

Evaluating the Physical Properties of Novel Zinc Phosphate and Zinc Polycarboxylate Cements Containing Zinc Oxide Nanoparticles

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Abstract

Background: Zinc oxide (ZnO) that is a main component of Zinc Polycarboxylate and Zinc Phosphate conventional cements has been incorporated into many dental materials for mechanical enhancement. Moreover, by decreasing the particle size of ZnO down to nano-scale, its beneficial effects would tremendously increase because the nanoparticles have considerably higher surface to volume ratio compared to micro-particles.

Objectives: The aim of this study was to assess mechanical, physical, and chemical properties of Zinc Polycarboxylate and Zinc Phosphate cements containing Zinc oxide nanoparticles.

Methods: Three powder formulations were prepared for either of the cements based on the nanoparticles content (0 wt%, 10 wt% before, and 10 wt% after sintering the powder). The prepared groups were compared with each other in terms of their compressive strength, setting time, film thickness, and acid erosion resistance using one way ANOVA and Tukey HSD statistical tests ($\alpha = 0.05$).

Results: Incorporating zinc oxide nanoparticles did not significantly change neither the film thickness nor the acid erosion resistance of the cements ($P > 0.05$). Nevertheless, the setting time of zinc phosphate significantly decreased by adding nanoparticles ($P < 0.05$) while there was no statistical difference in zinc polycarboxylate groups ($P > 0.05$). On the other hand, although incorporating nanoparticles significantly reduced the compressive strength of zinc phosphate ($P < 0.05$), it was noticeably improved in zinc polycarboxylate groups ($P < 0.05$).

Conclusions: By incorporating 10 wt% of nano zinc oxide into zinc phosphate and zinc polycarboxylate cements, their compressive strength are more affected rather than their setting time, film thickness, and acid erosion resistance.

Keywords: Nanoparticles, Zinc Oxide, Dental Cement, Film Thickness, Setting Time, Acid Erosion, Compressive Strength

1. Background

Zinc oxide (ZnO) is an inorganic particle, which has been incorporated in many dental materials due to its unique properties (1-3). ZnO, which is a tooth colored metal oxide powder, can reinforce many dental materials mechanically and it can impart the antibacterial effect into the dental agents, as well (1-3).

Among ZnO-containing dental materials, Zinc Polycarboxylate and Zinc Phosphate cements have been used for many years in dentistry (4). However, by introduction of resin cements, the application of these conventional cements has considerably decreased due to superior mechanical behavior of resin cements. Nevertheless, Zinc phosphate and Zinc polycarboxylate are still more preferred by some dentists because of their simple clinical

procedures (4). Moreover, they are less technique sensitive and their efficacy is less affected by oral conditions (4). Additionally, adhesive cements, which are multistep, can result in complicated clinical procedure especially in the posterior region (5, 6). Therefore, enhancing the properties of Zinc phosphate and Zinc polycarboxylate cements would be desirable because they are more user-friendly in dental clinics.

Although promotion of these cements has been frequently investigated (4, 7-9), additional studies are always recommended.

Meanwhile, in recent years, nano technology has introduced different nanoparticles that could be incorporated in dental materials. Indeed, nanoparticles can lead to improvement of mechanical properties of many dental materials such as establishing anti-bacterial effects, radiopacity,

etc. in dental agents (10, 11).

It has been documented that reducing the particle size down to nano scale is quite beneficial in dental materials because the smaller nanoparticles could be better distributed in most dental material matrices and the mechanical strength would improve dramatically (2). Besides, as the size of the particle decreases, the film thickness would decrease too that is a critical property in dental cements (9, 12). Therefore, integrating nanoparticles into dental cements can exert favorable effects on their clinical performance.

On the other hand, since the main disadvantage of conventional dental cements such as Zinc Polycarboxylate and Zinc Phosphate is their low mechanical strength (4, 7-9), nanoparticles can solve their shortcomings.

