24 hours (8-10). Experimental nano-hybrid groups with Portland cement base contained SiO_2, Al_2O_3 and TiO_2 nanoparticles, micro-silica particles and bismuth trioxide (opaquer). SiO_2, TiO_2 and Al_2O_3 nanoparticles were present in equal amounts (less than 5%) in the experimental groups to enhance mechanical properties and accelerate the setting time (11-14). Al_2O_3 nanoparticles were added to increase the compressive strength and modulus of elasticity (15). TiO_2 nanoparticles were added to confer antibacterial and photoelastic properties (16). Group B had the highest level of calcium hydroxide and group C contained 1% nano-hydroxyapatite. Type of Portland cement was type 5 in group A, 1-525 in group B and type 2 in group C. All these cements were fabricated in the laboratory of Parseh Dental Promotion Center (Tehran, Iran). MTA Angelus was considered as the control group for the purpose of comparison with the experimental groups (Table 1).

In this in vitro, experimental study, hydraulic cement powder was mixed with water in 3:1 ratio as recommended by the manufacturer. This was done until obtaining a sand-like consistency in commercial samples. In experimental samples, the ratio was 2.5:1 until reaching a putty consistency. The cements were transferred to stainless steel molds with an internal diameter of 4±0.1mm and height of 6±0.1mm (ISO9917-1). Sample size was calculated to be 10 samples in each group according to previous studies (17,18).

Table 1- Composition of hydraulic cements

hydraulic cements	Composition
A	Type 5 Portland cement, nano-SiO_2, nano-Al_2O_3, nano-TiO_2, micro-silica particles, bismuth trioxide, calcium hydroxide

B	1-525 Portland cement, nano-SiO_2, nano-Al_2O_3, nano-TiO_2, micro-silica particles, bismuth trioxide, calcium hydroxide†
C	Type 2 Portland cement containing nano-SiO_2, nano-Al_2O_3, nano-TiO_2, micro-silica particles, bismuth trioxide and 1% nano-hydroxyapatite
MTA Angelus	SiO2, K20, Al2O3, Na2O, Fe2O3, SO3, CaO, Bi2O3, MgO. Insoluble residues (crystalline silica, calcium oxide and potassium sulfate and sodium)
MTA Angelus+ nano-oxide particles	SiO2, K20, Al2O3, Na2O, Fe2O3, SO3, CaO, Bi2O3, MgO. Insoluble residues (crystalline silica, calcium oxide and potassium sulfate and sodium) + nano-TiO_2, nano-SiO_2 and nano-Al_2O_3

†: this group consist of calcium hydroxide more than others.

*: Due to secrecy of factory use are not permitted to mention the percent's & the differences

Mixing was done by the same operator and manually in all groups. A digital scale was used to measure the weight of powder and liquid to be mixed. For example, when two drops of liquid had to be mixed with the powder, the dropper was held vertically and one intact, air-free drop was dropped. It was weighed by the digital scale. After primary setting, the samples were removed from the mold. Those with cracks or surface irregularities were excluded. Sound samples were stored at 37°C and 95±5% humidity and were subjected to compressive strength testing in a universal testing machine (Zwick/ Roell Z020, Zwick GmbH & Co.KG, Ulm, Germany) at a crosshead speed of 0.5mm/minute after 24 hours (19,20) and one month (19) according to ISO9917-1 standard (21,22). Two-way ANOVA was used to compare the compressive strength of the groups at different time points. One-way ANOVA was applied to assess the interaction effect of type of material and time on compressive strength. Tukey's HSD test was used for pairwise comparisons of the groups. Comparison of the two time points in each group was done using independent samples t-test. All statistical analyses were performed using SPSS version 21 (SPSS Inc., IL, USA).

Results

Table 2 shows the mean and standard deviation of compressive strength in the groups and pairwise comparisons. The results showed that compressive strength was affected by the type of cement and time (both $P<0.001$). At 24 hours and 30 days, MTA Angelus+ nano-oxides and MTA cement C showed the highest compressive strength, respectively. The lowest compressive strength at 24 hours and 30 days belonged to MTA cement B and MTA Angelus, respectively. At 24 hours, group B had a statistically significant difference in compressive strength with other groups and the lowest compressive strength belonged to this group (18.86±0.68MPa). Also, significant ordinal interaction effect of the two factors was noted on compressive strength ($P<0.0001$).

