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*RESEARCH ARTICLE - MEDICAL TECHNIQUES*

# Evaluation of the Effect of Nano and Micro Hydroxyapatite Particles on the Impact Strength of Acrylic Resin: In Vitro Study

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**Abstract**

Poly (methylmethacrylate) is considered the basis material for a denture base. However, such substance has some drawbacks such as poor impact resistance, which is thought to be the primary cause of fracture of denture base resins. The purpose of the study was to determine how Nano and Micro hydroxyapatite particles affected the impact strength of acrylic resin. Thirty specimens were constructed of heat-cured acrylic resin and were divided into three groups: Ten specimens for the control, 10 for 1%nano hydroxyapatite, and 10 for micro hydroxyapatite. Acrylic samples were subjected to an impact strength test via a Charpy-type. Data were then analysed using SPSS v20. The ANOVA test was used for comparison among the groups. Highly statistically significant differences among all studied groups (P-value <0.0001). Both 5% Micro hydroxyapatite and 1% Nano hydroxyapatite had a higher mean value than the control. Incorporating Nano and Micro hydroxyapatite into PMMA improved the impact strength of acrylic resins.



Publisher: Middle Technical University

**Keywords**: Nano Hydroxyapatite; Micro Hydroxyapatite; Acrylic Resin; Impact Strength; Silane Coupling Agent.

## 1. Introduction

The most common denture base material used in clinical production has been acrylic resin since Walter Bauer first polymerized it in 1936, replacing the traditional metal base [1]. Its extensive use is a result of several advantages rather than one standout quality, such as its acceptance of adequate aesthetic requirements, and having a noticeably clear processing technique in dental applications. On the other hand, there were several drawbacks to this material, particularly in terms of meeting the mechanical requirements of a prosthesis [2]**.** However, PMMA's weak strength, low impact strength, and fatigue resistance were its key drawbacks [3]. Therefore, it is essential to improve PMMA's performance when used as denture bases. Several techniques were used to change the characteristics of PMMA denture base materials. Two techniques for enhancing acrylic resin prosthetics were resin modification or reinforcement with nanoparticles [4-7]. The use of Nanofillers in dentistry in the last years improved the mechanical properties of denture base materials [8]. For clinical use of denture bases, outstanding mechanical performance, as well as adequate biocompatibility and biosafety, are also needed, as some inorganic materials may irritate or even injure oral mucosa and gingival tissue [9, 10]. It was claimed that hydroxyapatite nano- or micro powder has been successfully used as a biocompatible filler to reinforce the self-cured polymer matrix [11].

The mechanical properties of PMMA/HA composites may be constrained by the incompatibility of PMMA with HA, similar to the incompatibility of any inorganic filler. Hydroxyapatite nanoparticles were treated with 3-methacryloxypropyltrimethoxysilane to enhance their capacity to link and interact with acrylic resins. (-MPS). The silane coupling agent (3-methacryloxypropyltrimethoxy silane (-MPS)) was found to enhance greatly the physical and mechanical characteristics of poly methyl methacrylate and Nano HA [12]. The goal of this study was to determine how Nano- and Micro hydroxyapatite addition will affect the acrylic resin impact strength.

## 2. Materials and Method

In the current study, a salinized Nano hydroxyapatite (particles size (40nm), Hualanchem, China) and micro hydroxyapatite (particles size (0.52µm), SUNKOO Ltd, South Korea) were used. Heat-cured acrylic resins (Czech Republic) were used for the fabrication of acrylic samples.

### *2.1. Samples groups*

Thirty samples were made for this study, including ten control samples, ten specimens containing 1% HA nanofiller, and ten specimens containing 5% HA microfiller.

| Nomenclature & Symbols | | | |
|---|---|---|---|
| PMMA | Poly Methyl Methacrylate | NS | Non-Significant |
| MMA | Methyl Methacrylate | MPa | MegaPascal |
| HA | Hydroxyapatite | P | P-Value |
| G | Gram | S | Significant |
| mm | Millimeter | SD | Standard Deviation |
| SPSS | Statistical Package for the Social Sciences | SE | Standard Error |
| Max. | Maximum | Min. | Minimum |
| HS | High Significant | | |

*2.2. Samples design*

In this study, plastic strips were made via cutting plastic plates using the laser-cutting machine as illustrated in Fig. 1. Acrylic samples (80 mm length x 10 mm width x 4 mm thickness) were made according to ISO.179-1, 2000 for unnotched specimens.