However, a few previous studies have incorporated Zinc oxide nanoparticles into dental cement and they have reported controversial results about mechanical improvement (2, 3). Accordingly, some studies have claimed that incorporating nano-ZnO into dental materials can lead to better compressive strength (2) while in other investigations (3) the dental cements containing nano-ZnO showed lower mechanical strength (3). Therefore, supplementary research on incorporation of nano-ZnO into dental materials has been suggested in many previous documents (2, 3).

2. Objectives

The present study was conducted to assess the mechanical, physical, and chemical properties of Zinc Polycarboxylate and Zinc Phosphate cements containing zinc oxide nano-particles.

3. Methods

The current investigation was an experimental in-vitro study.

3.1. Sample Preparation

As presented in Table 1, three formulations of each cement powder were prepared. After mixing the powder, it was sintered at 1200°C for 4 hours. In order to achieve 0.3 μm particles, they were ball milled (Sanat Ceram, Iran) for 5 hours, and then sieved through a 0.3 μm diameter sieve.

The prepared powder was mixed with their liquid component according to ISO 9917 standard.

All the following tests were repeated 5 times for each group.

3.2. Compressive Strength

According to ISO 4049, the materials were inserted into a 4 mm diameter \times 6 mm height cylindrical stainless-steel mold placed on a glass slide and covered with another glass slide. After one hour, the specimens were removed from the mold and stored in an incubator (Dorsa, Iran) for 24 hours at 37°C and 30% humidity prior to the test. At least five specimens were tested for each formulation. The compressive strength was then determined with the universal testing machine (Sanatam, Iran) at a crosshead speed of 1 mm min⁻¹ until failure. The specimens were placed with their flat ends between the plates of the testing machine so that the progressively increasing compressive load was applied along the long axis of the specimens (13).

3.3. Film Thickness

The film thickness was tested according to the testing method set in ISO 3107 (2004). About 0.02 - 0.1 mL of the prepared samples were placed between two slabs and pressed with a 150 N vertical force for 10 minutes. The cumulative thickness of the two slabs was measured before and after the pressure and the difference was considered as cement film thickness.

3.4. Setting Time

The setting time test was conducted based on ISO 6876 (2001). In order to estimate the setting time, the cements were transferred to 5 mm thick molds (five samples for each group) after mixing. A time interval of 90 seconds after mixing was considered for each sample. Thereafter, a 400 g needle with a flat ending and 1 mm diameter was applied vertically on the surface of each sample every 30 seconds by a Rheometry device (Dorsa, Iran). This process was stopped whenever the indenter needle could not penetrate more than 4.9 mm into the samples. The priority recorded time at the step before ending was considered as setting time.

3.5. Acid Erosion

The erosion resistance test was performed according to the method described by Billington et al. (14). Eight cylindrical samples (2 mm height \times 5 mm diameter) were prepared for each group. After mixing, the prepared samples were compressed between the two clamps for one hour and stored in 30% humidity at 37°C for 23 hours. Thereafter, an acid erosion testing machine (Dorsa, Iran) was used to perform the test. The amount of weight loss per hour was described as erosion rate while the specimens were exposed to 0.02 M lactic acid at 37°C for periods up to 24 hours.

Table 1. Powder Content in Different Tested Formulations (Weight Percentage (wt%)^a)

Variables	Zn.Ph 1	Zn.Ph 2	Zn.Ph 3	Zn.PCa 1	Zn.PCa 2	Zn.PCa 3
ZnO (Merck, USA)	90	80	80	90	80	80
Nano-ZnO (WorleeChem, India)	0	10 (BS)	10 (AS)	0	10 (BS)	10 (AS)
SiO ₂ (Merck, USA)	1.4	1.4	1.4	1.4	1.4	1.4
BaO (Merck, USA)	0.1	0.1	0.1	0.1	0.1	0.1
MgO (Merck, USA)	8.5	8.5	8.5	8.5	8.5	8.5
Liquid	A	A	A	B	B	B

Abbreviations: AS, After Sintering; BS, Before Sintering; ; Zn.PCa, Zinc Polycarboxylate; Zn.Ph, Zinc Phosphate.

