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# The Influence of Adding Zero-Dimensional Nanomaterial on Certain Mechanical Properties of Repaired Heat -Cured Acrylic Denture Base

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**Abstract**

Denture fracture is a prevalent issue in prosthodontic practice that affects patients and prosthodontists. This study aims to investigate the influnce of MgO nano fillers on flexural and impact strengthof repaired heat–cured acrylic resin denture base. Materials and methods: Sixty heat-cured acrylic resin specimens have been created according to the ISO standard 1567:1999/2003(E) for denture base polymers. Thirty specimens were created for flexural strength test with dimension of ( 65mm x10mm x2.5 mm ±0.1mm) and the other thirty specimens for impact strength test with dimension of (50mm x 6mm x 4mm ).Curing was done in a water bath machine,andcompleted the finishing and polishing. Then, the specimens separation were done by a separating disc . Each group was subdivided as follows: Group (A): control repaired done with self-curing resin without MgO nanoparticles, Group (B) and (C): the repaire wasdone with self-curing resin with the addition of MgO nanoparticles at concentrations of 2wt.%and 4wt.% (n=10). Curing repaired specimens in Ivomat device. Data were analyzed using Tukey HSD test andaP-value ≤ 0.05 was considered- a statistically significant.Results: TheTukey HSD test indicated significant difference P-value ≤ 0.05 in theflexural strength between control group and group 4wt. % MgO, whereas no significant differencewas identified between 2wt.%MgOwith4wt.%MgO. However,there was no significant difference in the impact strength (P > 0.05) for all groups.Conclusions: The incorporation of 2wt.% of MgO nano fillers into repaired PMMA resin had no effect on flexural or impact strength.

## Introduction:

Although poly (methyl methacrylate) (PMMA) resin is a popular material for denture base manufacturing, it has significant limitations one of these is denture fracture [1] Low transverse and impact strength results in prostheses that are unable to retain durability over time. During intra-oral function, denture base acrylic resin is submitted to a variety of masticatory volars, compressive, tensile, and shear stresses, which cause fatigue[2,3]. Impact outside the mouth may cause the prosthesis to fracture. This sort of fracture happens along or near the midline and is more common in maxillary dentures than in mandibular dentures [4 ,5]. New denture manufacture is a therapy option; however, it is time demanding and pricey for the patient.So denture repair is deemed optional and preferable [6]. A good denture repair should match the denture base's original color, be quick, and restore the denture's strength, maintaining the dimensional stability, easily repaired process and quickly performance[7]. Numerous studies have been conducted to study various repair surface layouts, repair materials, reinforcement, and surface treatment in order to increase repair intensity and evade recurring fracture[8]. Several materials, including auto polymerized, visible light polymerized, and microwave polymerized acrylics, have been utilized to repair fractured denture bases[9]. Auto polymerized acrylic resin has been the most often used repair material for more than sixty years. It gained popularity due to its ease of use and repairs quickly. However, one of its biggest disadvantages is its poor strength[10]. Heat polymerized acrylic resin has excellent mechanical qualities.Nevertheless, its use is limited. Due to time-consuming laboratory processes and the possibility of denture deformation caused by heat[2]. As aresult ,autopolymerized resin repair is frequently used for denture base repair becuase it is simple to use, saving chair side time, and laboratory processing is not a prerequisite. Additionally,during the repair process, less time is spent by the patient without a denture [11]. Fractures of repaired dentures occur commonly as a result of insufficient binding strength between denture base resin and repair resin material[12]Regardless of the approach utilized, the interface of the old and new materials is clearly identified as a region of stress concentration during transverse strength testing[13]. The combination of denture base resin and repair material used may also have an impact on the ultimate strength of denture repair[6]. Repair surface design is particularly significant for the repair process since it effects bond strength. The bevel joint displayed the highest mechanical qualities among butt, round, bevel, rabbet, inverse rabbet, inverse, and knife edge joints in addition to its simplicity of clinical application[14]. Furthermore, the 45-degree bevel increased the bond area and changed the interfacial stress distribution toward shear stress rather than tensile stress, which is damaging[8], altering the fractured type from weak adhesive to strong cohesive fracture [15] . Acrylic resin surface treatment, whether chemical or mechanical, alters the surface topography or chemistry of the treated surface to improve adhesion[16]. Surface conditioning enhances surface energy, which leads to improvement in wetting for the purpose of bonding. In order for the repair surface to be prepared and modify its topography so that adequate surface adhesion can be produced and enhance shear bond strength, chemical solvents such as acetone, methyl methacrylate , chloroform, and methylenechloride are utilized[17]. Repair material reinforcement, in addition to repair surface design and treatment, is regarded as a significant aspect in denture repair effectiveness. Previously, fractured acrylic dentures were reinforced with metal, wire, mesh, fiber (glass, nylon, aramid, ultra-high modulus polyethylene, etc.), and fillers[8]. Nowadays, nanotechnology has forayed the dental field , resulting in improved denture repair qualities[18]. Metal oxides are among the several types of nanoparticles that are useful, not only for the variety of their physical and chemical characteristics, but also for their

