

Mechanical Performance of Heat-Polymerized PMMA Reinforced with 0.7 wt % Nanoparticles

An Evaluation of Tensile Strength, Flexural Strength, and Shore D Hardness

Ali Ehsan Kareem¹, Tameem Khuder Jassim¹, Nagham Bahjat Kamil¹, Samir Samier², Rawaa Ali Rahmat¹

¹College of Dentistry, Mustansiriyah University, Baghdad, Iraq

²College of Dentistry, University of Kerbala, Kerbala, Iraq

Abstract

This study's goal was to determine how adding 0.7 w t% aluminum oxide (Al₂O₃), copper oxide (CuO), and zinc oxide (ZnO) nanoparticles affected the mechanical characteristics of heat-cured poly (methyl methacrylate) (PMMA), particularly in terms of tensile strength, strain, flexural strength, and Shore D hardness. After heat-curing PMMA samples, they were divided into four groups: an unmodified PMMA group and three experimental groups that were reinforced with 0.7 wt% of Al₂O₃, CuO, or ZnO nanoparticles, respectively. Tensile strength, flexural strength, and Shore D hardness tests were used to determine mechanical properties. ANOVA was used to evaluate the differences between the groups. Every mechanical property that was evaluated showed a significant difference between the four groups ($P < 0.001$). Control group's highest stress whereas Al₂O₃ group's maximum stress value was significantly lower. Maximum stress values for CuO and ZnO groups were 56.32 ± 3.44 MPa and 71.59 ± 1.05 MPa, respectively. The mean maximum strain was similar for Control and CuO, lowest for Al₂O₃, and highest for ZnO. All three nanoparticle-reinforced groups showed an increase in flexural strength when compared to control group. When nanoparticles were added, Shore D hardness also rose to 80.70 ± 0.9 . The mechanical performance of heat-polymerized PMMA was considerably altered by addition of 0.7 wt % nanoparticles. ZnO showed the best tensile response by producing highest maximum strain and sustaining maximum stress around control, and Al₂O₃ showed most gain in flexural strength and surface hardness. CuO improved flexural strength and hardness while producing an intermediate mechanical response. These results suggest that choosing the right nanoparticles is crucial in dental applications.

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Email: dr.nagham_b@uomustansiriyah.edu.iq

Introduction

One of the most frequent and clinically significant problems in removable prosthodontics is denture base fracture, which can lead to poor patient outcomes, functional impairment, and higher maintenance expenses [1]. Due to its favorable qualities, including ease of handling, acceptable aesthetics, biocompatibility, and

affordability, polymethyl methacrylate (PMMA) has long been the preferred material for denture bases [2]. Despite serving these functions, PMMA has intrinsic limits because to its poor impact strength, fatigue resistance, and crack initiation and propagation under functional and parafunctional pressures. These subpar mechanical performances shorten the lifespan of prosthetics and result in midline

fractures, especially in patients who need strong occlusal loads or poorly fitting dentures. In addition to mechanical limitations, denture base materials are frequently subjected to a variety of variables, including temperature, moisture, pH, microbial activity oscillations, and a mixed surface and microorganisms [3]. Denture stomatitis and oral infections are made more likely by this environment, which

also encourages biofilm formation and bacterial colonization [4]. You have thus become aware of the increasing significance of improving the mechanical and antibacterial performance of denture base materials.

Recently, new methods to improve the performance of polymeric dental materials have been suggested using nanotechnology. There may be potential for overcoming PMMA matrices' inherent shortcomings with the use of nanoparticles. These nanoparticles interact with the polymer matrix more effectively due to their unique physicochemical characteristics, which include a high surface area-to-volume ratio and increased reactivity's [5]. However, a number of parameters, including as the type, concentration, size, surface treatment, and quality of particle dispersion within the polymer matrix, has a significant impact on the reinforcing effect. For material performance to significantly increase, these variables must be optimized [6]. Aluminum oxide (Al_2O_3), copper oxide (CuO), and zinc oxide (ZnO) nanoparticles have shown encouraging results among the many nanofillers studied for denture base reinforcement because of their superior mechanical qualities and advantageous physicochemical features. Al_2O_3 nanoparticles provide for efficient stress distribution throughout the PMMA matrix because of their high hardness, elastic modulus, and wear resistance. ZnO nanoparticles are especially appealing for dental applications since they have been shown to display antibacterial activity and improve mechanical characteristics. CuO nanoparticles' ability to enhance mechanical behavior and offer antibacterial qualities that can help lower microbial colonization on denture surfaces has also drawn further attention. Because of variations in nanoparticle concentration, fabrication methods, particle dispersion, and experimental protocols, published studies have reported inconsistent results regarding the magnitude of mechanical improvement despite these encouraging findings [7].

