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Comparison Study on the Influence of Al_2O_3 Nanoparticle Size and Ternary Hybrid on Several Properties of a PMMA Denture Composite

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Abstract:*In esthetic restorative dentistry, resin composites are generally encountered. Because of their excellent biocompatibility, lack of taste, odour, tissue discomfort, and toxicity, insolubility in body fluids, ease of use, good clinical/ aesthetic properties, stable colours, optical characteristics, efficient pigmentation, low cost, and repairability, they are becoming more common. Currently used composite resins have some flaws, particularly inconsistent mechanical properties. For a long time, particles of various sizes have been used as fillers to enhance these properties. In prosthodontics, poly(methyl methacrylate) (PMMA) is commonly used as a dental base material. It does, nevertheless, have several weaknesses, including low strength and weak thermal properties. The effects of different sizes of alumina (*Al_2O_3*) particles on the poly(methyl ethacrylate) (PMMA) denture base were determined in this experiment. PMMA powder was blended with *Al_2O_3* added to methyl methacrylate (MMA).For PMMA polymeric materials strengthened with *Al_2O_3* (40 nm, 140 nm and 0.3-0.8* μm*) in various concentrations, the mechanical properties were studied.Also, the synergetic influence of the three nanoparticle sizes in the resin matrix was studied.Furthermore, thermogravimetric (TGA) analysis and water absorption and solubility characteristics of the composites prepared were also examined. Among the most significant properties to be obtained for good denture materials is high mechanic properties, water absorption and solubility, since they have different attributes in the aqueous condition of the oral cavity. Results showed clear properties optimization, especially for the size-hybrid alumina composites.*

Keywords*:PMMA denture base, nanoparticles, mechanical properties, alumina*

Dental decay normally destroys the enamel and dentine of the tooth. Denture function and structure are adversely affected by damage, but dental interventions may recover or even restore them. Restorative dental materials are being used for clinical treatment and reconstruction of damaged teeth, and the recovery of their functions. The dental teeth aesthetic development is based on dental resin and dental ceramic [1,2]. Bite related forces are applied to the occlusal part of the tooth during mastication. These forces involve the vertical and lingual side forces of compression [1].

While an optimal material is still yet to be developed [3], the most commonly used denture material is acrylic resin [4]. Since their development in the 1950s, polymer composites have been used predominantly in dentistry [5]. Polymer biomaterials are used to substitute metallic materials (amalgam), because of their additional advantages, such as lightweight and adaptable characteristics [6].

Polymethyl methacrylate (PMMA) seems to be the most generally used polymer as a denture base material due to its numerous advantages [7], such as ease of preparation, including finishing and polishing, biological compatibility, aesthetic appeal, optical characteristics [8], good oral stability, and low cost [9]. PMMA also does not necessitate costly equipment [10] and is non-toxic with an efficient pigmentation [11].

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The main disadvantage is that the pure PMMA denture basis mechanical properties do not comply with the appropriate manufacturing standard [12]. It is a resin with insufficient ductility [13], exhibits poor strength [8], low flexural strength, low resistance impact [14], ageing tendency [15], insufficient surface stiffness, and weak antibacterial properties [16], all of which can result in mechanical failure.

Composites and nano-composites as dental polymers have recently been developed [17]. With the integration of nanotechnology and engineering abilities in the dentistry field, better materials for dental applications have been developed [18].

Resin composites are the ideal medium in dental restorative applications due to their excellent biocompatibility, superior aesthetic properties as fillings, lower cost as compared to the ceramic materials, and good bonding potential to the tooth structure [19].

The majority of dental composites are made up of a polymer phase matrix and a dispersedphaseof inorganic fillers [20].The organic matrix is usually composed of polymethacrylates, epoxy andpolyethene [21, 22].Fillers are usually fabricated to respect diverse compositions, shapes and also size distributions, examples being glass or ceramic particles, fibres, nanotubes etc. Their role is to strengthen the polymer or polymer-system properties [5], which lead to aesthetic, optical and mechanical improvements. Also, nanoparticles have proven to enhance the dental material's resistance to wear and improve fatigue [23].

Due to the increased active surface of the nanoparticles that lead to high surface energy at the filler-matrix interface, these fillers can improve flexural strength. A significant increase in impact strength of an acrylic resin was observed after adding to the resin a mixture of 1% of ZrO_2 and TiO_2, same concentration [24]. In another valuable study, the authors studied the inclusion of particles with enhanced rigidity in PMMA resins, which in turn resulted in increased wear resistance [25].