antibacterial capabilities[19]. Among the well- known metal oxide nanoparticles, magnesium oxide MgO has received a lot of interest due to its innovative uses in industries including petrochemical products, coatings, detection, adsorption, ceramics, catalysis, electronics, and treatment of warfare agents, as well as chemical waste and various fields[20] . The disadvantage of the widely utilized repair materials is their low strength. The present focus of dental material research is on finding the best repair material with acceptable strength and a long shelf life. The effect of MgO nanoparticles on flexural and impact strength has not been studied. As a result, this study has been carried out to find out the reinforcing effects of various MgO concentrations on the flexural and impact strength of repaired PMMA denture base material. The null hypothesis was that adding varying concentrations of nano MgO to repaired denture base would increase their flexural and impact strength.

## Materials and Methods:

**specimens preparation**

Atotal of 60 heat polymerized acrylic resin specimens were ready for the flexural and impact strength tests. Based on the "American dental association ANSI/ADA " specification no.12 for denture base polymers, metal patterns with dimensions( 65 x 10 x 2.5 ± 0.1mm) length, width, and thickness respectively were fabricated for the flexural strength test[21]. Regarding the impact strength test, the dimensions of metal patterns were (50 x 6 x 4mm) Length ,width and thickness respectively according to ISO standard 1567:1999/2003 (E) for denture base polymers[22]. Molds preparation for acrylic resin were done according to the conventional flasking method[23]. The metal patternswere coated with a separating medium (DENTSLIY DE Trey GmbH,England) and allowed to dry. Half thickness of the patterns profundity were embedded in the investment material (Dental stone type III, Zhermack S.P.A). The metal flask was opened after a complete setting of investment material. Then ,the resulted molds were prepared for acrylic packing by removing the metal patterns [23], as shown in Fig.(1). As indicated in the manufacturer's specifications, heat polymerized acrylic resin(Veracril®, Colombia )was prepared in aratio of (40g powder) to (17 ml monomer) for each mold and then, placed into the mold cavity at the dough stage. Trail closures were done, and the flask was sealed and bench pressed for half hour. Acrylic resin specimens were treated for 8 hours in a water bath curing unit (Model HH S2) at 74 °C, followed by 1 hour in a thermal curing unit at 100 °C [8, 12]. Then opened the flask and The samples underwent the typical heat curing procedure applied in denture manufacturing, in which burs, abrasive paper, and pumice were all used for finishing and polishing [8, 24]. The repaired groups were divided into control group (repaired with self-curing resin without adding of MgO NP and two tested groups based on MgO nanofiller concentration as following:

Thirty specimens of flexural strength were subdivided into three groups:

**Group A F:control group repaired with autopolymerized acrylic resin without nanofiller addition .**

**Group B F:group repaired with 2wt.% MgO nanoparticles.**

**Group C F: group repaired with 4wt.% MgO nanoparticles.**

**The other thirty specimens of impact strength were subdivided into three groups:**

**Group A I:control group repaired with autopolymerized acrylic resin withoutnanofilleraddition .**

**Group B I:group repaired with 2wt.% MgO nanoparticles.**

**Group C I: group repaired with 4wt.% MgO nanoparticles.**

**The Process of Preparation of Specimens for Repair**

All specimens were numbered on both ends for the ease of reassembling, as shown in Fig.(2). For flexural strength test,a line was drawn on the center of the specimen then from this line, two lines were drawn 2.5mm away from both sides of the top of the center line on the right and left to establish a 5-mm repair gap.

