

## **Compressive and Diametral Tensile Strength of Dental Stones with SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> Nanoparticles**

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### **Abstract**

It was investigated the effect of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> nanoparticles on mechanical properties of Type III and Type IV dental stones. A total of 200 disc-shaped specimens in 3 mm diameter and 6 mm height for compressive tests and specimens in 6mm diameter and 3mm height for diametral tensile tests were prepared (n=10) (Control, %1 SiO<sub>2</sub>, % 5 SiO<sub>2</sub>, %1 Al<sub>2</sub>O<sub>3</sub>, %5 Al<sub>2</sub>O<sub>3</sub>). Before mechanical tests, specimens were kept in dry condition for 7 days. Compressive and diametral tensile tests were performed in an universal test machine. The data were statistically analyzed using ANOVA and Tukey's HSD test. The interaction between nanoparticles and type of dental stone was found to be significantly important (p<0.05). The lowest diametral strength was observed in 5% SiO<sub>2</sub> nanoparticle added Type IV dental stone. The lowest compressive strength was observed in 5% SiO<sub>2</sub> nanoparticle added Type III dental stone. For both dental stones the compressive and diametral strength values decreased by adding SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> nanoparticles. The diametral and compressive tensile strength values decreased as increasing %wt of SiO<sub>2</sub> and

Al<sub>2</sub>O<sub>3</sub> nanoparticles for both dental stone and improved dental stone. The incorporation of %1-%5 SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> nanoparticles to Type III and Type IV dental stones decreased the diametral and compressive strength. As the %wt of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> nanoparticles increased, the compressive and diametral tensile strength of both dental stone and improved dental stone decreased.

**Keywords:** Compressive strength, Dental stone, Diametral tensile strength, Nanoparticle.

**Clinical Significance:** An important and recent change in inorganic fillers has been the application of nanotechnology to the development of dental products. Such modified materials have improved their mechanical and physical properties, leading to better clinical performance. How the incorporation of nanoparticles affects the mechanical properties of the stone has not been established.

### **Introduction**

Dental stone is versatile and important for the production of precise casts that represent clinical situations [1]. Gypsum products have been considered to be among the most widely used model and die materials [2]

Gypsum is a mineral composed of calcium sulfate dihydrate, with the chemical formula  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ . Gypsum is found in nature as flattened, often twined crystals and transparent cleavable masses known as selenite. It is also available in compact and granular forms [3]. Gypsum materials are chosen as a die material for the reason that is easy manipulation, economic disposition compatibility and their passion towards most impression materials [4]. Die materials play an important role during the fabrication of indirect dental restorations and prostheses. Indirect method of fabrication of inlays, crowns and bridges demand die materials that are of the highest quality with respect to accuracy and strength [5]. Successful die materials should have good strength and hardness to withstand normal laboratory and clinical handling. Recently, many attempts have been made to enhance the properties of gypsum products through the addition of chemical materials [6].

A die should be accurate in every respect, i.e. dimensionally stable over time, minimal setting expansion, ease and efficiency of manipulation, compatible with impression materials, hard enough to withstand the fabrication process, resistant to the inadvertent abrasions caused during fabrication, good transverse strength and a stable shelf life [5]. At present, gypsum materials for clinical use are categorized as Types I through V as per No. 6873 of The International Standard Organization (ISO). Type I is a dental plaster for impressions. Type II is a dental plaster for fabricating models. Type III is a dental stone for creating models. Type IV is a low-expansion, high-strength dental stone for fabricating models and dies. Type V is a high-expansion, high-strength dental stone for fabricating models and dies. To produce a precise model and die, both minimal expansion during hardening and strength must be excellent [1]. Stone casts are key materials in the dental

laboratory and clinical practice and must accurately reproduce the structures obtained from the impressions. To accomplish this, accuracy techniques and appropriate materials are required. The Type IV dental stone is widely used to fabricate the dies and master casts for fixed and removable partial prostheses, due to its superior mechanical properties such as compressive strength, hardness and expansion properties when compared to other dental Stones [7]. It is essential to obtain a strong cast with smooth and hard surface characteristics in order to allow for wax sculpting, especially at the cervical margin without cast abrasion. A hard surface is necessary for a dye stone to be resistant to abrasion, because the cavity preparation is filled with wax that is carved flush with the margins of the dye [8]. The criteria used to select the stone include its mechanical properties (such as, surface roughness), [9,10] diametrric tensile strength (DTS), [11,12-14] compressive strength, [10,11,13] wear resistance, [14,15] surface hardness [1,13,14] and ability to reproduce the detail [15,16]. The compressive and diametral tensile strengths have been the most common laboratory testing modalities to characterize mechanical and physical properties of dental stone. [17] The strength of gypsum – based products is usually expressed in terms of compressive strength, which is directly related to the material's ability to fracture resistance when subjected to compressive tensions. Thus the dental stones' compressive strength is an important factor in the rehabilitation work of dentistry [18]. In the set gypsum material, the number of crystals formed during setting and their inter-meshing and enlargement determines the strength [19].

