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Original Article

A comparison of the effect of different forms of Graphene and Polyvinylpyrrolidone on physically strengthening PMMA

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Abstract

Aim: The aim of this study was to compare the effects of powder, mat, and sheet forms of graphene and polyvinylpyrrolidone (PVP) on the bending strength of polymethyl methacrylate (PMMA).

Methodology: A total of 75 PMMA samples (64 x 13 x 3 mm) were obtained and separated into five groups of 15, one of which was the control group. No strengthening material was added to the control group samples. In the PMMA+Polyvinylpyrrolidone powder (PVPP) (Group B) and PMMA+Graphene powder (GP) groups (Group D), PVP (0.5%) and graphene (0.01%) powders, respectively were added to the PMMA. In the PMMA+Polyvinylpryrolidone fiber mat (PVPM) (Group C) and PMMA+Graphene sheet (GS) groups (Group E), a PVP mat and graphene sheet, respectively were placed in the center of a square PMMA resin mold. To measure the samples' resistance to bending, the three-point bending test (ISO 1567) was applied. Fracture lines were examined under a scanning electron microscope (SEM), and bending strength was analyzed using one-way analysis of variance (ANOVA).

Results: A statistically significant difference was determined between the GS (118.5093 ± 11.94859 Mpa) and PVPP (105.5267 ± 6.80595 Mpa) groups. The mean bending resistance values were numerically higher in the PVPM (115.8487 ± 17.15440), GS (118.5093 ± 11.94859), and GP (116.2680 ± 16.03207) groups compared to the control group (108.2407 ± 24.23104). The graphene sheets and PVP mats showed higher resistance to bending than the powder form of either material.

Conclusion: In clinical dental prosthesis use, PVP mats and graphene sheets are preferable with respect to both PMMA strengthening and color.

Keywords: Polyvinylpyrrolidone, PMMA, Graphene sheet, PVP fiber mat, bending test

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Introduction

Polymethyl methacrylate (PMMA) is the most widely used material in the production of dental prostheses due to positive characteristics such as ease of use in the laboratory, low cost, constant stability within the mouth, and being light, aesthetically appropriate, and color-compatible (1) However, PMMA is not accepted as an ideal prothesis material because of its poor mechanical and physical properties (2) Notably, PMMA prostheses may break under repeated dynamic loading or as a result of dislodgement (3). Failure under impact is the most common reason for prosthetic fractures which are time-consuming and costly to fix (4). In a survey study of PMMA fracture types, Darbar et al. reported that 68% of PMMA prostheses fractured within a few years, and of these fractures, 33% were due to the separation of prosthetic teeth from acrylic systems, 29% were midline fractures in upper full prostheses, and 38% originated from other factors (5). Prosthesis fractures, which are generally

seen during use, are thus a problem that still requires a solution (6).

The most commonly used materials for dental provisionalization are autopolymerized poly (methyl methacrylate) (PMMA) resins. PMMA is a glassy and fragile material which demonstrates low fracture toughness (7). Traditionally, metal wires and plates have been used to strengthen denture base materials (8). However, metal strengthening materials do not sufficiently bind with the resin matrix. Moreover, strengthening with metal is not clinically accepted given its low aesthetic properties (6). Researchers have thus attempted to strengthen PMMA with various other materials, such as fibers (glass, aramid, natural fibers, carbon graphite), oxides (aluminum, zirconium, titanium, magnesium), and most recently nanodiamonds (7). Previous studies have shown that of the fibers used, silanized glass fibers are most used for strengthening, having been reported to greatly improve the physical properties of denture base material (9). Recent studies have also suggested that nano filling materials improve the thermal properties and thermal stability of PMMA, as compared to pure PMMA, given their large surface area, small size, and homogenous distribution. However, the exact properties of resin strengthened with nano filling materials depend on the size, shape, type, and concentration of the added particles (9). Polyvinylpyrrolidone (PVP) is one type of biocompatible, hydrophilic, and non-toxic nano filling material (10). It is generally used as a tablet binding material and blood plasma expander but is also an excellent polymer that is already a component in many biomedical applications (11).