^aA, 65% H₃PO₄ solution containing Al and Zn; B, 35% poly (acrylic acid) solution (molecular weight 35000).

3.6. Statistical Analysis

For statistical analysis of the data, after exploring the normal distribution of the data using Kolmogorov-Smirnov test, the One-way ANOVA and Tukey HSD Post HOC tests were performed while the level of the significance was set at $\alpha = 0.05$.

4. Results

The comparative data related to three formulations of zinc phosphate and zinc polycarboxylate are demonstrated in [Tables 2 and 3](#), respectively.

4.1. Compressive Strength

As presented in [Tables 2 and 3](#), the compressive strength of all groups was above the least amount that is accepted according to ISO 9917 (2003). Meanwhile, in zinc phosphate groups, One-Way ANOVA discovered a statistically significant difference between three groups ($df = 2$, $F = 6.49$, $P = 0.01$). Besides, Tukey HSD Post Hoc comparison revealed that formulation No.3 had significantly lower compressive strength compared to formulations No.2 ($P = 0.01$) and No.1 ($P = 0.02$). Therefore, incorporating zinc oxide nanoparticles into Zinc phosphate powder reduced the compressive strength of the cement especially when the nanoparticles were added after sintering the powder.

On the other hand, zinc polycarboxylate subgroups were significantly different from each other using One-Way ANOVA ($df = 2$, $F = 8.73$, $P = 0.005$). Subsequently, Tukey HSD Post Hoc test showed that formulation No.3 had significantly higher compressive strength than formulations No.1 ($P = 0.004$) and No.2 ($P = 0.04$). Whilst there was no statistically significant difference between No.1 and No.2 groups ($P = 0.38$). Hence, addition of zinc oxide nanoparticles to the zinc polycarboxylate after powder sintering enhanced the compressive strength of the cement. In contrast, before sintering the powder, the 10 wt% of nano-

ZnO did not have any significant effect on the compressive strength of Zinc polycarboxylate cement.

4.2. Film Thickness

[Tables 2 and 3](#) illustrate that all the samples had fill thickness lower than 25 μm that is accepted by ISO 9917 (2003). One-way ANOVA revealed that there was no statistically significant difference between the tested groups in either zinc phosphate or zinc polycarboxylate cements ($df = 2$ and 2, $F = 0.71$ and 1.14, $P = 0.50$ and 0.35 for zinc phosphate and zinc polycarboxylate, respectively). It means that incorporating zinc oxide nanoparticles in the study cements would not have any adverse effect on their film thickness.

4.3. Setting Time

Since the setting time of zinc phosphate formulation No.3 was less than 2.5 minutes, it was not standard according to ISO 9917 (2003), while the other zinc phosphate groups and all the zinc polycarboxylate formulations were in the standard range (2.5 - 6 minutes). However, as can be seen in [Table 2](#), the setting time of three Zinc phosphate subgroups were distinguishable from each other using One-way ANOVA ($df = 2$, $F = 18.35$, $P = 0.000$). Precisely, the Tukey HSD Post Hoc comparison revealed that incorporating zinc oxide nanoparticles into the cement powder significantly decreased zinc phosphate setting time compared to the unmodified group ($P = 0.03$ and 0.00 for formulations No.2 and No.3, respectively). Moreover, No.2 group was noticeably lower than No.3 ($P = 0.02$). Conversely, one-way ANOVA analysis showed that the zinc polycarboxylate groups did not have any significant difference with each other ($df = 2$, $F = 1.21$, $P = 0.33$).