The applications of these nanotechnologies has rapidly expanded into all areas of health care science including that of odontological science [20]. Particles such as quartz, colloidal silica, silica glass containing barium, strontium, and zirconia have been used in dental materials

as different types of inorganic fillers. Different shapes and sizes of filler particles are used in commercial products and can affect the properties of the materials [21]. An important and recent change in inorganic fillers has been the application of nanotechnology to the development of dental products, with the main goal of improving their mechanical properties [22]. With the emergence of nanoscale technology, materials, including adhesives and composite resins, have been modified by these technological advancements. Such modified materials have improved their mechanical and physical properties, leading to better clinical performance [22,23]. With improvements in this technology, new dental materials with nanoparticles are expected. How the incorporation of nanoparticles affects the mechanical properties of the stone has not been established.

The purpose of this study was to evaluate the mechanical properties (diametral tensile and compressive strength) of Type III and IV stones after the addition of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> nanoparticles in different concentrations. The null hypothesis (H<sub>0</sub>) was that the addition of these particles would not change the mechanical properties of the dental stones studied.

### Materials and Methods

In present study, Type III dental stone and Type IV improved dental stone were used as gypsum material. SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> nanoparticles were used as reinforcing materials. Used materials and manufacturers were given in Table 1. Specimens were divided into two groups as A and B. (Table 2) A and B groups were divided into five subgroups according to reinforcing nanoparticle type and % weight of nanoparticle. Specimen groups were given in Table 2. A total of 200 disc-shaped specimens (100 specimens for compressive tests and 100 specimens for diametral tensile tests) were prepared (n=10). Specimens in 3 mm diameter and 6 mm height for compressive

tests and specimens in 6mm diameter and 3mm height for diametral tensile tests were prepared.

The specimens used in the mechanical tests were prepared with the aid of two teflon molds. The stone powder and nanoparticles were weighed using a precision digital scale (Denver Instruments), and distilled water was measured using a 10 mL glass pipette (Satelit) as recommended by the stones' manufacturers and according to the test groups for the nanoparticles. (Figure 1) Nanoparticles (Table 3) were mixed for homogeneous distribution for 2 h at room temperature (Fritsch Pulverisette-5, Idar-Oberstein, Germany) with a rotation speed of 400 cycles/minute in a dry condition. Steel balls with a diameter of 7 mm with a diameter of 7 mm were used for the mixing process. The dental stones were mechanically spatulated under a vacuum (Polidental) following the time recommended by the manufacturers and poured into the mold under vibration (Table 3). The specimens were allowed to set for 1 hour before separating from molds. Then, they were waited in dry condition for 7 days before mechanical tests. Compressive tests were performed in an universal test machine with 1 mm/min. cross head speed. Compressive loading were applied until specimen was broken and compressive load values were recorded. Compressive strength values were calculated by Equation 1. Where  $\sigma$  (MPa) is compressive strength,  $F$  (N) is compressive load at fracture and  $d$  (mm) is specimen diameter.

$$\sigma = \frac{4F}{\pi d^2} \quad \text{Eq. (1)}$$

Diametral tensile tests were performed in an universal test machine with 1 mm/min. cross head speed. Diametral tensile strength were calculated by Equation 2. Where  $\sigma$  (MPa) is diametral tensile strength,  $F$  (N) is diametral tensile load at fracture,  $d$  (mm) is specimen diameter and  $h$  (mm) is specimen height.

$$\sigma = \frac{2F}{\pi dh}$$

Eq.(2)

tensile strength values of dental stone with %1 Al<sub>2</sub>O<sub>3</sub> and that of with %5 Al<sub>2</sub>O<sub>3</sub>nanoparticlewere 4.1 and 3.8 MPa.