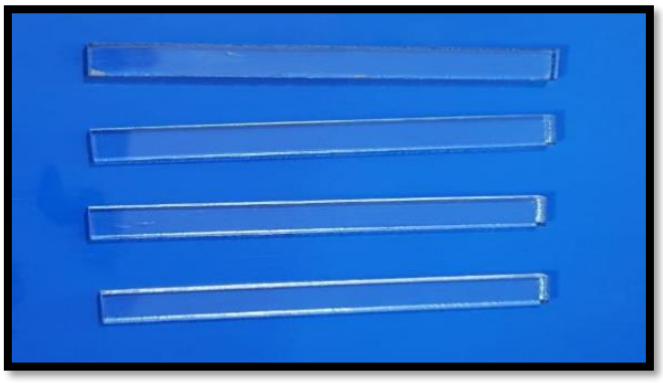

Fig. 1. Plastic patterns for the required impact strength test

*2.3. Silanation of hydroxyapatite*

Using (Trimethoxysilyl) propyl methacrylate (Sigma–Aldrich, USA) in a mixture of 90% methanol and 10% distilled water, the HA nanoparticles were silanized. In a beaker, 10 ml of Trimethoxysilyl was dissolved in 70 ml of acetone and 30 ml of deionized water. The liquid combination (acetone, water, and silane) was then combined with a magnetic stirrer (Faithful, China) at 350 rpm at room temperature for 4 hours while containing 30 grams of HA (Nano, or Micro). The mixture was dried in an oven (Faithful, China) for 24 hours at 110°C. The final powder was grinded and sieved using a 75-micron mesh size and then added to the PMMA in a magnetic stirrer at room temperature for 2 hours at 350 rpm. Finally, the resultant powder was mixed with the monomer according to the manufacturer's instructions [13].

*2.4. Proportioning and mixing ratio of HA nanofiller and microfiller*

Table 1 demonstrated the quantities of acrylic resins, nanofiller hydroxyapatite, and micro filler hydroxyapatite.

Table 1. Proportion of the acrylic resins and hydroxyapatite used in the study

| Groups | Amount of HA (g) | Amount of acrylic powder (g) | Amount of acrylic monomer (ml) |
|---|---|---|---|
| Control | 0 | 22 | 10 |
| 1%wt.Nano hydroxyapatite | 0.22 | 21.78 | 10 |
| 5%% wt. Micro hydroxyapatite | 1.1 | 20.9 | 10 |

*2.5. Preparation of the acrylic samples*

The lower part of a metal flask was coated with Vaseline, and the dental stone (Silky rock, Turkey) was mixed according to manufacturer guidelines and poured into the lower part of the flask. The plastic patterns were lubricated with Vaseline and positioned in the mid-surface of the dental stone. After a complete set of the stone surface, it was then lubricated with the separating medium and left to dry. Then the upper part of the metal flask was positioned over the lower part, and filled with another mix of dental stone. The mold was left to set for 60 minutes [14]. Following that, the two parts of the flask were separated and plastic patterns carefully were removed as shown in Fig. 2 [15].

The stone parts were coated with the separating medium before packing them with acrylic dough [16]. The dough mixture was taken from the mixing jar and rolled before being put into the mold [17]. To ensure that the dough flowed evenly throughout the mold space, the two parts of the flask were put together and placed under pressure. The trail was blocked off, and to make packing simpler, a polyethylene sheet was utilized [18]. The clamped flask was put in a water bath and then heated to a temperature of 74°C for 1.5 hours, followed by 30 minutes increase to reach boiling point. This process was used to cure acrylic specimens. Once curing, the metal flask was left to cool. The acrylic specimens were then removed from the stone mold and deflasked after this time. A lathe polishing machine (Italy) was used to finish and polish each specimen. A bristle brush and a ruge wheel with pumice in a lathe-polishing machine were used to achieve a gloss surface. All specimens were submerged in cold water in a rubber bowl. To ensure that the measurements were as stated, a digital Vernier was used to measure each specimen. For two days, all specimens were kept for 2 days in distilled water at 37°C before testing [15].

*2.6. Impact strength testing*

The impact strength test was carried out with a Charpy-type impact testing instrument (Tmi, testing machines Inc. Amity Ville, New York, USA) according to (ISO 179, 2000) as demonstrated in Fig. 3 and 4. A free-swinging pendulum struck the specimen as it was suspended

horizontally at its ends. (Apendulums with a testing capability of 2 joules were utilized). When struck by a quick blow, the scale reading indicated the impact energy absorbed to shatter the specimen in joules. This test was determined in $KJ/m^2$ using the formula below: Impact strength = E/B. D X $10^3$ [19].

- The letter E indicates the impact absorbed energy (in joules).
- The letter B indicates the width of the specimens (in millimeters)
- The letter D indicates the thickness of the specimens (in millimeters)

Fig. 2. Mold preparation for impact test specimens

Fig. 3. Before impact testing, a position the sample in the test device (Charpy type)

Fig. 4. After impact testing, a position the sample in the test device (Charpy type)

## 3. Results

All data were analysed using SPSS v 20. The ANOVA test was used for comparison among the groups, the results indicated highly statistically significant differences (*P*-value <0.001). The highest mean of impact strength was (13.661± 0.412 MPa) for 1% Nano and the lowest mean value was (11.547± 0.373 MPa) for control as demonstrated in Table 2 and Fig. 5.