Then, a1.25mm was marked on the right and left from the bottom of the center line to to establish a 2.5-mm repair gap[24]. Allmarkings were perpendicular to the specimen's long axis.The specimens were cut along these lines using a low speed diamond disc and profuse irrigation [12], and the two lines drawn on either side of the central line were utilized as guidelines for typical 45° bevel joint preparation[25].The dimensions of pairs of repair group samples were evaluatedusing a digital caliper (Renfert GmbH, Germany) [8]. For impact strength specimens, a 5mm away from both sides of the top of the center line on the right and left to set up a 10-mm repair gap.Then, a1.25mm was marked on the right and left from the bottom of the center line to set up a 2.5-mm repair gap, After that, the samples were cut with a diamond disc (as mentioned in preparation of flexural strength specimen) in a bevel direction according to the linesmarked on the samples until creating a bevel joint of 45° between both pieces [24], as illustrated in Fig.(3) (A, B).

**The Process of Repair**

The two parts of the sample numbered on both ends to be repaired were realigned in its index and fixed into the previously-coated mold with a separating medium[12], as demonstrated in Fig.(4).The methyl methacrylate monomer was applied to the repair surfaces for three minutes (MMA)[26].The repair compound (self-curing denture base resin superacryl® plus, SpofaDentala.sCzech Republic) was made exactly as directed by the manufacturer . The proportion for mixing the self-curing acrylic resin was 6.556g powder to 4ml monomer for each mold of control group .The packing of acrylic resin material into the repair gap was done while the acrylic at fluid stage[27] , which may have contributed to a better bond and a lower percentage of adhesive failure.The slight excess of material wasused to account for polymerization shrinkage. To complete the polymerization processes, the flask that includes repairing the samples was placed in a pressure pot (Ivomat device IP3,ivoclarvivadent GmbH) for 15 minutes at 2bar pressure[12]. When the resin polymerization was finished, the flask sections were opened and the samples were gently extracted from the flask using a wax knife. The specimens were brought back for finishing. The extra acrylic was removed using a tungsten carbide bur, and then with a 600-grit abrasive paper and large amounts of water. Later, they were polished in a dental lath machine. To validate the measurements of repaired specimens, a digital caliper (Renfert GmbH,Germany) was used [25].

**Incorporation of MgO Nanofillers to the Repair Material**

The amount of MgO nanofillers particles (50nm ,MW=40.30,Assay 99.9 %,CAS number1309-48-4,US Research Nanomaterials,USA) wereweighed using an electronic balance using a 4- digit (AS 220.R2 Plus Radwag Poland). According to the other studies [25, 28 ,29 , 30 ,31 ,32 ,33 ,34 , and 35 ]that used a magnetic stirrer for mixing nanomaterial or for silanization treatment , this study utilized the magnetic stirrer for mixing the repairing materialswithout heating. The MgO NP was added separately to the monomer at concentrations of 2wt.% and 4wt.%and thoroughly mixed using magnetic stirrer(Korea, model HY-HS11) by using a clean and dry beaker for three minutes to ensure that particles were evenly distributed. Then,the repair gap was overfilled after mixing the monomer and nano-filler with a polymer to be then packed and consequently, the steps would be complete as the same previous procedure for the control group. Later, the repaired specimens were finished and polished for all the tested groups to be prepared for the transverse strength and impact strength tests. For 48 hours at 37 °C, all of the samples were submerged in distilled water[36] .

**Flexural Strength Test**

In this investigation, an Instron universal testing machine (JIANQAO,China) was utilized to measure the flexural strength of acrylic specimens using a three-point bending technique, as shown in Fig.(5). The acrylic specimen was placed on the

bending fixtureto besupported by two horizontal parallel arms.The distance between those arms is 50mm. At a crosshead speed of 5mm/min, a loading force of 50 kgf was delivered to the center of the test specimen until the fracture occurred[8,37]. The calculation of the flexural strength of test specimen was done as follows:

Flexural strength = $3PL/2BD^2$ where P is the peak load (in newton). L is the length of the span (expressed in mm). B is the width of the specimen (expressed in mm) D is the specimen thickness (expressed in mm).Thevalue of flexural strength is expressed in Mega Pascale (MPa).

**Impact Strength Test**

A pendulum Charpy-type impact test machine was used to perform an impact strength test (TMI NO 43-1, USA)[38,39]. Each specimen was horizontally positioned at its ends, as shown in Fig. 6. (A, B). At room temperature[40],a free pendulum was struck by a fixed height in the center.A pendulum with a testing capacity of two joules was employed[39]. There was a digital record for the value of the impact strength ($KJ/m^2$) with the help of the following formula: impact strength =E/b x d where E is the absorbed energy, b is the sample width,d is the sample thickness.