### Statistical analysis

The results of compressive strength and diametral tensile strength data were analyzed by multiple factorial ANOVA followed by Tukey's honestly significant difference (HSD) test with a general linear model procedure in SSPS 17.0 (SPSS Inc., Chicago, USA). A significance level of 0.05 was used for statistical tests.

### Results

Mean and standard deviations of compressive strengthfor groups were given in Table 4 and Figure 2. Mean compressive strength of Type III dental stone (control group) was 50.6 MPa.While mean compressive strength values of dental stone with %1 SiO<sub>2</sub> andthat of with %5 SiO<sub>2</sub> were21.8 and 13.9MPa, mean compressive strength values of dental stone with %1 Al<sub>2</sub>O<sub>3</sub> and that of with %5 Al<sub>2</sub>O<sub>3</sub> were 21 and17.1MPa.

Mean compressive strength of Type IV improved dental stone (control group) was 36.1 MPa. While mean compressive strength values of dental stone with %1 SiO<sub>2</sub> and that of with %5 SiO<sub>2</sub> were24.7 and 14.2MPa, mean compressive strength values of dental stone with %1 Al<sub>2</sub>O<sub>3</sub> and that of with %5 Al<sub>2</sub>O<sub>3</sub> were 25 and 17MPa.

Mean compressive strength of control group of Type III dental stone was higher than mean compressive strength of control group of Type IV improved dental stone.The compressive strength values decreased as increasing %wt ofSiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> nanoparticlesfor both dental stone and improved dental stone.

Table 5 and Figure 3showed mean and standard deviations of diametral tensile strength values for groups.Mean diametral tensile strength of Type III dental stone (control group) was 8.8 MPa. While mean diametral tensile strength values of Type III dental stone with %1 SiO<sub>2</sub> and that of with %5 SiO<sub>2</sub> nanoparticlewere4.9 and 2.4 MPa, mean diametral

Mean diametral tensile strength of Type IV improved dental stone (control group) was 8.8 MPa. While mean diametral tensile strength values of Type IV dental stone with %1 SiO<sub>2</sub> and that of with %5 SiO<sub>2</sub> nanoparticlewere5.4 and 2.1MPa, mean diametral tensile strength values of dental stone with %1 Al<sub>2</sub>O<sub>3</sub> and that of with %5 Al<sub>2</sub>O<sub>3</sub> nanoparticlewere 5.9 and 4.9 MPa.

Mean diametral tensile strength of control groups of Type III dental stone and Type IV improved dental stones were same. The diametral tensile strength values decreased as increasing %wt ofSiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> nanoparticlesfor both dental stone and improved dental stone.

### Discussion

The null hypothesis was rejected. The incorporation of nanoparticles significantly decreased the mechanical properties of dental stone. Although they are not directly used as dental restorative materials gypsum products are important adjunctive materials used in many laboratory procedure [24].The technology applied to obtain small, shaped particles, and the sources of hemihydrates (obtained naturally from gypsum or chemically) are possible explanations for the differences in the behavior observed [25].The strength of gypsum - based products is usually expressed in terms of compressive strength, which is directly related to the material's ability to fracture resistance when subjected to compressive tensions. Thus the dental stones' compressive and diametral tensile strength are important factors in the prosthetic rehabilitation [18].Inadequate compressive strength, dimensional instability, technique sensitivity and susceptibility to abrasion, are some of the shortcomings of gypsum products [26].

De Cesero et al. [27] also evaluated the mechanical properties of dental stone after the addition of silica nanoparticles in different concentrations. The addition of silica nanoparticles effected the diametral tensile strength of Fuji Rock when 5 wt% was added and the compressive strength in both concentrations ( $p < 0.05$ ). Similarly, in our study, the addition of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  nanoparticles in both concentrations also decreased the diametral and compressive strength in both concentrations but highly effected when 5 wt% was added. The mechanical properties of dental stone materials are influenced by several factors [27]. The water-to-powder ratio significantly affects compressive strength, [28,29] because water creates pores inside the material that weaken it because there are fewer crystals by volume. Within the limitations of this study, the use of nanoparticles to improve mechanical properties and variation in the powder-water ratios recommended by the manufacturer could have affected the results of our research like De Cesero et al. [27] detected the additives as the reason of the similar results in study. The lack of standardization of diametral tensile strength methodology in the literature makes it difficult to compare results. De Cesero et al. [27] found the mean compressive strength values of control group, 1% wt and 5% wt silica nanoparticles addition groups for Type IV dental stone (Fuji Rock) as 42.9, 31.2 and 29.8 MPa respectively whereas it was 36.1 MPa, 24.7 and 14.2 MPa for Type IV dental stone (Elite Rock) with 1% wt and 5% wt  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  nanoparticles in our study. Mean compressive strength values of Type IV dental stones decreased as increasing concentration of  $\text{SiO}_2$  nanoparticles in our study like De Cesero et al. [27].

