

Chemical, Thermal and Physical Properties of Al₂O₃ Nanoparticles Addition Zinc Polycarboxylate Cements

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ABSTRACT

In this study microhardness, wettability, chemical and thermal properties of 3 wt% and 5 wt% Al₂O₃ nanoparticles addition zinc polycarboxylate cements were investigated.

Materials and Methods: 3 wt% and 5 wt% Al₂O₃ nanoparticles were added to zinc polycarboxylate cement. Groups of specimen: Group 1. Zinc polycarboxylate cement without nanoparticle (Control group) Group 2. 3 wt% Al₂O₃ nanoparticles addition zinc polycarboxylate cement Group 3. 5 wt% Al₂O₃ nanoparticles addition zinc polycarboxylate cement. Microhardness, wettability, chemical and thermal properties of specimens were examined.

Results: As the % weight ratio of Al₂O₃ nanoparticles increased microhardness value increased. As the % weight ratio of Al₂O₃ nanoparticles increased the contact angle increased. The interaction between zinc polycarboxylate cement and nanoparticles resulted in the changes of zinc polycarboxylate cement peaks. Nanoparticles displayed catalytic effect during solidification of zinc polycarboxylate cement so groups with nanoparticles harden than control group earlier.

Conclusion: The highest microhardness value occurred in cement with 5 wt% Al₂O₃ nanoparticles. Addition of Al₂O₃ nanoparticles increased the contact angle of the specimens. FTIR analysis showed the changes of zinc polycarboxylate cement peaks. Nanoparticles displayed catalytic effect during solidification of zinc polycarboxylate cement.

Keywords: Zinc polycarboxylate cement, nanoparticle, microhardness, wettability, FTIR.

INTRODUCTION

A number of commercially available dental cements differ in chemical composition and therefore possess significantly different physical, mechanical and biological properties^[1]. The zinc polycarboxylate cement is formed from heat-treated zinc oxide and aqueous polyacrylic acid^[2]. Zinc polycarboxylate cements (ZPC) are preferred for luting fixed restorations because they reveal good pulp compatibility. These cements did not cause initial pain that takes place after luting when cast restorations were inserted with polycarboxylate cements; because they did not cause a severe reaction. However, these cements shrink more extensively than other materials such as zinc phosphate cements. This shrinkage, due to cross-linking of polymer chains with zinc atoms, possibly affects the integrity of bulk restorations^[3].

Nano-sized materials show unique properties depending on their size. Especially, metal and metal oxide nanoparticles have been widely investigated because of their potential for many applications^[4]. Several studies were performed to investigate the properties of different nanoparticles addition dental cements. 1, 2, 5, 10, 15 and 25% (w/w) (ytterbium fluoride) YbF₃ and (barium sulphate) BaSO₄ nanoparticles were added to commercial glass-ionomer cement. Surface hardness increased at low concentrations of nanoparticles but it significantly decreased at nanoparticle concentrations of 10% or above. None of cements was significantly harder than control^[5].

SiO₂, TiO₂ and Al₂O₃ nanoparticles were added to dental glass-ionomer cement, Fuji II. Surface hardness increased in most of modified cements compared to dental glass-ionomer cement, Fuji II^[6]. Surface microhardness showed a small insignificant increase in 3% (w/w) TiO₂ nanoparticles addition glass ionomer compared to control group. It decreased significantly in 5% and 7% (w/w) TiO₂ nanoparticles addition glass-ionomers^[7]. 0.25, 0.5 and 1% wt/wt metallic silver nanoparticles had no significant effect on the glass transition temperature of acrylic bone cements. The collected FTIR spectra analysis showed no differences in the position or magnitude of intensity peaks between different cements, indicating that there was no formation of new chemical groups^[8]. In this study microhardness, wettability, chemical and thermal properties of zinc polycarboxylate cements with 3 wt% and 5 wt% Al₂O₃ nanoparticles were investigated.

MATERIALS AND METHODS

Zinc polycarboxylate cement was used as cement material and Al_2O_3 nanoparticles were used as reinforce material for this study. 3 wt% and 5 wt% Al_2O_3 nanoparticles were added to zinc polycarboxylate cement. Specimens were divided to three groups. (n=10) Group 1. Zinc polycarboxylate cement without nanoparticle (Control group) Group 2. 3 wt% Al_2O_3 nanoparticles addition zinc polycarboxylate cement Group 3. 5 wt% Al_2O_3 nanoparticles addition zinc polycarboxylate cement. Specimen preparation process was shown in Figure 1.