The mechanical characteristics of particle-polymer composites are affected by their particle size parameter [5]. Topouzi noted that, with the inclusion of 0.25 wt.% of SiO_2 nanoparticles of average size 12 nm, the fracture hardness of PMMA resin in provisional restoration dramatically improved [26]. In another study, the addition of nano-sized ZrO_2 particles, the authors concluded, have greatly improved PMMA denture base fracture resistance [27].

In addition to particle size, important in a PMMA resin composite, the choice of their quantity in the resin matrix is of great interest, also. In the distinct works the authors showed that flexural strength has increased at filler concentration of 7 wt.%, in an experiment that evaluated mechanical properties of PMMA reinforced with low concentrations of zirconia nanoparticles (1.5 wt.% up to 7 wt.%, and sizes ranging in the 2-15 nm interval) [27].

According to researchers worldwide, the addition of Al_2O_3 particles will lead to the strengthening of the mechanical properties of polymers.The addition of <10μm Al_2O_3particles in dental composites by 50 wt.% increased flexural performance by over 100% [28].Safarabadi and colleagues used in their research a mixture of hydroxyapatite (HA) and aluminium oxide (Al_2O_3) nanoparticles to be incorporated in a PMMA dental materia l[29]. They concluded that a hybrid filler consisting of 10 wt.% HA and 6 wt.% Al_2O_3 showed the maximum flexural strength.

For this study, the authors chose to opt for the use of threealuminium oxide nanoparticle sizes, 40 nm, 150 nm and 0.3-0.8 µm, and observe their influence on PMMA resin properties.From our knowledge, the influence of different sizes of Al_2O_3 nanoparticles, and the use of all of them in a ternary hybrid, on the mechanical properties of the considered resin system has not been discussed.

2. Materials and methods

2.1. Materials

Polymethyl methacrylate resin (PMMA, *Prod. No. 182265*) was used as a composite matrix. Benzoyl peroxide (BPO, *Prod.No.513474*), methylmethacrylate (MMA, *Prod. No. M55909*) and ethylene glycol dimethacrylate (EGDMA, *Prod. No. 335681*) were used in the acrylic mixing. The mentioned substances were acquired from Sigma Aldrich. The alumina nanoparticles were in nanopowder form (α-Al_2O_3, Skyspring Nanomaterials Inc.) with different sizes (40 nm – *Prod. No. 1319NH*, 150 nm – *Prod. No.*

*1317NH*and 0.3-0.8 µm – *Prod.No. 1321DL*).

2.2. Manufacturing of nanocomposites

In the first step, the nanoparticles were mechanically mixed with the monomer solution (MMA and EGDMA, 9:1 ratio), for 1 h. Afterwards, PMMA and BPO powders were mixed with the monomer solution with a 2.5:1 ratio of powder to liquid. When the prepared solutions were viscous enough, the mixtures were transferred in desired moulds under temperatures gradually ranging from 70°C to 100°C for a total of 2 h, which was the heat polymerization step. Mixing was conducted in a light-enclosed environment. The summary of the compositions of the studied materials ispresented in Table 1.

Table 1. Material compositions for the studies of this work

Resin group	Code	Fillertype /size [nm]	Fillerconcentration [wt.%]
I.	P_0	-	-
II.	PA_40_6	Al_2O_3 / 40	6
	PA_150_6	Al_2O_3 / 150	6
	PA_500_6	Al_2O_3 / 500	6
III.*	P_1	Al_2O_3 / 40 / 150 / 500	1
	P_6	Al_2O_3 / 40 / 150 / 500	6
	P_11	Al_2O_3 / 40 / 150 / 500	11
	P_16	Al_2O_3 / 40 / 150 / 500	16

**Note: All hybrid filler ratios are 1:1:1 by weight percentage*

In the case of the highest dimension alumina powder, for the sake of clarity, they will be noted as 500 nm. To support this statement regarding the choice of this name code, this powder was subjected to dimensional analysis by the DLS (Dynamic Light Scattering) method (DynaPro Plate Reader III, Wyatt Technology, USA). The result was an intensity size distribution, which resulted in a Z-Average Size of 0.523 microns, allowing us to namethe powder, converted to nanometers, as 500.

Three resin groups were prepared, which the first group is the unmodified PMMA resin (control group). The other two groups were divided into distinct subgroups. For group II, the subgroups are depending on the size of nanoparticles, whilst their concentrations do not differ, being set at 6 wt.%. In the case of group III, in the composition of these materials, the quantity of nanoparticles differs but also their concentration. However, in these subgroups, a mass ratio between the types of nanoparticles of 1:1:1 was fixed. Nevertheless, for all subgroups, a number of 5 samples were manufactured.