**StatisitcalMethod**

Data on flexural and impact strength were analyzed with the SPSS software version 20. The flexural and impact strength tests yielded mean and standard deviation results. Tukey HSD was used to compare the differences between test groups. P value at the 0.05 level was used to determine the level of significance.

# Result:

**Flexural Strength Test :**

Table (1) and Fig.(7). represented the mean values and standard deviation of flexural strength of all acrylic resin repairs. The highest flexural strength value was recorded in control group (repaired with auto polymerized PMMA+ 0 wt.% nano fillers) (44.3415 ± 8.46780 Mpa) followed by the group repaired with auto polymerized PMMA reinforced with 2wt.% MgO nanoparticle (37.0385 ± 8.03474Mpa).In contrast, the lowest mean value of flexural strength was obtained in the group repaired with autopolymerized PMMA reinforced with 4wt.% MgO nanoparticle
( 31.6080 ± 8.44786 MPa).

The comparison between mean values of flexural strength was summarized in Table (2). Tukey HSD recorded a non-significant difference in the flexural strength mean values withinthe group repaired with autopolymerized reinforced with 2wt.% MgO nanoparticle and the group repaired with autopolymerized reinforced with 4wt.% MgO nanoparticle.Similarly,there was also a non-significant difference within control group (free of nanoparticle) and the group repaired with auto polymerized reinforced with 2wt.% MgO, but the difference in comparing the control group with group repaired with 4wt.% MgO nanoparticles was significant.

**Impact Strength Test***:*

Table (3) and Fig.(8) represented the mean values and standard deviation of impact strength test for all groups. Control group revealed the highest mean value for impact strength (11.3033 ± 2.40174 $KJ/m^2$ )while the group autopolymerized PMMA + 2wt.%MgO and group 4wt.% MgO showed a similar difference of impact strength (9.8767 ± 0.87012 $KJ/m^2$), (9.5483 ± 1.30392 $KJ/m^2$ ), respectively.

Tukey HSD test for comparison between groups was shown in Table (4).The data revealed non-significant improvements for all the repaired groups i.e. control group (free of nanoparticle) and those groups reinforced with 2wt.% MgO and 4wt.% MgO nanoparticle.

# Discussion:

This *in vitro* study has been conducted for assessing the consolidating effects of various MgO concentrations on the transverse and impact strength of a repaired denture base.

**Flexural strength**

Several approaches were used to repair the resin dentures fractured to their previous strength. Preparing the surfaces of the joining areas is critical to guaranteeing the prosthesis's long-term service life[26]. The repair process had a considerable impact on the strength of the restored denture. Generally speaking,the selection of repair material is generally determined by the repair material's strength,the design of the repair surface and the reinforcement of repair material selected[8]. The null hypothesis was rejected, where the current investigation found that the control group (cold cure acrylic resin without reinforcement) had higher fracture strength. This conclusion is consistent with Polyzois et al. [7] who found that specimens repaired using cold cure acrylic resin without reinforcement had higher fracture strength. while the findings of this study contradict with Heidari et al.[36]. Leong and Grant [2], Berge [41] , and Rached et al. [42] conducted research demonstrating that dentures restored with the use of self-curing acrylic resin could be shattered at the spot treated , potentially as a result of the decrease in strength of the resin.The lower cold cure acrylic resin strength appears to be due to an insufficient polymerization process [10]. In comparision to the control specimens, test specimens of PMMA/MgO nanoparticles showed a significant decrease in flexural strength values. This decrease might be attributed to MgO Nanoparticles' brittleness and inability to withstand flexural stress[43]. It might possibly because of the presence of MgO nanoparticles within or between PMMA chains, which could weaken and disrupt the intramolecular forces.The individual chain ,which will be shortened and easily distorted under stress, may weaken and break the cross-links between polymer chains, leading to interruptions within PMMA matrix and uneven dissipation of the adsorbed load. Also, it may weaken the intermolecular attraction forces between polymer chains, which impair the ability of PMMA matrix to transfer the adsorbed load by shear movement between its chains. Furthermore, it may limit the mobility and deformation ability of PMMA chains during the testing method[40].This reduction in flexural strength in group 4wt.% MgO nanoparticles in comparison tothe control group which the statistical analysis showed a significant difference between them Table (2). This can be attributed to a variety of factors, comprising a rise in the percentage of the filler, resulting in several defects which influence the strength of the material; particle piling up found in the resin; and more filler particles after matrix being saturated, which causes a break in resin matrix continuity[5].Such outcomes can be attributed by the inter-facial layer generated between the additives and the PMMA matrix, the possibility of filler conglomerate , and the degree of additive dispersion in the repair material[12]. While the results of 2wt.% MgO nanoparticles did not show a significant difference in comparison to the control group, this could be imputed to the good allocation of the nano size particles, which allows them to get in and top up the areas between polymeric chains, leading to raising interfacial bonding strength between polymeric chains and the nanoparticles [3] . Furthermore, in accordance with Gad et al., 2016 the filler concentration directly controlled the increase in flexural strength [8] .