4.3.1. Acid Erosion

As demonstrated in [Tables 1 and 2](#), all the examined groups were acid-eroded less than 2 mm/min which were

Table 2. Mechanical (Compressive Strength), Physical (Film Thickness) and Chemical (Setting time and Acid Erosion) Properties Related to the Examined Three Formulations of Zinc Phosphate Cement (Formulations No.1 to No.3 Correspond to Zinc Phosphate Cements without Nano-ZnO, Containing 10 wt% before and 10 wt% After Sintering the Powder, Respectively)^a

Variables	C.S, MPa	F.T, μm	S.T, min	A.E, mm/min
Zn.Ph 1	75.88 \pm 8.98 ^a	18.40 \pm 2.96 ^a	3.70 \pm 0.57 ^a	1.78 \pm 0.27 ^a
Zn.Ph 2	77.40 \pm 8.86 ^a	18.00 \pm 3.74 ^a	2.66 \pm 0.61 ^b	1.79 \pm 0.32 ^a
Zn.Ph 3	56.58 \pm 12.32 ^b	20.40 \pm 3.43 ^a	1.56 \pm 0.48 ^b	1.69 \pm 0.14 ^a

Abbreviations: A.E, Acid Erosion; C.S, Compressive Strength; F.T, Film Thickness; S.T, Setting Time; Zn.Ph, Zinc Phosphate.

^aValues presented as mean \pm S.D, Same letter (a and b) did not have any significant difference with each other ($P \geq 0.05$).

Table 3. Mechanical (Compressive Strength), Physical (Film Thickness) and Chemical (Setting Time and Acid Erosion) Properties Related to the Examined Three Formulations of Zinc Polycarboxylate Cement (Formulations No.1 to No.3 Correspond to Zinc Polycarboxylate Cements without nano-ZnO, Containing 10 wt% Before and 10 wt% After Sintering the Powder, Respectively)^a

Variables	C.S, MPa	F.T, μm	S.T, min	A.E, mm/min
Zn.PCa 1	76.68 \pm 7.12 ^a	18.60 \pm 3.13 ^a	3.60 \pm 0.41 ^a	1.75 \pm 0.32 ^a
Zn.PCa 2	87.96 \pm 20.56 ^a	16.80 \pm 2.16 ^a	3.96 \pm 0.42 ^a	1.75 \pm 0.32 ^a
Zn.PCa 3	110.25 \pm 5.21 ^b	19 \pm 8 ^a	3.60 \pm 0.41 ^a	1.60 \pm 0.16 ^a

Abbreviations: A.E, Acid Erosion; C.S, Compressive Strength; F.T, Film Thickness; S.T, Setting Time; Zn.PCa, Zinc polycarboxylate.

^aValues presented as mean \pm S.D, Same letter (a and b) did not have any significant difference with each other ($P \geq 0.05$).

categorized as standard based on ISO 9917 (2003). Moreover, no statistically significant difference was recorded by one-way ANOVA between the three formulations of either zinc phosphate or zinc polycarboxylate groups (df = 2 and 2, F = 0.22 and 0.45, P = 0.79 and 0.64 for zinc phosphate and zinc polycarboxylate, respectively). Therefore, it could be claimed that incorporating 10 wt% zinc oxide nanoparticles into zinc phosphate and zinc polycarboxylate cements did not have a deleterious effect on their acid erosion property.

5. Discussion

Our investigation revealed that admixing 10 wt% of nano-ZnO into Zinc polycarboxylate or Zinc phosphate dental cements did not have any significant adverse effect on their film thickness, setting time, or acid erosion property.

Meanwhile, the results of the current survey showed that incorporation of 10 wt% zinc oxide nanoparticles into zinc polycarboxylate cement would increase its compressive strength. This finding is in accordance with those of previous research that claimed that addition of some amount of zinc oxide nanoparticles would enhance the mechanical properties of various dental materials (15-17). Indeed, the mechanical improvement by nano-ZnO is related to their very small size that leads to a good distribution of these nanoparticles between the larger particles, producing a higher density of filler in a specified area (9).