Electrospinning is a nano dimension production method that emerged with the observation of fluid behavior under high voltage. As written in early studies, by applying an electrical field at a certain distance, water droplets pour onto a spiral trajectory to form a conical structure. Using this technique for polymer production, the polymer is dispersed within an appropriate solution and melted in a glass syringe. The syringe needle is then pumped with the syringe pump and subjected to high voltage. After the formation of a Taylor cone, nanofibers varying in diameter from 30 nm to 1 µm accumulate in an earthed mound (12).

Researchers have comprehensively examined current graphene-based polymer composites as well for the improvement of electrical and mechanical polymers. The composites' extraordinary electrical (~5000 Wm-1 K-1) and mechanical properties (Young's modulus = ~1 TPa) and broad specific surface area (2630 m^2 g-1) allow them to be used in various engineering applications and as a polymer filling material. Yet, as graphene is extremely expensive, it is generally only used as a filling material in graphene oxide polymer-based materials (13, 14). Di Carlo et al. examined PMMA and graphene-reinforced PMMA (G-PMMA) in respect to the latter's bending resistance and its effect on the elastic modulus, informing that this material could ease and innovate the production of dental prostheses (15).

In the examination of additional materials used to strengthen PMMA, researchers have also investigated parameters such as different added materials (6, 8, 9, 16) different stress areas in the dental prosthesis (17), different upper palate shapes (18), and thermal conduction (19). However, very few studies have examined the effect of the physical properties of different added materials on PMMA's resistance to impact.

The aim of this study was to compare the effects of powder, mat, and sheet forms of graphene and PVP materials on PMMA's bending resistance. The null hypothesis was that different physical forms of the same material do not have the same effect on PMMA's bending resistance.

Materials and Methods

Table 1 presents the materials used in this study. To prepare the PMMA resin, chrome molds (64 mm x 13 mm x 3 mm) were manufactured according to the ASTM D4812 standard (standard test method for impact resistance of unnotched cantilever beam, electrospinning). The midpoint of the chrome molds' height was marked for placement of the graphene sheet (Nanografi nano technology, Jena, Germany) and PVP (Sigma Aldrich, Taufkirchen, Germany) fiber mat in the center of the PMMA resin sample. A total of 75 PMMA samples were prepared in five groups of 15.

The five groups were:

- Group A: PMMA control group (C)
- Group B: PMMA+Polyvinylpyrrolidone powder (PVPP)
- Group C: PMMA+Polyvinylpryrolidone fiber mat (PVPM)
- Group D: PMMA+Graphene powder (GP)
- Group E: PMMA+Graphene sheet (GS)

Table 1. Materials used in the study (PMMA:Polymethylmethacrylate, PVP: Polyvinylpyrrolidone)

Material Name	Manufacturer
PMMA	Meliodent, Kulzer, Germany
Graphene powder	Sigma Aldrich, Germany
Graphene sheet	Nanografi nano technology, Germany
PVP	Sigma Aldrich, Germany
Separator gel	Isolant/D.M.L, Germany

PVP Fiber Mat production

In this study, a fiber mat produced from PVP was used to strengthen PMMA. PVP was selected because it is biocompatible, soluble in water, and is easy to use. In total, 2 gr PVP (C_6H_9NO) were dissolved in 10 ml ethanol and mixed in a magnetic mixer until homogenous. The solution was withdrawn into a 10-ml syringe and placed in the syringe pump in the electrospinning apparatus. The flow rate was fixed and set at 5 ml/h for approximately 2 h until sufficient fibers formed. A Gamma ES30 high voltage power force was used in the electrospinning apparatus, with positive (+) and negative (-) ends attached to the feeding and collection units. The voltage applied in the electrospinning procedure provided movement from the left feeding unit to the collecting unit, and the fibers were thinned in this order.