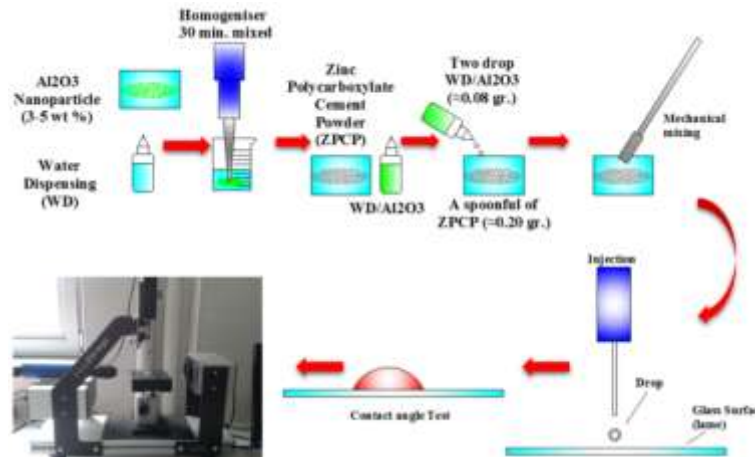


Figure 1: Specimen preparation process.

The materials and manufactures used in study were given in Table1.

Table (1): The materials and manufacturers.

Material	Manufacturer
Zinc Polycarboxylate cement (Poly-F Plus)	Dentsply, Konstanz, Germany
Al_2O_3 nano powder (99.5% pure, powder size 40-50 nm)	MKNANO, Canada

Microhardness test

Vickers microhardness values of groups were measured by using Duroline M microhardness device at 10 g of loading.

Wettability test

The contact angles of groups were measured using Easey Drop contact angle measurement device by dropping 5 times from every parameter drop on clean glass.

Thermal analysis

Differential Scanning Calorimetry (DSC) (METTLER TOLEDO DSC 1STAR System) at a heating rate of 10 °C/min from 30° to 600 °C under a nitrogen flow of 30 ml/min was used to examine thermal effect of Al_2O_3 nanoparticles addition zinc polycarboxylate cement.

Chemical analysis

Fourier transform infrared (FT-IR) spectrum measurements were performed with BRUKER-Vertex 70 spectrophotometer. All analyses were made between 400 and 4000 cm^{-1} , sensitively of 2 cm^{-1} and at room temperature.

Statistical analysis

Statistical analysis was performed with Kolmogorov-Smirnov test of normal distribution and one-way ANOVA followed by Tukey's honestly significant difference (HSD) test with a general linear model procedure in SPSS17.0 (SPSS Inc., Chicago, USA). One-way ANOVA followed by Tukey's HSD test was used with in each group to compare effectiveness of reinforcement with different concentrations. A significance level of 0.05 was used for statistical tests.

RESULT AND DISCUSSIONS

Microhardness test

According to microhardness test results significant increase in microhardness value of cement occurred by adding 5 wt% Al_2O_3 nanoparticles ($p < 0.05$). No significant difference was found between control group and cement with 3 wt% Al_2O_3 nanoparticles ($p > 0.05$) (Table 2, Figure 2).

Table (2): Results of microhardness test.

Groups	Mean microhardness	Standard deviation
Group 1 (without nanoparticle)	32,95	3,93
Group 2 (3 wt% Al ₂ O ₃ nanoparticles)	38,73	2,80
Group 3 (5 wt% Al ₂ O ₃ nanoparticles)	46,40	4,87

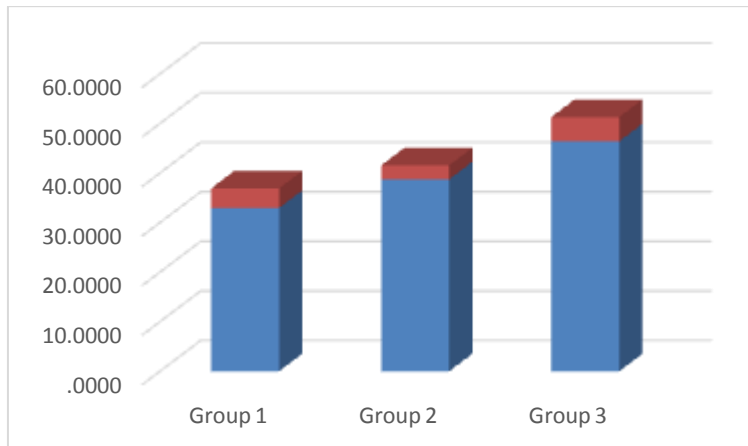


Figure 2: Results of microhardness test.