Direct comparative studies incorporating Al_2O_3 , CuO , and ZnO nanoparticles at the same concentration are still scarce, even though several studies have assessed the reinforcing effect of individual nanoparticles on PMMA denture base resin. By standardizing the concentration of nanoparticles, associated to concentration is reduced, and the inherent reinforcing capacity of each nanofiller can be compared more accurately. Thus, by measuring maximum stress, maximum strain, flexural strength, and Shore D hardness, the current work sought to examine and compare the mechanical performance of heat-polymerized PMMA reinforced with 0.7 wt % Al_2O_3 , CuO , and ZnO nanoparticles. The results of this study should help choose the best nanoparticle

reinforcement to enhance the longevity and clinical performance of PMMA denture base materials.

Material and Methods

Preparing Samples

The recommended powder liquid ratio of acrylic resin employed in this investigation is 2.5 g: 1 ml or a multiple, per manufacturer recommendations. The necessary concentration (0.7%) applied to the monomer liquid is determined by weighing the nanoparticles on an electronically sensitive scale. The monomer containing nanoparticles is quickly mixed with acrylic powder [8] to reduce the possibility of particle aggregation and phase separation. After that, the mixture is covered and left to develop into dough. The flask's mold, which has already been covered with a separating media, is filled with acrylic resin during the dough stage. Finally, the flask is closed under hydraulic press pressure until metal-to-metal contact is achieved, and it is then left under press 20 bar for five minutes. The clamped flask is then placed in a water bath at 70°C for about 90 minutes before the temperature is raised to boiling temperatures for at least 30 minutes [9]. Before removing the acrylic specimens from the mold, let the flask cool gradually in the water bath. Prior to testing, all samples were conditioned in water at 37°C for 48 hours in accordance with ADA specification No. 12.1999 [10]. Each test (hardness, tensile strength, and flexural strength) used 20 specimens in total.

Sample Grouping

Twenty specimens were broadly divided into two groups: n=5 control group and n=15 experimental groups. The experimental group of nanoparticles (Al_2O_3 , CuO , ZnO) is further divided into 3 subgroups with n=5 specimens at concentrations (0.7%) (Figure 1) (Al_2O_3 , CuO , ZnO) nanoparticles with an average diameter of 35-45 nm were purchased from Areej Al-Furat company for materials processing and laboratory devices.

Tensile strength

The method used in this study followed ISO527-1: (2019) [11], standards. The dimension of the specimens was 75×4-6 (±0.2) ×2(±0.2) mm Dumbbell-shaped for tensile strength. The size of all the specimens was measured by a micrometer accurate to ±0.01 mm. A digital vernier caliper with an accuracy of ±0.01 mm was used to measure each specimen's size. The rectangular cubic specimens were fastened with levers and positioned at pertinent positions on a universal tensile strength measurement device (Model HZ-1007A, Dongguan Lixian Instrument Scientific Co., Ltd., Dongguan, China). The tensile bond

strength based on MPa was computed using the following formula:

Bond strength (MPa) = Amount of force (N)/Crosssection surface area (mm²).

Flexural Strength (FS) Bending Test

A three-point bending test was performed to determine the flexural strength of acrylic specimens in compliance with ISO 20795-2: (2013) [12], requirements using a universal testing machine (Tinius Olsen, Model H50KT, UK). The machine's loading plunger was adjusted to produce a steady displacement rate of 5 mm/min, and distance between the support centers was set to 50 mm. In accordance with the ISO 20795-2: (2013) [12], standard, five specimens (n = 5) from each group were prepared with a length of 65 mm, a width of 10 (±0.2) mm, and a height of 3.3 (±0.2) mm. The specimens were stored and prepared according to the method described by Mahmoudi Anzabi et al. [13]. The apparatus recorded the maximum force required to fracture each specimen (in Newtons), and the following equation was used to calculate the flexural strength (σ) in megapascals (MPa):

$$\sigma = 3Fl/2bh^2$$

Where F is the maximum load applied to the specimen in newtons; l is the distance in millimeters between the supports; b is the specimen's width in millimeters measured just before water storage; and h is the specimen's height in millimeters measured just before water storage.