Preparation of the control group

For the control group, the PMMA resin (Meliodent, Kulzer, Hanau, Germany) was mixed according to the manufacturer's instructions at the polymer/monomer ratio of 34 mg powder/14 ml liquid until it reached a dough-like consistency. To release the samples from the molds more easily, a petrol-based gel (Isolant/D.M.L., Germany) was spread in the molds. The prepared mixture was poured into the molds, which had been lined with glass. To avoid porosity, a cylinder was passed over the top of the mixture, and finally, glass was placed on top and pressure applied. The PMMA resin was then hardened in a polymerization device at 100°C for 30 min under 2 bars of pressure (Polymer 180, Zhermack SpA-Badia Polesine, Italy). Following polymerization, excess material was removed with a tungsten carbide mill at 15,000 rpm, then surface finishing was completed with 200- and 600-grade sandpaper.

Preparation of the PVPM and GS groups

Before mixing the polymer and monomer for the PVPM and GS samples, the graphene sheet was wetted in a monomer for 5 min in a petri dish to better bind the graphene with the PMMA resin. The graphene sheet was removed from the monomer and left at room temperature for 1 h to dry. As with the control group, the PMMA resin was poured into the prepared molds up to the marked midpoint. A fiber mat (64 mm x 13 mm x 10 µm) and graphene sheet (64 mm x 13 mm x 35 µm) were cut to the appropriate size and placed in their respective molds, which were then filled to the top with PMMA resin. The polymerization and finishing processes were the same as with the control group.

Preparation of the PVPP and GP groups

For the PVPP and GP samples, the 0.5% PVP and 0.01% graphene nanoparticles were weighed on electronic sensitive scales (A&D Company, Japan) and mixed with the PMMA matrix. The molds were filled with the PMMA resin mixture, polymerized, and finished per the same procedure as described for the control group.

Three-point bending test

All samples' bending resistance was tested with the three-point bending test (ISO 1567). The samples were placed in a test device with cylindrical supports 3.2 mm in diameter placed at 50-mm intervals (Esetron Mecathronics, Ankara, Turkey). Loading was applied to the samples at 5 mm/min cross speed. Values were recorded in Newtons (N), and bending resistance was calculated using the formula $Fs = 3PL/2\ bd^2$, in which P refers to maximum load, L to sample length, b to sample width, and d to sample thickness. The fractured samples were then examined under a scanning electron microscope (SEM) at x200 and x5000 magnification.

Statistical analysis

The data obtained in this study were statistically analyzed using SPSS V26 software (SPSS Inc., Chicago, IL, USA). According to the Shapiro-Wilk normality test, the data conformed to a normal distribution. Bending strength was analyzed with one-way analysis of variance (ANOVA). The data did not show a homogenous distribution according to the Welch test. To determine differences between groups, paired comparisons were made with the post-hoc test. A value of $p<0.05$ was accepted as statistically significant.

Results

The mean bending strength values of the tested PMMA resin samples are shown in Figure 1 and Table 2. The one-way ANOVA results showed a significant difference between the groups ($p=0.007$, $p<0.05$). According to the post-hoc paired comparison, a statistically significant difference was determined between the GS (118.5093 ± 11.94859 Mpa) and PVPP (105.5267 ± 6.80595 Mpa) groups. No significant difference emerged between the other groups. The mean bending resistance values were numerically higher in the PVPM (115.8487 ± 17.15440 Mpa), GS (118.5093 ± 11.94859 Mpa), and GP (116.2680 ± 16.03207 Mpa) groups compared to the control group (108.2407 ± 24.23104 Mpa). The differences between the GS and GP ($p=0.539$) groups and between the PVPM and PVPP ($p=0.158$) groups in terms of bending were not statistically significant (Kruskal Wallis, $p>0.05$).