Wettability test

In this study the comparison of contact angles was done between the control group and the specimens containing different concentrations of Al₂O₃ nanoparticles. The contact angles of three group specimens calculated and deviation values were determined with statistical analysis. These values were shown in Table 3 and Figure 3.

Table (3): Mean contact angles and deviation values of groups.

Groups	Mean contact angle	Standard deviation
Group 1 (without nanoparticle)	49,74	1,73
Group 2 (3 wt% Al ₂ O ₃ nanoparticles)	77,3	4,45
Group 3 (5 wt% Al ₂ O ₃ nanoparticles)	85,5	5,3

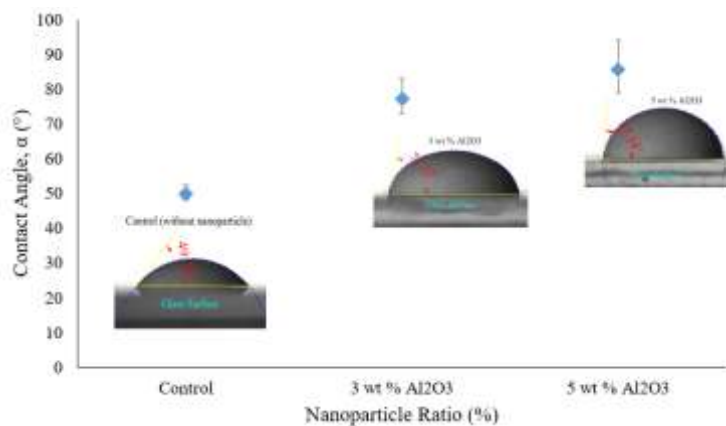


Figure 3: Contact angles of groups.

One-way analysis of variance showed a significant difference between mean values of contact angle ($p < 0.05$). Statistical analysis using the posthoc Tukey HSD significant differences test revealed that Al₂O₃ nanoparticles addition increased the contact angle of the specimens. Highest mean contact angle was observed in Group 3 (cement with 5 wt% Al₂O₃ nanoparticles), while the lowest was seen in Group 1 (cement without nanoparticle) (Fig 3).

Thermal analysis

DSC analysis was shown in Figure 4. It was observed that control group hold more moisture than the other groups. Nanoparticles displayed catalytic effect during solidification of zinc polycarboxylate cement so groups with nanoparticles harden than control group earlier.

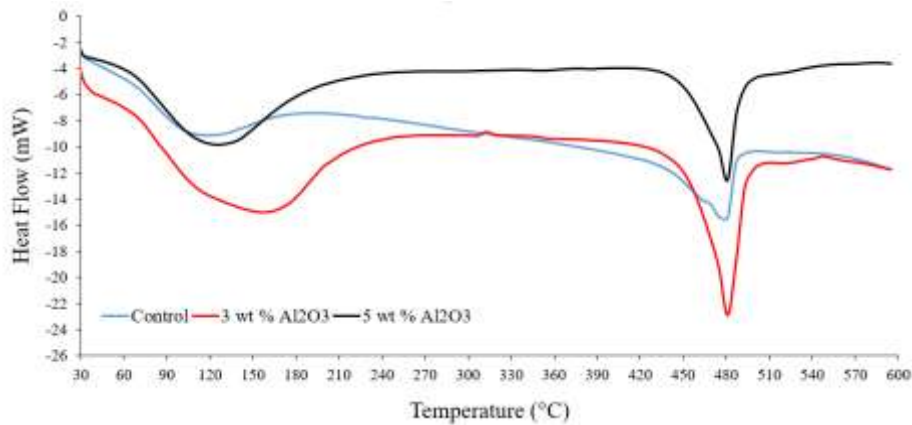


Figure 4: DSC graphics of groups.

Chemical analysis

The characteristic peaks of FT-IR spectrums for control group and groups with nanoparticles were shown in Figure 5.