To reach dispersion levels, the filling volume fraction was low. As a remark, it is well established that inorganic fillers show good hydrophilicity, very high specific surface area and active chemical properties, which in response lead to an agglomeration tendency when mixed in high quantities [26]. As a response, the choice for these concentrations was with respect to agglomeration avoidance and the others researchers experience.

Following polymerization, all composite samples were surface polished with a polishing unit (Struers DPU-10, USA) with fine abrasive paper (500, 1000 and 1200), as declared in ISO 20795-1 standard.

2.3.Experimental procedures

Mechanical properties were determined with a universal testing machine-Instron 1122, USA.Fracture toughness tests were evaluated with respect to ISO 13586 standard, consisting of creating a fine and sharp crack on the notch with a scalpel blade. The sample size was kept at a notch length of 4 mm and 80 mm/ 10 mm/ 4mm. The displacement rate was 1 mm/min and a total of 10 specimens were tested from each composite type. Flexural strength testing was done following ISO 20795-1 standard. Sample size was 64 mm/ 10 mm/ 3.3 mm. The set crosshead speed was 5 mm/min. A total of 10 specimens were tested from each type. Hardness tests have been used effectively as an indirect means of measuring resin-based polymerization depth. In this study, the Vickers microhardness technique was respected. All samples were indented with a square-based pyramid indenter and load application of 300 g for 10 s.

Average hardness measurements were presented. Microhardness procedures were done with respect to the ASTM E384-89 standard.The thermogravimetric analysis (TGA) was used to observe the thermal stability of the obtained composites. A TGA 6 (Perkin Elmer, USA) instrument was used to determine thermal stability, with 10°C/min heating rate, in an inert gas (nitrogen) and a measuring interval from 0°C to 600°C. Water absorption of the composites and the calculations were determined according to ISO 1567 standard. Firstly, specimens were dried at 37°C for 24 h and then weighted to find the sample dry mass. Afterwards, the samples were immersed in pure water (distilled) for upto 28 days and weighed again to determine the sample wet mass. Water solubility tests and the calculations of weight difference were performed with respect to the standard ISO 1567, also.

3. Results and discussions

Loading (concentration), type and size of fillers are important criteria for improving the mechanical properties of dental composites.

Fracture toughness is a basic characteristic of material for determining the quality of the material if a crack occurs. Such fractures exist in brittle structures that cannot bend plastically or redistribute stresses. It is a great instrument in analysing material behaviour when cracks are intentionally generated. Flaws and cracks will typically develop spontaneously or nuclearly after a period of operation and rapid fractures can occur under mild mechanical stresses[5].

Figure 1 shows the fracture toughness of the studied composites. The control materials showed a fracture toughness value of 1.77 MPa $m^{1/2}$. After the addition of alumina particles in the resin structure, fracture toughness increases, regardless of size, type or concentration, when compared with the control group. In the case of the neat resin, high stress is concentrated in front of the notch. Composites of well-distributed nanoparticles have a more linear stress distribution, which improves toughness.

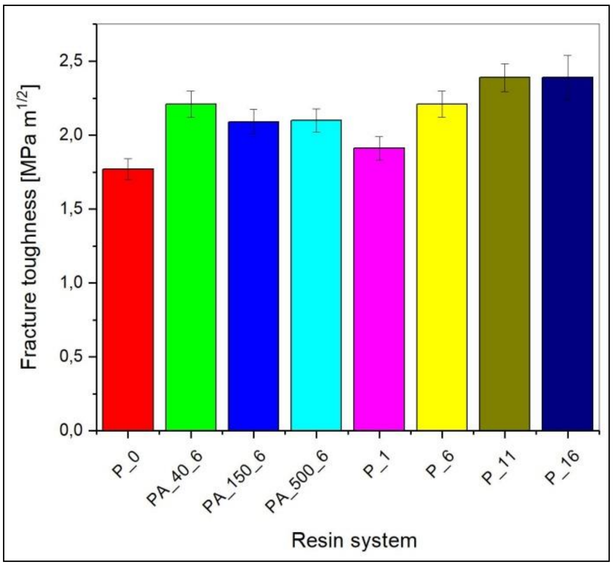

Figure 1. Fracture toughness of the studied samples

In group II composites, it is observed that the mechanical properties of particle–polymer composites are significantly influenced by particle size. It is observed that the 40 nm Al_2O_3-blended resin reached superior values, 2.21 MPa $m^{1/2}$, compared with the materials with higher particle sizes (150 nm and 500 nm alumina, 2.09 MPa $m^{1/2}$and 1.93 MPa $m^{1/2}$, respectively). It is possible that, due to the highest active surface area of the smallest nanoparticles, better interaction with the polymer chains can be obtained, and due to the smaller size, a higher number of interactions in the polymeric structure can be realized.