SEM images of the reinforcement material in the PVPM and GS groups are shown in Figures 4 and 9, respectively. Figure 5 shows the presence of broken fiber ends in small circles penetrating the PMMA. The graphene sheet's irregular surface appears along the midline in Figure 8. The joining of the gaps between

the PMMA and graphene sheet appear as a gap along the line in Figure 9. As seen in Figures 4 and 8, small but intense fracture lines formed in front of the strengthening material on the side where force was applied.

This did not occur behind the strengthening material. The fractured surfaces in Figures 2 and 3 show a planar fracture model specific to fragile materials.

In Figures 6 and 7, there is no material line in the midline, as is the case with PVP in mat form and Figures 10 and 11 in sheet form of graphene. It is observed that the powder forms of PVP and graphene are completely mixed in PMMA.

Table 2. Sample size, mean, standard deviation values of the samples.

Group	N	Mean	Std. Deviation
Control	15	108,2407[ab]	±24,23104
PVPP	15	105,5267[ab]	±6,80595
PVPM	15	115,8487[a]	±17,15440
GP	15	116,2680[ab]	±16,03207
GS	15	118,5093[b]	±11,94859
TOTAL	75	112,8787[ab]	±16,64377

The superscripts with the same letters in the same column were not significantly different by one-way ANOVA and Post -Hoc multiple range test at p<0.05