De Cesero et al. [27] also found the mean diametral tensile strength values of control group, 1% wt and 5% wt silica nanoparticles addition groups for dental stone (Fuji

Rock) as 6.4, 5.2 and 4.5 MPa respectively whereas it was 8.8 MPa, 5.4 and 2.1 MPa for Type IV dental stone (Elite Rock) with 1% wt and 5% wt  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  nanoparticles in our study. Mean diametral tensile strength values of Type IV dental stones decreased as increasing concentration of  $\text{SiO}_2$  nanoparticles in our study as De Cesero et al. [27].

Taqa et al. [6] added 0.5%, 1%, 1.5% and 2% concentrations rosin, nigella stavia oil and sodium lauryl sulfate (different chemical materials) to Zeta, Elite and Dental Stone (different Type III dental stones). Compressive strength and surface hardness of specimens were determined. The highest compressive strength value were obtained in Zeta having 1% rosin as we found the same result for added nanoparticles in our study.

Khalaf et al. [30] used silver nitrate powder 1% and copper sulfate powder 1% while treating the Type IV dental stone specimens with the disinfecting powders. 1%  $\text{AgNO}_3$  and 1%  $\text{CuSO}_4$  produce great reduction in compressive strength than the control group and other experimental groups, This may be related to the increase in the rate of reaction so that some of the hemihydrates crystals does not get hydrated to form dihydrate crystals, this increase the unreacted hemihydrates contents in the materials and thereby produces a weaker product [18]. Of the set material, [19] the prepared dental stone specimens with the additives have shown a reduction in compressive strength in comparison to the control specimens like our study, this could be either related to the presence of additional excess water in the mixture or to the decrease interaction (inter crystallization cohesion) between the gypsum crystals related to decreased amount of gypsum crystals as a result of increased concentration of additives in a given volume of gypsum material [30].

Kati et al. [31] investigated the effect of some additives (cured resin, pulverised stone, pulverised plaster and

glass fibers) and drying methods (air and microwave) on compressive strength of dental plaster and stone. They found higher compressive strengths differently from our study which we used nanoparticles.

On the other hand, because of thermal and mechanical aging processes are not performed, it is not known whether these differences in the values will be observed clinically. The lack of this factor may be a limitation in our study, but can be investigated in future studies. Within the limitations of our study, the incorporation of both SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> nanoparticles adversely effected the mechanical properties of the dental Stones independently from the type of the stones and nanoparticles. Studies should test the mechanical effects of other types and %wt of nanoparticles on different commercial brands of dental stones.

### Conclusion

Based on the findings of the in vitro study, the following conclusions were derived:

1. The incorporation of nanoparticles to Type III and Type IV dental stones decreased the diametral and compressive strength.
2. As %wt of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> nanoparticles increased, compressive and diametral tensile strength decreased for both dental stone.

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- Table Legends
- Table 1: The materials and manufacturers
- Table 2: Specimen groups
- Table 3: Dental and improved dental stone properties
- Table 4: Mean and standard deviations of compressive strength values for groups
- Table 5: Mean and standard deviations of diametral tensile strength values for groups

**Table 1.**The materials and manufacturers

Materials	Manufacturer
Elite Model SpA, Italy	Zhermack
Elite Rock Italy	Zhermack SpA,
Al <sub>2</sub> O <sub>3</sub> nanopowder (99.5% pure, powder size 40-50 nm)	MKNANO, Canada
SiO <sub>2</sub> nanopowder coated with silane coupling agent (99.5% pure, powder size 15 nm)	MKNANO, Canada

**Table 2.**Specimen groups

Groups	Description
Group A	Dental stone(Elite Model)
Group B	Improved dental stone(Elite Rock)
Subgroups	
1	without nanoparticles (control group)
2	containing %1 wt SiO <sub>2</sub> nanoparticles
3	containing %5 wt SiO <sub>2</sub> nanoparticles
4	containing %1 wt Al <sub>2</sub> O <sub>3</sub> nanoparticles
5	containing %5 wt Al <sub>2</sub> O <sub>3</sub> nanoparticles