Shore D Hardness

For hard materials, the Durometer hardness tester (Shore D, Shaw, Model: LD-YJ, China) was used to measure surface hardness. The American National Standard and American Dental Association (ANSI/ADA, 2002) [14], were followed in its fabrication. Each specimen had five measurements taken in various locations, and the mean value was determined. Since the shore durometer gages the indenter's relative movement, it is a dimensionless number.

Statistical Analysis

The mean ± standard deviation (SD) was used to express all experimental results. IBM SPSS Statistics for Windows Version 18.0 [15] was used to do the statistical analysis. For the cytotoxicity experiments, one-way analysis of variance (One-way ANOVA) was used to examine differences between experimental groups. A statistically significant value was defined as $P < 0.05$.

Results

Table 1 shows that while G2, G3, and G4 contained PMMA modified with 0.7 weight percent of Al_2O_3 , CuO , and ZnO nanoparticles, respectively, the control group (G1) included unmodified heat-polymerized PMMA. The four

groups differed significantly in every mechanical property that was examined. Maximum stress, maximum strain, flexural strength, and Shore D hardness showed statistically significant differences between the groups ($P < 0.001$), as demonstrated by one-way ANOVA. It was discovered that the maximum stresses varied considerably among the different experimental groups. The average maximum stress for the control group (G1) was 72.88 ± 1.14048 MPa. The maximum stress values significantly decreased to 18.6 ± 1.5 MPa, the lowest value among the four groups, upon the addition of 0.7 weight percent Al_2O_3 nanoparticles. The sample's maximum stress after CuO was added was 56.315 ± 3.43621 MPa, whereas the ZnO-reinforced group (G4) had a maximum stress of 71.587 ± 11.04743 MPa. As a result, the ZnO group's maximum stress value was extremely similar to the control groups. While the mean maximum strain for the control group was 7.495 ± 0.13039 %. At 6.452 ± 1.26279 %, the group containing Al_2O_3 had the lowest mean value. The mean maximum strain of the CuO group was 7.142 ± 0.64732 %, which is comparable to the control group. With a maximum strain of 11.374 ± 3.25598 %, the ZnO group achieved the highest value.

The control group's mean flexural strength was 103.036 ± 0.707 MPa, according to the flexural strength data. Compared to the control, all three of the nanoparticle-reinforced groups showed greater mean flexural strength values. The Al_2O_3 group has the highest value, measuring 116.422 ± 2.704 MPa. The CuO group reported 109.106 ± 1.268 MPa, whereas the ZnO group recorded 111.326 ± 3.299 MPa.

The results of the hardness tests showed that, at 65.32 ± 0.41473 Shore D, the control group had the lowest average hardness. With a hardness value of 80.7 ± 0.97468 Shore D, the Al_2O_3 group obtained the highest hardness value. The CuO group came in second with 80.6 ± 1.11803 Shore D. The medium hardness value for the ZnO group was 71.5 ± 6.7328 Shore D. In Figure 3, the graph (A) displays the average maximum tensile stresses for each of the four experimental groups. Maximum tensile stress was measured at the highest level of 72.88 MPa in the control group (G1). The Al_2O_3 modified sample (G2) exhibits a dramatic decline in tensile stress, with the lowest result among all groups examined at 18.6 MPa. In the CuO modified sample (G3), the maximum tensile stress value then increases to 56.32 MPa. Further, a rise is noticed in the case of the ZnO modified sample (G4) to a value of 71.59 MPa, which is practically equivalent to the control. On the other hand, Graph (B) shows the mean of the greatest strain. The mean strain in the control group was 7.5%. The mean strain decreased to 6.45%, the lowest of the four groups, following

the addition of Al_2O_3 nanoparticles. At 7.14%, the CuO group had the next lowest figure, which was near the control. However, out of the four groups, the ZnO group showed the highest strain, at 11.37%. As a result, the graph's trend differs from that of maximum stress.