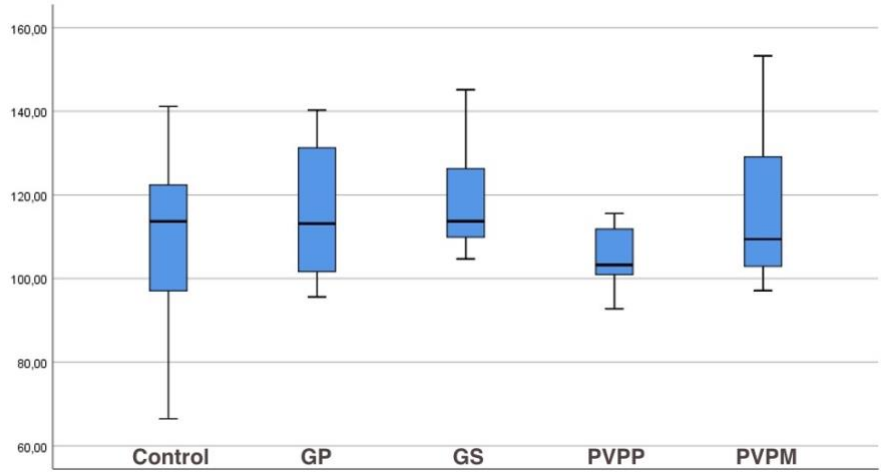

Figure 1. Boxplot graphic of the studied polymethyl methacrylate

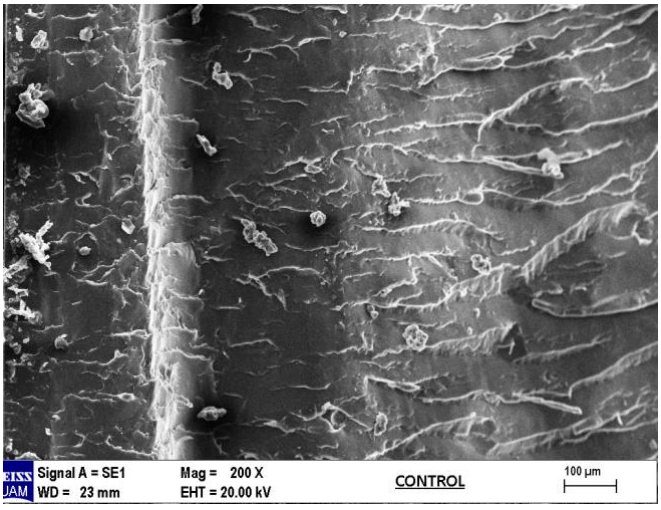

Figure 2. SEM image x200 magnifications for control group,

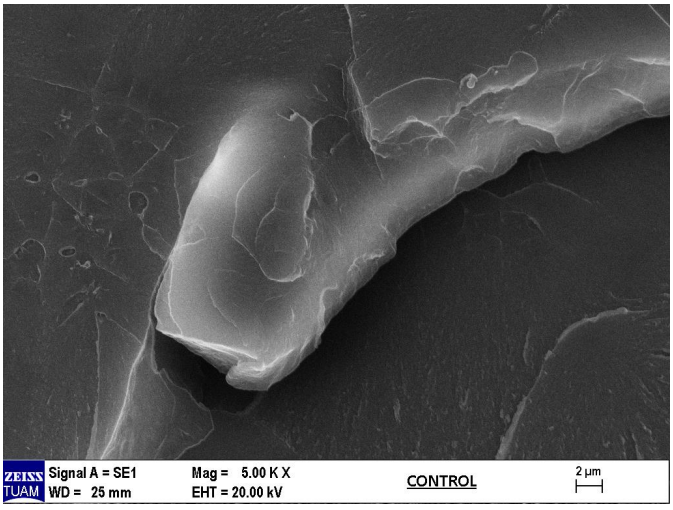

Figure 3. SEM image x5000 magnifications for control group

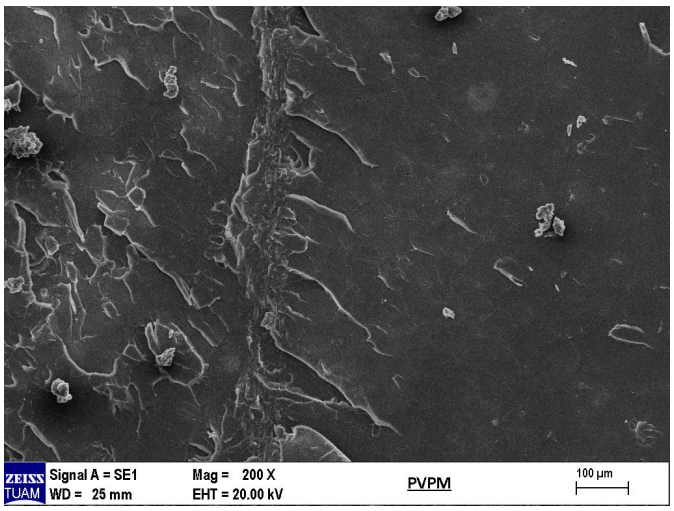

Figure 4. SEM image x200 magnifications for PVPM group