**Table 3.**Dental and improved dental stone properties

Product	Water/Powder Ratio (g/ml)	Mixing Time (sec)
Elite Model	100/30	40
Elite Rock	100/20	30

**Table 4.** Mean and standard deviations of compressive strength values for groups

Material	Group	Mean ± SD
Elite Model	Control	50,6±2,9
	%1 SiO <sub>2</sub>	21,8±1,5
	%5 SiO <sub>2</sub>	13,9±1,3
	%1 Al <sub>2</sub> O <sub>3</sub>	21±1,6
Elite Rock	Control	36,1±2,7
	%1 SiO <sub>2</sub>	24,7±1,8
	%5 SiO <sub>2</sub>	14,2±1,2
	%1 Al <sub>2</sub> O <sub>3</sub>	25±2,3
	%5 Al <sub>2</sub> O <sub>3</sub>	17±1,7

**Table 5.**Mean and standard deviations of diametral tensile strength values for groups

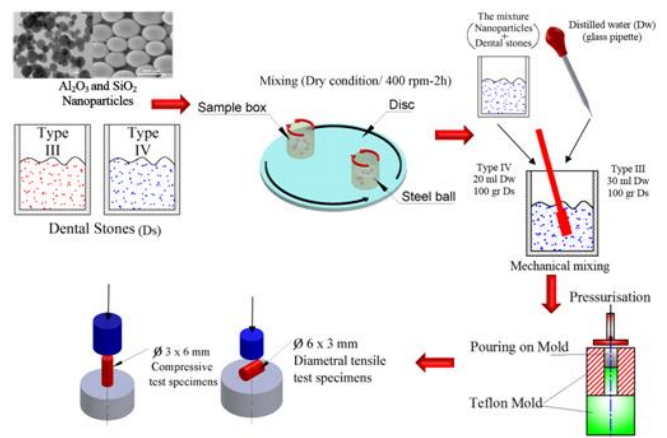
Material	Group	Mean ± SD
Elite Model	Control	8,8±0,8
	%1 SiO <sub>2</sub>	4,9±0,5
	%5 SiO <sub>2</sub>	2,4±0,2
	%1 Al <sub>2</sub> O <sub>3</sub>	4,1±0,3
Elite Rock	Control	8,8±0,8
	%1 SiO <sub>2</sub>	5,4±0,5
	%5 SiO <sub>2</sub>	2,1±0,2
	%1 Al <sub>2</sub> O <sub>3</sub>	5,9±0,6
	%5 Al <sub>2</sub> O <sub>3</sub>	4,9±0,6

**Figure Captions**

Figure 1 The preparation of the sample and mechanical test configuration

Figure 2: Compressive strength values of groups

Figure 3: Diametral tensile strength values of groups



**Figure 1 .** The preparation of the sample and mechanical test configuration



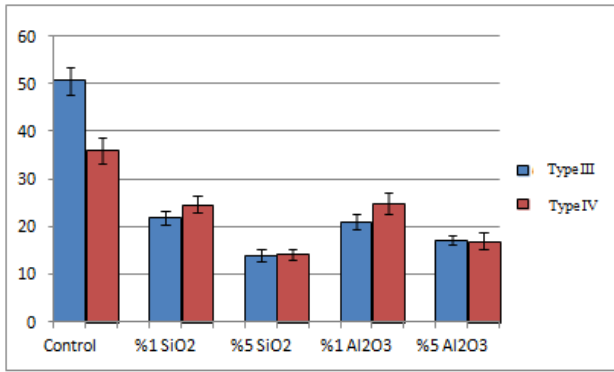


Figure 2 Compressive strength values of groups.

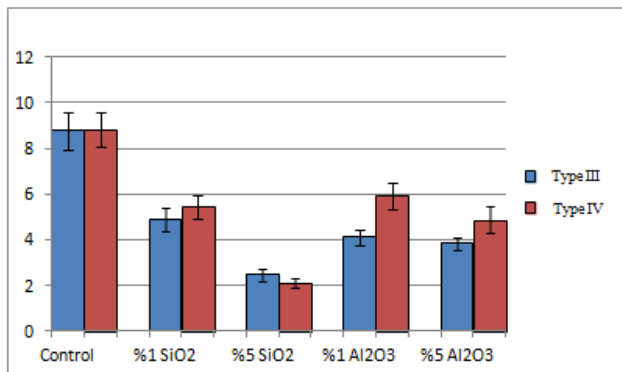


Figure 3 Diametral tensile strength values of groups.