The trend shown in the third graph (C) is entirely different. With a mean of 103.04 MPa, the control sample (G1) exhibited the lowest flexural strength. After adding Al_2O_3 , there was a noticeable increase in flexural strength; the G2 group recorded the highest flexural strength value of 116.42 MPa. The ZnO group's average value was 111.33 MPa, whereas the CuO group's was 109.11 MPa. As a result, all three of the nanoparticle-modified groups had higher flexural strengths than the unmodified control. The means of the Shore D hardness values are displayed in Graph (D). With a mean of 65.32, the control sample exhibited the lowest hardness. With a mean of 80.7, the most notable rise was seen following the addition of Al_2O_3 , and the CuO samples had a similarly high mean of 80.6.

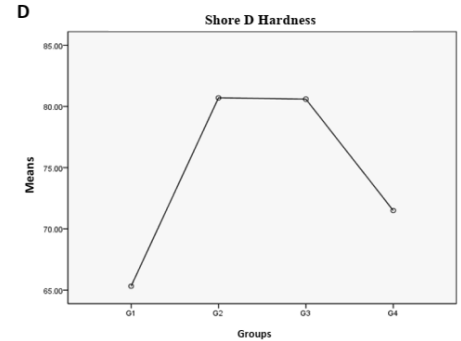
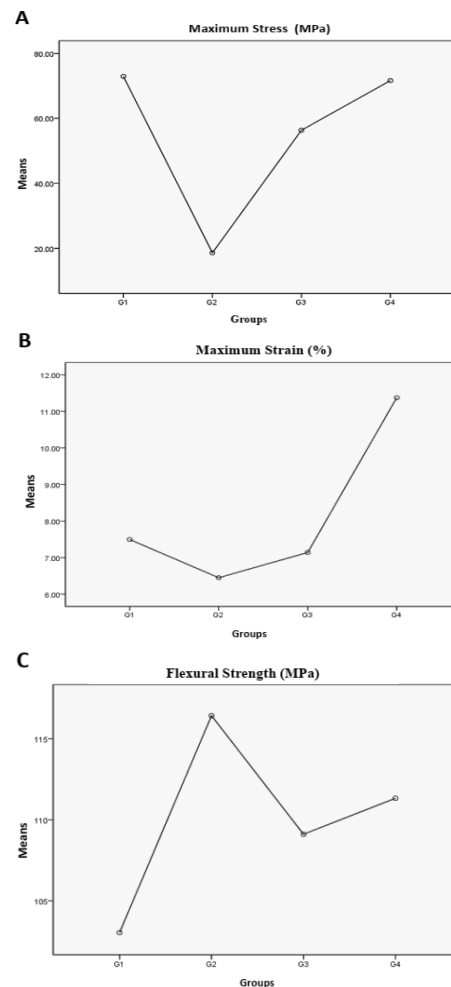


Figure 2. Mean mechanical properties of the tested groups.

Discussion

The impact of 0.7 wt % Al_2O_3 , CuO, and ZnO nanoparticles on the mechanical characteristics of heat-polymerized PMMA was evaluated in this work. It was discovered that the addition of nanoparticles did not consistently affect every mechanical property; rather, the impact varied according to the kind of nanoparticle and mechanical property examined. This is consistent with current understanding that suggests the composition, concentration, size, distribution, and interaction of nanoparticles with the polymer matrix affect the mechanical properties of dental acrylic resins in distinct ways. The introduction of nanoparticles may not always result in improvements in all mechanical parameters, according to a systematic review and meta-analysis of dental acrylic resins modified by nanoparticles, which discovered significant variances in mechanical properties [16].

The inclusion of 0.7 wt % Al_2O_3 nanoparticles significantly reduced the maximum stress, which is the most important finding from the current study. When Al_2O_3 nanoparticles were added to the specimens, the maximum stress dropped from 72.88 MPa in the control specimens to 18.6 MPa. The addition of nanoparticles does not provide an increase in tensile strength, even though Al_2O_3 is a strong and rigid ceramic that was considered in the literature as the reinforcing phase for PMMA. The degree of nanoparticle dispersion, particle and matrix adhesion, particle concentration, and processing parameters all affect mechanical characteristics; Proper dispersion of nanoparticles in the matrix and efficient stress transfer between the polymer matrix and reinforcing phase are critical to the effectiveness of nanoparticles in enhancing the mechanical characteristics of polymers. If not, nanoparticle aggregation may result in stress concentrations that cause early crack and failure start. The concentration, size, and distribution of nanoparticles are given consideration in recent evaluations of dental acrylic resins reinforced with nanoparticles [17].