According to the literature [5, 30, 31], the addition of high-strength fillers is responsible for the increase in fracture toughness. Furthermore, toughening processes such as crack pinning/bowing, crack deflection, matrix–filler interactions, and crack bridging improved resistance to crack propagation.

In the case of group III composites, fracture toughness increased with increasing filler loading.The highest fracture toughness were observed for the resin systems with the highest concentrations of nanomaterial (P_11 with 2.35 MPa $m^{1/2}$ and P_16 with a value of 2.36 MPa $m^{1/2}$).Since all type-sizes of particles are blended into the PMMA resin, it is possible that the synergetic properties of each type could be efficiently integrated into the final resin composite mechanical property. As an observation, the latter composites, with the highest filler concentrations, show very close values. This could mean that higher concentrations, which are not debated in this study, could lead to a regression in resin composite properties, especially when discussing fracture toughness. According to Topouzi, this tendency (i.e., reduction in fracture toughness) may be attributed to excessive filler dispersion in the PMMA matrix, resulting in the agglomeration and the formation of microcracks [26].

The incorporation of low content of large and small particles can increase fracture toughness. Flexural strength can be improved when ceramic fillers are added to dental composites [32].

The flexural test results of the manufactured compositesare shown in Figure 2. At first, it is observed that the neat resin has the lowest flexural strength, with a mean value of 96.74 MPa. For the composites from group II, the flexural strength is inversely proportional to particle size. The material with the smallest particles reached the highest results, 155.64 MPa. Further increasing size will inevitably lead to weaker flexural strength. Smaller particle sizes result in more particles sharing the applied stress in a given tested area.

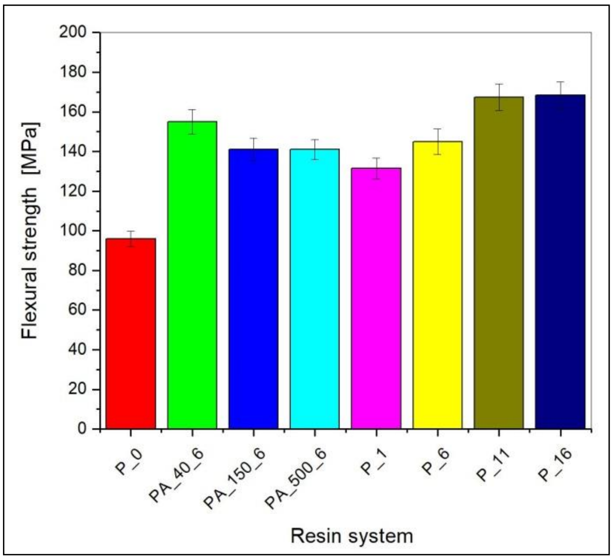

Figure 2. Flexural strength of the studied samples

In the case of 3rd group materials, flexural strength values are directly proportional to added filler concentration, reaching a peak value of 168.45 MPa (P_16). Again, it is observed that for the highest filler loading resin composites, values are comparable, meaning that a possible performance threshold is reached near the 16 wt.% filler concentration. Further increasing the amount of fillers could cause a decrease in flexural strength, which could be explained by the agglomerating tendency of filler particles, especially for the nanoparticles that could lead to faster initiation of sample failure.

Uniformly dispersed nanoparticles inhibit crack proliferation and increase flexural strength substantially. Flexural strength is improved due to the increased surface area of filler particles due to decreased particle size, which results in high surface energy at the filler-matrix interface.

Flexural modulusvaluesshow a higher difference between the materials from the 2nd group, whereas, in the case of 3rd group composites, the difference is negligible. Figure 3 displays the results of the flexural modulus of the analysed resin composites.

The integration of Al_2O_3 particles into dental composites increases flexural modulus. When compared with the neat resin, in this study, the flexural modulus of PMMA composites increases by

61.49 % for the material with the lowest nanoparticle size, leading to an increase of only 19.78 % in the case of the largest particle size composite. On the other side, in group III, the highest increase was 75.4 % for the highest filler hybrid concentration.