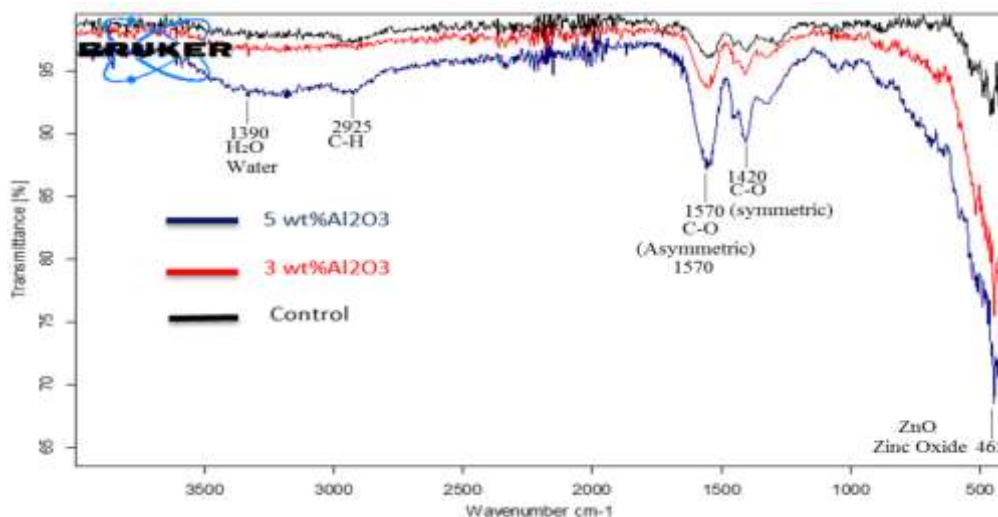


Figure 5: FTIR of groups.

After solidification the changes of absorption bands were seen easily. Approximately at 1570 and 1420 cm^{-1} peak intensity increased as % weight ratio of nanoparticles increased. The interaction between zinc polycarboxylate cement and nanoparticles resulted in the changes of zinc polycarboxylate cement peaks. Prentice et al. 2006 investigated the effects of 1, 2, 5, 10, 15 and 25% (w/w) (ytterbium fluoride) YbF_3 and (barium sulphate) BaSO_4 nanoparticles on reactivity and strength of commercial glass-ionomer cement. Surface hardness increased at low concentrations of nanoparticles but it significantly decreased at nanoparticle concentrations of 10% or above. None of cements was significantly harder than control.

Zhao and Xie 2009 investigated the effect of nanoparticles on wear resistance and surface hardness of dental glass-ionomer cement, Fuji II. SiO_2 , TiO_2 and Al_2O_3 nanoparticles were used. Surface hardness increased in most of modified cements compared to dental glass-ionomer cement, Fuji II. Slane et al. 2015 added 0.25, 0.5 and 1% wt/wt metallic silver nanoparticles in to acrylic bone cement. Mechanical, material and antimicrobial properties of acrylic bone cement impregnated with silver nanoparticles were examined. Silver nanoparticles had no significant effect on the glass transition temperature of the cements. The collected FTIR spectra analysis showed no differences in the position or magnitude of intensity peaks between different cements, indicating that there was no formation of new chemical groups.

Slane et al. 2014 incorporated 0.1 and 0.2 wt/wt mesoporous silica nanoparticles (one plain variety and two modified with functional groups) in to commercially acrylic bone cement. Material and mechanical properties were investigated. ATR-FTIR spectra analysis showed no differences in the position of intensity peaks between different cements, indicating that there was no formation of new chemical groups. Brajkovic et al. 2014 investigated surface free energy, contact angle, average roughness and fractal dimension of zinc-based, glass-ionomers, resin modified glass ionomer and resin cements. Zinc phosphate cements showed significantly total surface free energy and significantly lower dispersive component of surface free energy compared to other groups.

CONCLUSION

The aim of this study was to investigate microhardness, wettability, chemical and thermal properties of zinc polycarboxylate cements with Al₂O₃ nanoparticles. It was found following results from experimental studies.

1. As the % weight ratio of nanoparticles increased microhardness value increased.
2. The addition of Al₂O₃ nanoparticles increased the contact angle of the specimens.
3. Nanoparticles displayed catalytic effect during solidification of zinc polycarboxylate cement so groups with nanoparticles harden than control group earlier.
4. The interaction between zinc polycarboxylate cement and nanoparticles resulted in the changes of zinc polycarboxylate cement peaks.

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