Table 2. Descriptive statistics of impact strength (MPa) for all studied groups

| Groups | No. | Mean | SD | SE | Min. | Max. | ANOVA Test |
|---|---|---|---|---|---|---|---|
| Control | 10 | 11.547 | 0.373 | 0.118 | 11.180 | 12.220 | *P*<0.001 (HS) |
| 1%Nano hydroxyapatite | 10 | 13.661 | 0.412 | 0.130 | 13.040 | 14.440 | |
| 5% Micro hydroxyapatite | 10 | 13.291 | 0.458 | 0.145 | 12.740 | 14.180 | |

*HS is highly significant

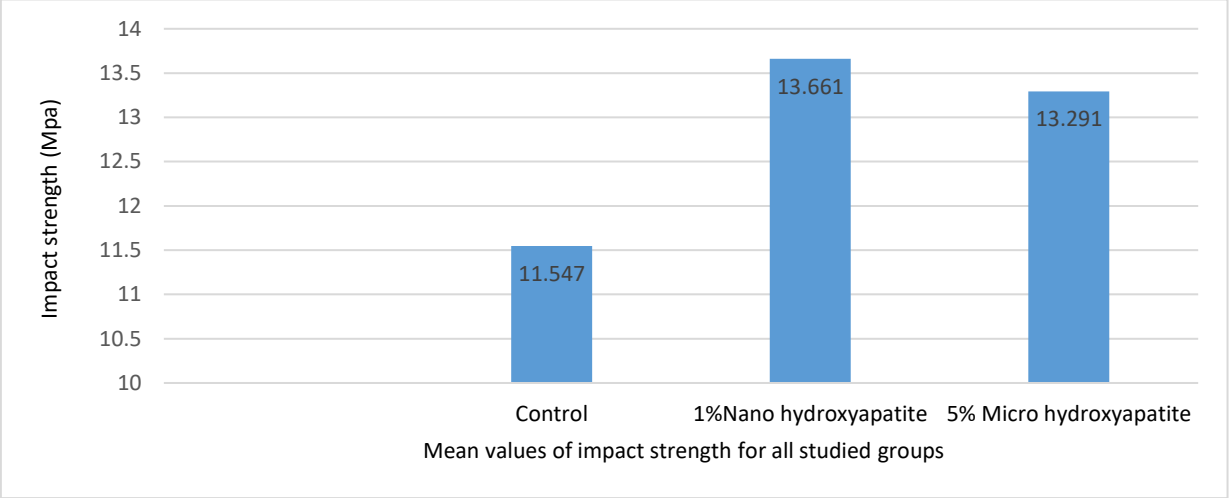


Fig. 5. Bar chart illustrated the mean values of impact strength (MPa) among all groups

## 4. Discussion

The effect of modified Nano and Micro hydroxyapatite filler on the mechanical characteristics of heat-cured denture base material was examined in this work. Because of its bioactivity and biocompatibility, hydroxyapatite was utilized in this study as well as in numerous biomedical disciplines such as dental material and bone substitute [20]. Impact strength is a measurement of how much energy a material can withstand before breaking suddenly. A complete denture resin should ideally have enough impact strength because dropping the prosthesis could result in extra orally high impact forces. The material's other qualities must not be hampered by this high-impact strength [21].

The results of this study indicated that the addition of 1% Nano hydroxyapatite and 5% micro hydroxyapatite to heat-cured acrylic resins increased their impact strength by treating the particles with coupling agents, which improved their bonding with the polymer matrix and prevented the particles from clustering, which in turn reduced crack propagation. On the other hand, the mechanical characteristics of acrylic resin were significantly influenced by the concentration, size, and distribution of HA particles in the polymer matrix as well as by strong adhesion at the interface [22].

Few articles about the impact of Nano hydroxyapatite and micro hydroxyapatite on the characteristics of acrylic resins have been published. Safi [23] found an improvement in impact strength after adding the same concentration of zirconia nanofiller to PMMA even though the improvement was statistically not significant. The findings of the current study were in agreement with Zange and Tanner [24], who demonstrated a significant increase in impact strength when hydroxyapatite filler was added to polyethylene composite to use this biomaterial for skull implants. This study's findings on impact strength are consistent with those of Asar et al. [25], who found that adding (alumina, titania, and zirconia) to PMMA independently increased impact strength. The current findings were in agreement with those of Zainab [26], who claimed that the use of nano-hydroxyapatite increased the impact strength of acrylic resins by forming an effective polymer/microfiller network that in turn increased impact strength. The current findings contradict those of Diwya [27], who discovered that adding hydroxyapatite reduced the impact strength of acrylic resins. Diwya and his colleagues did not treat the hydroxyapatite particles with a saline coupling agent and this had a weak matrix interaction between the polymer and hydroxyapatite. The current findings were in line with Karadi [22] who concluded that adding two percent of Nano hydroxyapatite improved the impact strength of polymethylmethacrylate because of using the coupling agents to treat HA particles, which enhanced interfacial bonding.

## 5. Conclusion

The incorporation of Nano and Micro hydroxyapatite into PMMA increased the impact strength of heat-cured acrylic resins.

## 6. Recommendations

- Evaluation of the effect of Nano and Micro hydroxyapatite on mechanical properties, such as surface hardness and surface roughness of acrylic resins.
- Assessment of the influence of different concentrations of Nano and Micro hydroxyapatite on the color stability of acrylic resins.
- Study the effect of Nano and Micro hydroxyapatite on physical properties, such as water sorption and porosity) of acrylic resins.

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