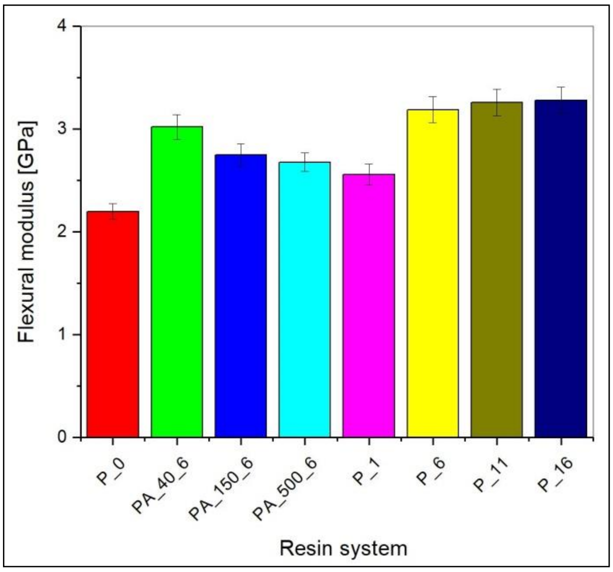

Figure 3. Flexural modulus of the studied samples

The increase in flexural modulus is due to the incorporation of inorganic material with a rigid structure, which enables the polymer matrix to migrate strain to the stiffer particles while also limiting polymer mobility due to matrix-particle interactions, transforming the overall material to a stiffer state.

Filler particles in the resin matrix improve mechanical properties such as hardness [33]. It is observed in Figure 4 that, regardless of the group discussed, the hardness increase is very straightforward. The neat composite has the lowest microhardness, 22.07 MPa. The highest microhardness is achieved in group III, with an increase of 90.90 % (P_16).

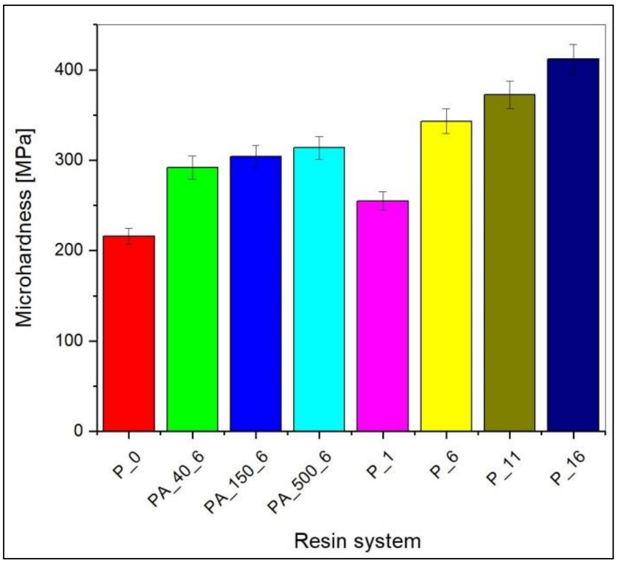

Figure 4. Microhardness of the studied samples

Since rigid inorganic particles have significantly higher stiffness than polymer matrices, inserting micro- or nanoparticles improves hardness efficiently. At higher concentrations, microparticles increased hardness more than nanoparticles.

Also, increasing filler loading will lead to increased hardness values, observations discussed by other authors, also [34, 35]. A uniform nanoparticle dispersionprovides sufficient distances between the particles, enhancing composite strength and stiffness [36]. As a result, filler particle size and filler content will influence the hardness of dental composites.

Figure 5 shows the thermogravimetric curves of the PMMA composites. The highest degradation temperature was found in microcomposites, followed by nanocomposites, and the lowest temperature was found in pure PMMA. PMMA-Al_2O_3 composites are more thermally stable than control resin. The TGA curves indicated no noticeable weight loss up to a temperature of 200°C. Further increasing the temperature showed an abrupt decrease in weight. Complete weight loss was achieved for the neat resin at approximately 400°C. Different thermal degradation behaviours were found in the samples. The degradation temperature of the group II and group III samples was significantly higher than that of the PMMA matrix.