**Impact Strength**

Due to the thermal insulating qualities of MgO nano fillers[20], they may minimize heat conduction during PMMA curing, in addition to the negative connection between the degree of nanofiller dispersal and the amount of the reacted monomer. These conditions may result in a rise in residual monomer and the creation of voids in polymer/nanocomposite. Van der walls forces are responsible for the interfacial adhesion of inorganic nano fillers with PMMA matrix [38,43] . These weak connections diminish the elastic modulus of the modified polymer at low pressures ,but they resort to shatter at high impact loads, making the material more brittle(decrease the toughness) . The findings of the investigation demonstrated that the repaired specimens of PMMA+ MgO nanoparticle displayed a substantial decrease in impact strength in comparison to the control group Table (3). This decrease might be attributed to the

brittleness of MgO nanoparticles and their disability to withstand impact loading [40] and might be related to the aggregation of nano MgO, which resulted in the creation of weakly connected clustersand increased the concentration of the stress surrounding the agglomerated nanoparticles ,causing the contacts at the interface to break down and de-bond between nanoparticles powder and the auto polymerizing acrylic resin. The results would be a rapid crak propagation [24].The result of the current study was in agreement with Shakir[38] and Gad et al. [8].However, the result of the present study disagree with Vikram and Chander [1]. The current study, on the other hand, found that the addition of MgO nanoparticles at concentrations of 2wt.% and 4wt.% had no significant effect or improvement on the impact strength Table (4). This might be because agglomerated mass which cannot provide mechanical property enhancement.Instead, they behave as micrometer-sized defects in the matrix, frequently causing property degradation[37]. The results obtained from this study showed that the nanoparticle concentration had a significant effect on the mechanical strength of acrylic resin.This is supported by Ghaffari et al. [19] Study that shown a decrease in strength as the concentration of NP increased. Furthermore, several research have found an unfavorable association between increasing NP concentration and strength [39].In this investigation, a satisfactory match of repair resin with denture base resin has beennoticedfor all the groups tested, with the exception of a tiny and insignificant discoloration detected with high concentrations of MgO, which did not demonstrate any aesthetic concern.

## Conclusions:

The following findings might be derived within the constraints of the current study: The control group manifested a higher value in flexural strength and impact strength. The incorporation of 2wt.% MgO nanofillers into repaired PMMA increased the value in transverse strength in comparison to that of 4wt. % MgO nano fillers. The incorporation of 2wt.% and 4wt.% of MgO nano fillers into repaired PMMA based resin had no effect on flexural strength or impact strength in comparison to control group.