Therefore, it is evident that this result is consistent with the idea that an appropriate nanoparticle formulation is required rather than just raising the concentration of nanoparticles. According to a recent review for Galant et al. [17], of the mechanical properties of PMMA/Ag nanoparticle composites, the tensile and flexural strengths vary according to particle size and concentration rather than increasing linearly with respect to nanoparticle concentrations. This demonstrates the general rule even when silver nanoparticles were used in place of Al_2O_3 , CuO, or ZnO nanoparticles. The ZnO group performance stood out from the rest. The maximum stress of 71.59 MPa was quite near to the 72.88 MPa regulation threshold. This implies that the PMMA material's tensile strength was mostly preserved at 0.7 wt % ZnO. Most notably, ZnO had the highest strain of 11.37%. To show that incorporating nanosized ZnO into PMMA may alter some of the polymer's physical properties, some research has been done with an emphasis on the impact of introducing ZnO nanoparticles on the properties of PMMA. Salahuddin et al. [18], discovered that adding ZnO improved the mechanical and thermal characteristics of PMMA. This may indicate that, compared to the Al_2O_3 sample, the ZnO sample offers a better trade-off between preserving its strength and deformability. However, as tensile deformation is influenced by several variables, including polymerization, unreacted monomers, filler distribution, and matrix-particle interaction, this finding needs to be regarded with caution.

It is also important to note that all the groups reinforced with nanoparticles had higher mean flexural strengths than the control group, with aluminum oxide (Al_2O_3) exhibiting the highest flexural strength (116.42 MPa). When compared to the control group, it represents an improvement of about 13%. It might be explained by the inorganic nanoparticles strengthening function within the polymeric material matrix. Well-distributed inorganic nanoparticles can aid in stress transmission and lessen the polymer matrix's deformability. When the contact is right, the high stiffness of aluminum oxide (Al_2O_3) nanoparticles can aid in preventing deformations while bending the material. According to recent research on heat-polymerized PMMA incorporating Al_2O_3 nanoparticles [19], the addition of Al_2O_3 led to increased flexural strength, with the degree of strength varying according to the concentration of Al_2O_3 . The outcomes of the current investigation may be connected to the observation.

However, the current study shows that while a certain concentration of Al_2O_3 produced the largest flexural strength, it also indicated the lowest tensile stress. Since the stress distribution in the tensile strength and flexural

strength testing differs, it would be inappropriate to infer any conflicts from this result. As a result, the increase in tensile strength and flexural strength cannot be directly related. These findings are consistent with earlier research that shown that the impact of nanoparticles on acrylic resin varied according on the quantity of nanoparticles utilized. According to Ozkan et al. [20], flexural strength was enhanced by 1 wt % TiO_2 , but it was diminished by other nanoparticles, including CuO, in high concentrations. Therefore, rather than if high concentrations of nanoparticles will always enhance PMMA, it is crucial to adjust nanoparticle concentration for each specific nanoparticle. It's interesting to note that the CuO group in this investigation had a flexural strength value of 109.11 MPa, which was higher than the control. This finding must be considered, too, as previous research has shown a variety of results regarding the impact of CuO on the flexural strength of acrylic resin. Ozkan et al. [20] found that the 3 wt% CuO group had a lower flexural strength than the control group. This suggests that the concentration of CuO has a substantial impact on the mechanical properties of the acrylic resin.

On the other hand, the hardness measurements demonstrated one of the biggest improvements caused by nanoparticles. When Al_2O_3 and CuO were added, Shore D's hardness increased from 65.32 in the control sample to 80.7 and 80.60, respectively. The corresponding improvement values are 23.5% and 23.4%. The addition of hard inorganic particles to the PMMA matrix is responsible for the hardness improvement. This is since nanoparticles restrict deformation in a single location, which hinders the mobility of the polymer chain and increases resistance to indentation. When the reinforcement particles are hard and evenly distributed throughout the polymer matrix, the hardness augmentation is more apparent. Research on ZnO-PMMA nanocomposites also shows that inorganic nanoparticles can improve PMMA hardness. Cierech et al. [21], showed that ZnO nanoparticles improve PMMA's hardness; a statistically significant improvement in hardness was seen when compared to the unmodified PMMA at the maximum ZnO nanoparticle concentration used in the test. According to the researchers, adding inorganic nanofillers might improve the polymer matrix's hardness. However, compared to ZnO, the hardness improvement of Al_2O_3 and CuO was more noticeable in the current investigation. This could be explained by variations in the particles' characteristics and how they interact with the PMMA matrix. However, they could only be regarded as likely contributors because particle shape, surface chemistry, and dispersion were not examined in the current

experiment. Increased surface hardness can result in improved wear and scratch resistance, making it another potentially significant element when choosing a dental material. According to Cierech et al. [21] hardness may be a crucial characteristic for denture base materials' resistance to surface wear.