This phenomenon has been previously described about micro hybrid resin composites compared to macro filled ones (9).

On the other hand, in the present study, incorporation of 10 wt% of nano-ZnO into zinc phosphate cement decreased its compressive strength. This is in agreement with the findings of Tabari et al. who added nano-ZnO to Zinc oxide eugenol and recorded reduced compressive strength (3). The reduction of compressive strength could be explained by the fact that this weight percentage of nanoparticles is very high for zinc phosphate cement because at very high weight percentages, the nanoparticles produce flaws and irregularities in the structure. Many published data also indicate that the addition of various nanoparticles to different dental materials for mechanical improvement has a threshold beyond which no further mechanical enhancement would be achieved (15). Indeed, at low mass fractions, nanoparticles would have a relatively good dispersion while at higher concentrations they began to form bundles and agglomerations that leads to defects and flaws in the mass. These structural defects deteriorate mechanical properties of the dental materials (15, 16).

Another important clinical aspect of dental cements includes their film thickness. Since marginal fit is one of the most important determinants for the longevity of indirect restorations (18-21), the cement film thickness is clinically critical. It has been documented that increasing the cement film thickness has a noticeable negative effect on the prosthesis retention (8). Accordingly, our results re-

vealed that incorporating zinc oxide nanoparticles (in the mentioned weight fractions) into zinc phosphate or zinc polycarboxylate did not have any significant effect on their film thickness. This is an interesting finding because zinc phosphate cement has the best sealing capacity even compared to glass ionomer, and it is categorized as the 'reference' or 'gold standard'(22, 23).

Therefore, addition of nano-ZnO did not significantly change this unique property of zinc phosphate.

Furthermore, since another major characteristic of a proper dental cement is their setting time that determines the ease of clinical use (9), evaluating the setting time is always important in dental cement investigations. On this ground, the result of the current study represented no significant difference in setting time between various formulations of zinc polycarboxylate. Since this cement has an acceptable clinical setting time, it can be concluded that incorporation of zinc oxide nanoparticles did not deleteriously change this favorable property of the mentioned cement. Nevertheless, this is not inconsistency with previous research. Tabari et al. reported faster setting time by smaller particles (3). They claimed that the smaller particles have higher surface to volume ratio and their reaction occur much faster (3). However, Tbari et al. used all the powders of their survey as nanoparticles while we used up to 10 wt% percentage. Therefore, it could be justified that incorporation of nanoparticles has a threshold beyond which the clinical efficacy of dental cement would be deleteriously affected. Meanwhile, our finding confirms this hypothesis because as is obvious in Tables 2 and 3, increasing the nanoparticles fraction leads to the reduced setting time although the statistical difference was not significant.

On the other hand, the setting time of zinc phosphate cement significantly decreased by addition of zinc oxide nanoparticles. Since it is in agreement with previous research (3), our hypothesis could be again confirmed that the percentage of 10 wt% nanoparticles for zinc phosphate is not suitable and lower amounts have to be examined.

5.1. Conclusion

By incorporating 10 wt% of nano zinc oxide into zinc phosphate and zinc polycarboxylate cements, their compressive strength is more affected rather than their setting time, film thickness, and acid erosion resistance.

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Footnotes

Authors' Contribution: Study concept and design, Mohammad Bagher Rezvani; acquisition of data, Mohammad Ali Hemmati, Mostafa Fatemi, Farhood Najafi; analysis and interpretation of data, Mostafa Fatemi, Farhood Najafi; drafting of the manuscript: Faeze Hamze; critical revision of the manuscript for important intellectual content, Faeze Hamze; statistical analysis, Faeze Hamze; administrative, technical, and material support, Mohammad Bagher Rezvani; study supervision, Mohammad Ali Hemmati.

Conflict of Interest: The authors confirm that they do not have any conflict of interest in this study.

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