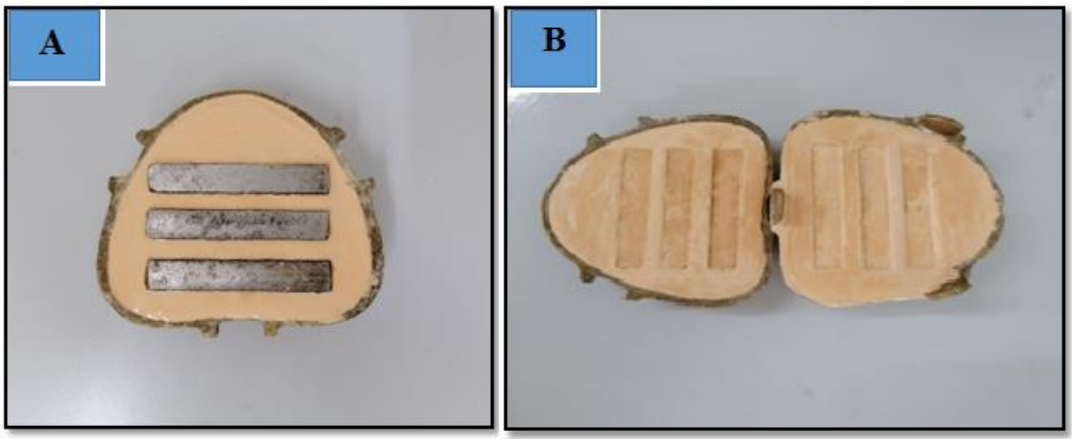


Fig.(1): A:Metal pattern flasking for flexural strength testing, and B: Prepared flexural strength patterns for packing process

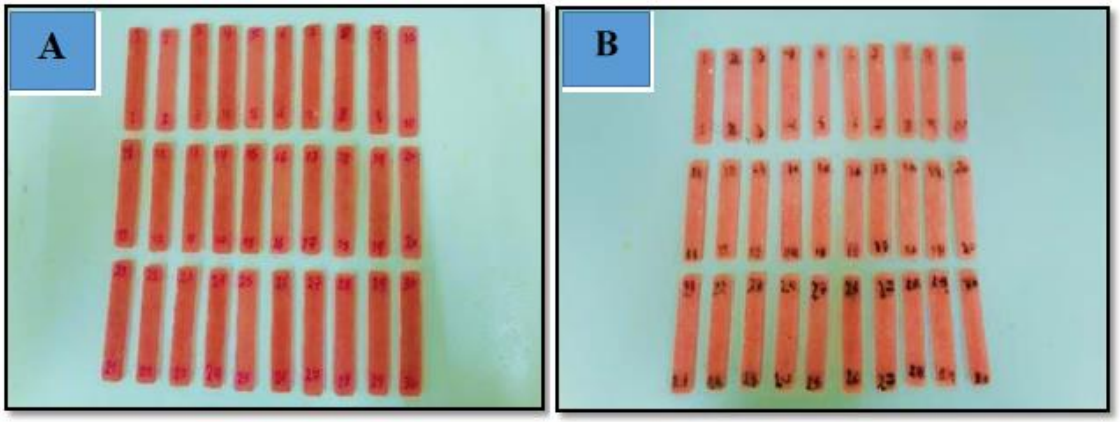


Fig. (2): A: Finished specimen for flexural strength test, and B: Finished specimens for impact strength test.

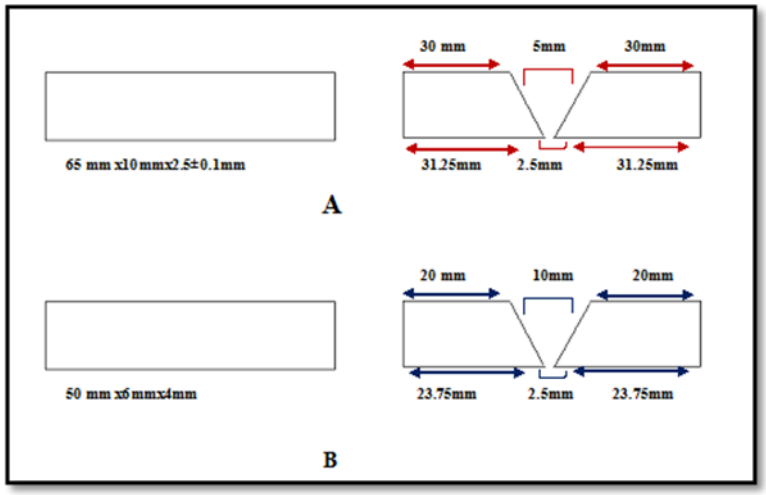


Fig. (3): Diagram illustrated specimen's dimensions as A: for flexural strength test, whereas B: Specimen for impact strength test with fracture the specimens into two pieces with bevel joint for A and B.

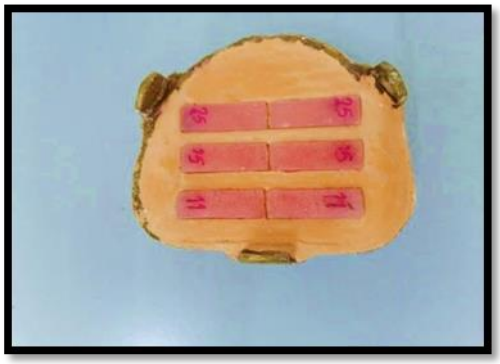

Fig. (4): Two parts of acrylic specimens fixed into the mold.