Overall, the present study shows that the mechanical characteristics of heat-polymerized PMMA are affected in a nanoparticle-dependent manner by the 0.7 wt % addition of Al_2O_3 , CuO, and ZnO nanoparticles. Al_2O_3 significantly reduced the tensile maximum strength, but having a better influence on the flexural strength and Shore D hardness. CuO had a small impact on tensile strength, while its hardness and flexural strength were greater. Conversely, ZnO exhibited the highest maximum strain and an ideal tensile strength that was close to the control. The findings show that the choice of nanoparticle for PMMA reinforcement relies on the desired mechanical quality required for the specific application; no single nanoparticle would improve all mechanical qualities at once. The results are in line with earlier research showing that the type, concentration, shape, size, dispersion, and interaction of the particles with the matrix all have an impact on the reinforcement of dental PMMA utilizing nanoparticles [17].

Conclusion

The current investigation showed that the mechanical characteristics of heat-polymerized PMMA were affected differently by the addition of 0.7 weight percent Al_2O_3 , CuO, and ZnO nanoparticles. ZnO demonstrated the best tensile performance, with a maximum stress of 71.59 MPa and the largest strain (11.37%), whereas Al_2O_3 demonstrated the most gain in flexural strength (116.42 MPa) and surface hardness (80.7 Shore D). CuO had a somewhat balanced and intermediate impact, especially on hardness. Overall, no nanoparticle enhanced every mechanical feature at once, suggesting that the type of nanoparticle and the mechanical quality being assessed determine the reinforcing impact. Within the constraints of this work, 0.7 wt% ZnO seems promising for applications that need tensile performance, but 0.7 wt% Al_2O_3 would be more appropriate when flexural strength and surface hardness are the most important factors.

Conflicts of Interest

The authors declare no competing interests.

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Figure 1. Flow chart depicting distribution of specimens.

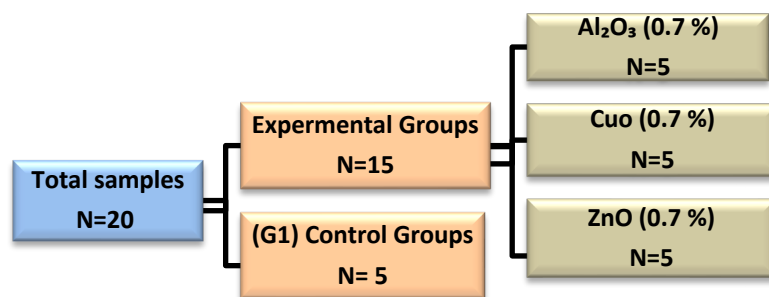


Table 1. Mechanical properties of PMMA reinforced with 0.7 wt% nanoparticles.

Groups	Maximum Stress (MPa) Mean $\pm$ SD	Maximum Strain (%) Mean $\pm$ SD	Flexural Strength (MPa) Mean $\pm$ SD	Shore D Hardness Mean $\pm$ SD
G1 Control	72.88 $\pm$ 1.14048	7.495 $\pm$.13039	103.036 $\pm$ 0.707	65.32 $\pm$ 0.41473
G2 Al ₂ O ₃ (0.7%)	18.601 $\pm$ 1.50125	6.452 $\pm$ 1.26279	116.422 $\pm$ 2.704	80.7 $\pm$ 0.97468
G3 CuO (0.7%)	56.315 $\pm$ 3.43621	7.142 $\pm$.64732	109.106 $\pm$ 1.268	80.600 $\pm$ 1.11803
G4 ZnO (0.7%)	71.587 $\pm$ 11.04743	11.374 $\pm$ 3.25598	111.326 $\pm$ 3.299	71.5 $\pm$ 6.7328
P-Value	< 0.001	< 0.001	< 0.001	< 0.001