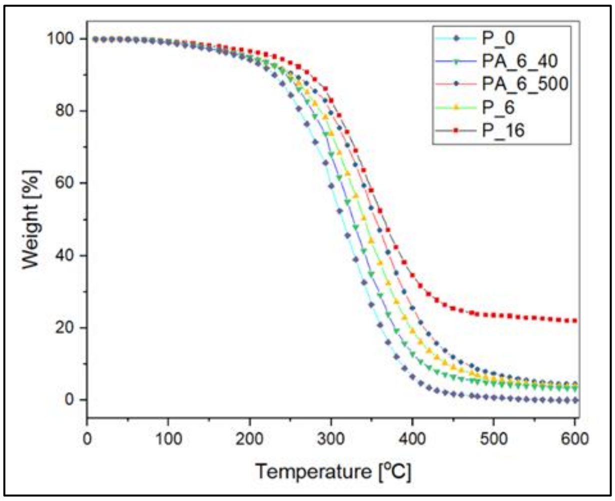

Figure 5. TGA curves for control resin and the resins with 40 nm and 500 nm alumina, 6 wt.% and 16 wt.% hybrids

Furthermore, no residual material was detected in the PMMA sample at temperatures up to 450°C. This activity is representative of the random chain scission processes that arise during the degradation of PMMA. Thermal behaviour of group II and group III composites samples were comparable, the exception being the composite with 16 wt.% Al_2O_3 size-hybrid. However, some variations were observed; microcomposites exhibited a significantly higher degradation temperature than nano-composites, suggesting microcomposites' high thermal stability. Over 300°C, the degradation rate increased exponentially. End groups have weaker bonding chains than polymer chain structures and begin to decay at about 250°C. PMMA degrades by random scission at temperatures above 300°C, and the depolymerisation rate increases due to main chain scission. As the temperature rises above 300°C, the PMMA degrades due to random scission, the depolymerisation rate increases due to main chain scission, and the entire polymer unzips entirely.

Water absorption and water solubility were higher for the alumina-based composite materials than those in the control group. Table 2 shows the values for water absorption and solubility analysis.

Acrylic resin can have good mechanical and physical properties, as well as water absorption and solubility. Water functions as a plasticizer, inducing dimensional changes and lowering the mechanical properties of the composites, such as hardness and fatigue limit.

The composites in this experiment have significantly higher water absorption and solubility values than the control group.

Table 2. Water absorption and water solubility tests for the studied samples

Resin system	Water absorption[μg/mm^3]	Water solubility[μg/mm^3]
P_0	15.01 ± 0.61	1.02 ± 0.04
PA_40_6	17.68 ± 0.74	1.21 ± 0.05
PA_500_6	15.33 ± 0.61	1.12 ± 0.04
P_6	21.02 ± 0.84	1.22 ± 0.05
P_16	37.26 ± 1.49	1.72 ± 0.07

This discovery can be due to the fact that water absorption and solubility are caused by the presence of soluble impurities, filler particle size and distribution, interfacial properties between the filler and the resin matrix, and composite homogeneity. The majority of water molecules share the free volume between the chains and crosslinks and microvoids or concentrate at the filler–polymer interface. As a consequence, water absorption will stimulate filler–matrix break-down and debonding. The plasticizing effect of water molecules often promotes hydrolytic deterioration of the matrix and filler–matrix interface, resulting in a decrease in mechanical properties. The solubility of resin composites is influenced by the filler form, filler processing, and the presence of air voids, which reduce material content. These findings are confirmed by these analyses that found the composite groups' water absorption levels to be greater than the control group's.

4. Conclusions

Within the limitations of the present study, the following conclusions can be drawn. The composite resins were manufactured by the addition of alumina particles, of different sizes and concentrations, to a PMMA denture resin. The addition of alumina particles to PMMA resin adversely affected the mechanical properties of the materials for 1 wt.%, 6 wt.%, 11wt.%and 16 wt.% and sizes of 40 nm, 150 nm and 500 nm. It has been found that the addition of smaller particle sizes shows better mechanical properties, but lower resistance to water adsorption and solubility. Concomitantly, for the ternary blended alumina particle composites, group III, mechanical properties show a direct trend with concentration but showed weaker water resistance properties. The size of filler particles has a significant impact on the fracture resistance of polymer dental composites. In addition to other considerations, this property of dental composites can be increased by adding nanoparticle fillers at low concentrations. Based on the present study results, when discussing an optimal particles size should be as small as possible, proven by the used nanoparticles of 40 nm in diameter. When discussing alumina size-hybrid, an optimal concentration appears to be the second-biggest concentration, 11 wt.%. This can be explained by the very similar fracture toughness, flexural strength and flexural modulus with the highest concentration composite, 16 wt.% Al_2O_3.

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