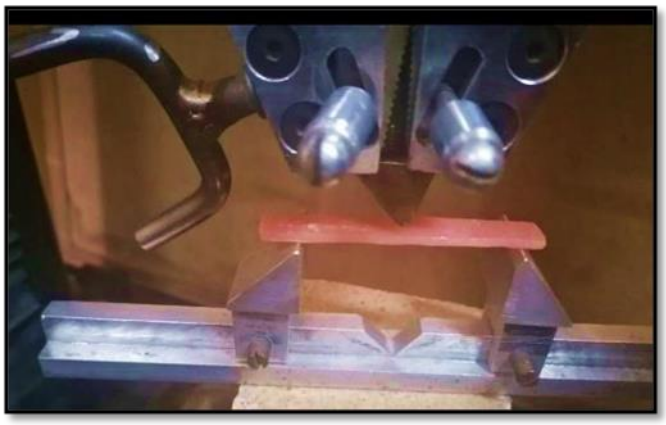

Fig.( 5): The acrylic specimen under the test of flexural strength.

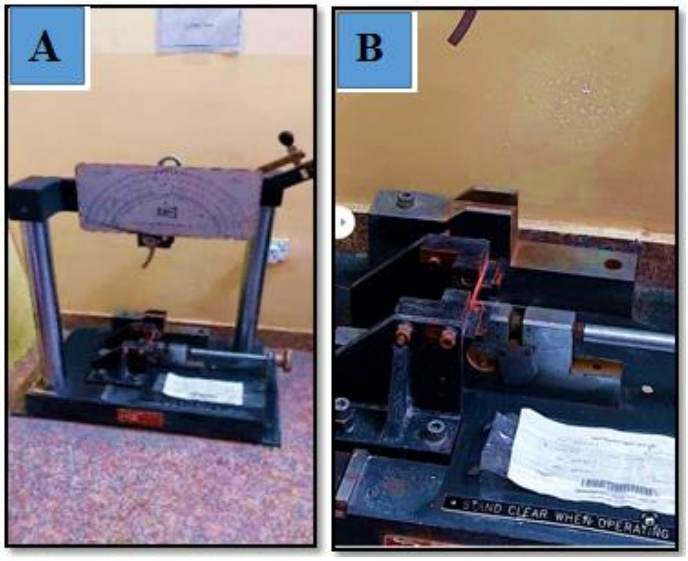


Fig.( 6): A,Charpy-type impact test machine; and B, The acrylic specimen under the test of impact strength

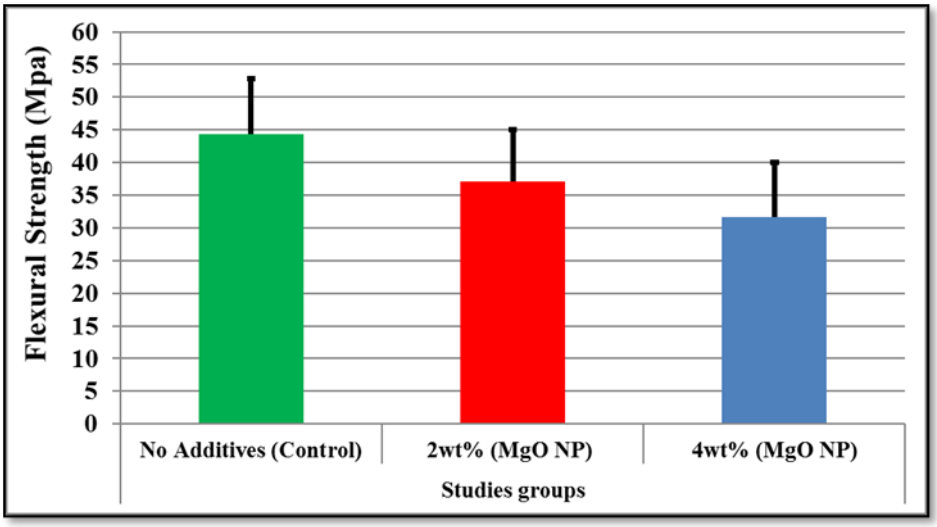


Fig.( 7): Bar chart showing the mean distribution of flexural strength test for all repaired groups.