Table 2- The mean compressive strength in the 3 groups at 24 hours and 30 days and pairwise comparisons

Time/Group	A	B	C	MTA Angelus+ nano-oxides	MTA Angelus
24 hours	35.9±1.71b	18.86±0.21a	33.74±0.55b	44.32±3.62c	36.23±1.97bc
30 days	82078±1034a	87056±1.26a	102.44±2.31b	85.63±1.87a	81.13±1.53a

Similar superscripted letters indicate absence of a significant difference between the groups.

Discussion

Several methods have been suggested to enhance the compressive strength of dental cements. The main focus of dental material research is currently on improving the biological properties of cements. Compressive strength of dental materials is important during functional loading of restorations. Studies on MTA have reported indirect loading of MTA (23,24) and it should always be covered with an intermediate material such as glass ionomer cement.

In the current study, compressive strength significantly increased from 24 hours to 30 days. According to Islam *et al*,(21) in 2006 this is due to continuous setting of material, which increases the strength and stability (21). The 24-hour compressive strength of MTA Angelus in the current study was similar to that reported by Silva *et al*,(10) in 2010 with the difference that they. measured the compressive strength at 21 hours. The 24-hour and 30-day compressive strength of MTA Angelus in the current study was not similar to the values reported by Tanomaru-Filho *et al*,(19) in 2012. In their study, the 24-hour and 21-day compressive strength values were half the values in our study, which may be due to differences in the methodology of the two studies. In their study, samples fabricated of each cement had 12mm height and 6mm diameter while in the current study, each sample had 6mm height and 4mm diameter. Also, the difference in the results may be due to the technique of mixing. In the current study, one operator mixed all samples manually with the naked eye. Discrepancies in homogeneity were possible. Also, there was a possibility that fracture line passes through areas with lower strength due to unequal homogeneity. However, if present, these discrepancies would be too small to affect the results. Also, both liquid and powder were weighed in this study before mixing since use of spoon provided by the manufacturer could have resulted in variable amounts of powder in terms of weight depending on the level of fullness and compression of particles (25). For the liquid, the size of drops dropped by different individuals or even the same individual may vary depending on the pressure applied and angle of holding the dropper (26,27). We tried our best to eliminate the effect of confounding factors affecting compressive strength such as environmental temperature, incorrect powder/liquid ratio and pressure applied to mold as much as possible as described above.

One novel technique to increase the strength and improve the mechanical properties of materials is to add nanoparticles to the cement matrix. Akbari *et al.* (7) added 8 and 10% nano-silica particles to MTA and concluded that despite a reduction in sintering time, no change occurred in flexural or compressive strength, which was somehow in line with our results. Based on the current results, at 24 hours, groups A and C did not have a significant difference with MTA Angelus in terms of compressive strength, which may be due to strong reactions between cement matrix particles and nanoparticles. Group B showed the lowest compressive strength with significant differences with other groups, which was probably due to agglomeration of nanoparticles in high concentrations, serving as weak points. Also, replacement of type II cement with 1-525 cement and presence of the highest percentage of calcium hydroxide in this group compared to other groups may explain the reduction in compressive strength and increased brittleness of the material. Addition of nanoparticles to MTA Angelus caused no significant increase in 24-hour and 30-day compressive strength values compared to the control group. Although the values were slightly, but not significantly, higher than that of the control group. Fast-set MTA Angelus was used in this study. If nanoparticles have been used by the manufacturer to decrease its setting time, there would be a risk of agglomeration of particles and creation of weak points in the cement, which explains no increase in compressive strength in our study. In group C, lower amount of calcium hydroxide was used and hydroxyapatite nanoparticles replaced hydroxyapatite powder.

Due to higher crystallization degree and greater colloidal stability, hydroxyapatite nanoparticles can improve the mechanical properties and handling of cement (28). Thus, 30-day compressive strength in this group was higher than that of other groups. Based on the results, despite reduction in setting time of experimental cements, due to no change in their compressive strength, use of glass ionomer cement as base material is still preferred to hydraulic cements. Researchers must continue to work on finding formulations of MTA with high enough compressive strength to resist functional forces directly and immediately after application.

Conclusion

Over time, compressive strength significantly increased in all groups. Compressive strength in experimental group C was significantly higher than that of other groups at 30 days due to the presence of nano-hydroxyapatite. Addition of nano-oxide particles to MTA Angelus did not significantly increase its compressive strength.

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