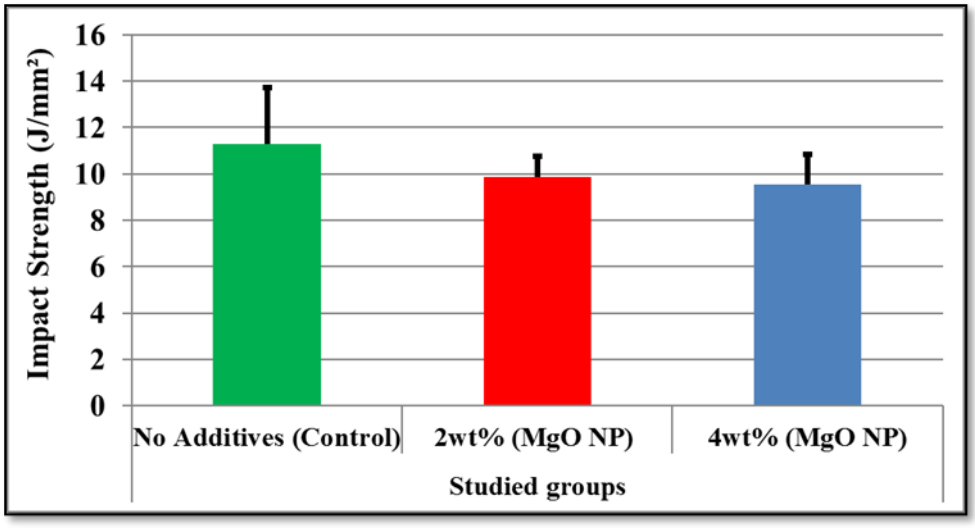


Fig.( 8):Bar chart showing the mean distribution of impact strength test for all repaired groups

Table (1): Descriptive Statistics of flexural strength test for all repaired groups (Mpa).

| Groups | N | Mean | Std. Deviation |
|---|---|---|---|
| **Autopolymerized PMMA-No Additives (Control)** | 10 | 44.3415 | 8.46780 |
| **Autopolymerized PMMA+2wt.% (MgO NP)** | 10 | 37.0385 | 8.03474 |
| **Autopolymerized PMMA+4wt.% (MgO NP)** | 10 | 31.6080 | 8.44786 |
| **Total** | 30 | 37.6627 | 9.48084 |

Table (2): Multiple comparisons using Tukey HSD test between mean values of flexural strength test (Mpa).

| (I) Groups | (J) Groups | Mean Difference (I-J) | Std. Error | P-Value | Sig. |
|---|---|---|---|---|---|
| **Autopolymerized PMMA-No Additives (Control** | Autopolymerized PMMA+2wt.% (MgO NP) | 7.3030 | 4.80309 | .310 | NS |
| | Autopolymerized PMMA+4wt.% (MgO NP) | 12.7335* | 4.80309 | .045 | S |
| **Autopolymerized PMMA+2wt.% (MgO NP)** | Autopolymerized PMMA+4wt.% (MgO NP) | 5.4305 | 4.80309 | .511 | NS |

*. The mean difference is significant at the .05 level

Table (3): Descriptive statistics of impact strength test for all repaired groups ($KJ/m^2$).

| Groups | N | Mean | Std. Deviation |
|---|---|---|---|
| **Autopolymerized PMMA-No Additives (Control)** | 10 | 11.3033 | 2.40174 |
| **Autopolymerized PMMA+2wt % (MgO NP)** | 10 | 9.8767 | .87012 |
| **Autopolymerized PMMA+4wt % (MgO NP)** | 10 | 9.5483 | 1.30392 |
| Total | 30 | 10.2428 | 1.74178 |

Table( 4): Multiple comparisons using Tukey HSD test between mean values of impact strength test ($KJ/m^2$).

| (I) Groups | (J) Groups | Mean Difference (I-J) | Std. Error | P-Value | Sig. |
|---|---|---|---|---|---|
| **Autopolymerized PMMA-No Additives (Control)** | Autopolymerized PMMA+2wt. % (MgO NP) | 1.4267 | .95601 | .322 | NS |
| | Autopolymerized PMMA+4wt. % (MgO NP) | 1.7550 | .95601 | .192 | NS |
| **Autopolymerized PMMA + 2wt.% (MgO NP)** | Autopolymerized PMMA+4wt. % (MgO NP) | .3283 | .95601 | .937 | NS |

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