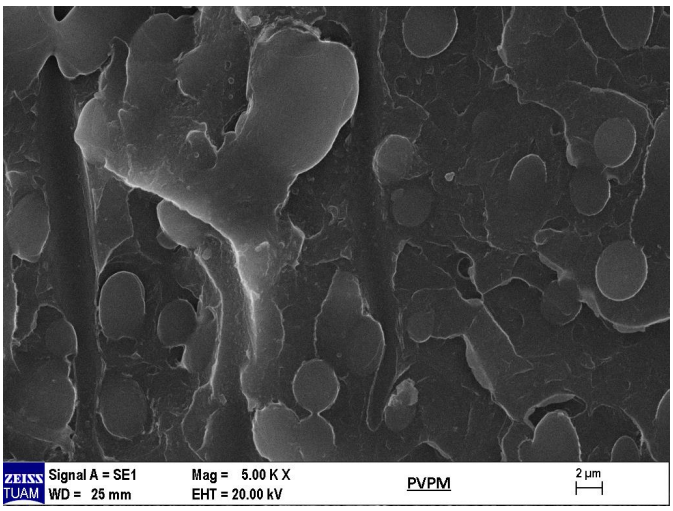

Figure 5. SEM image x5000 magnifications for PVPM group

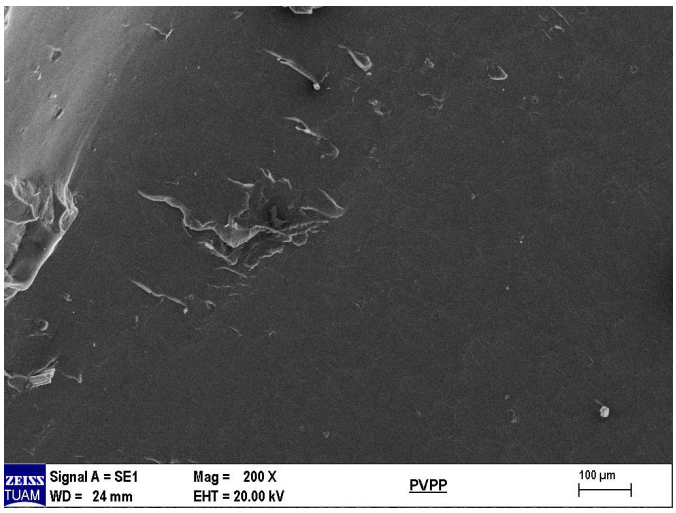

Figure 6. SEM image x200 magnifications for PVPP group

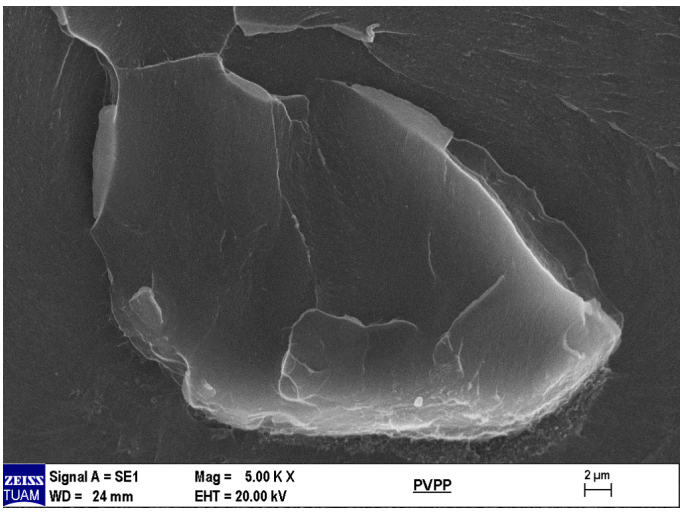

Figure 7. SEM image x5000 magnifications for PVPP group

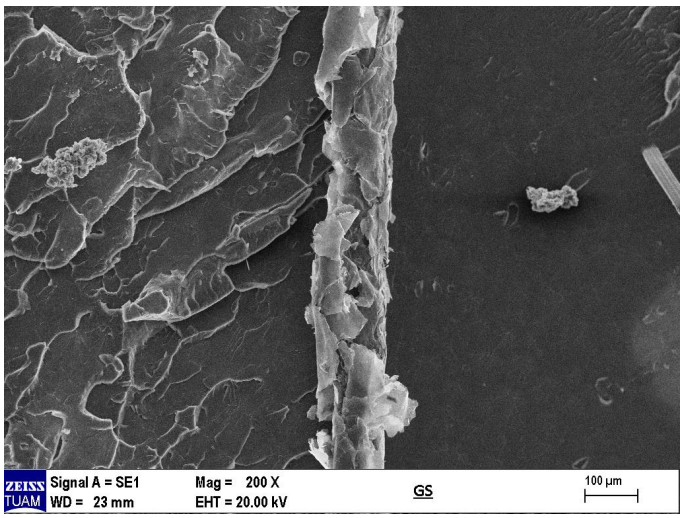

Figure 8. SEM image x200 magnifications for GS group

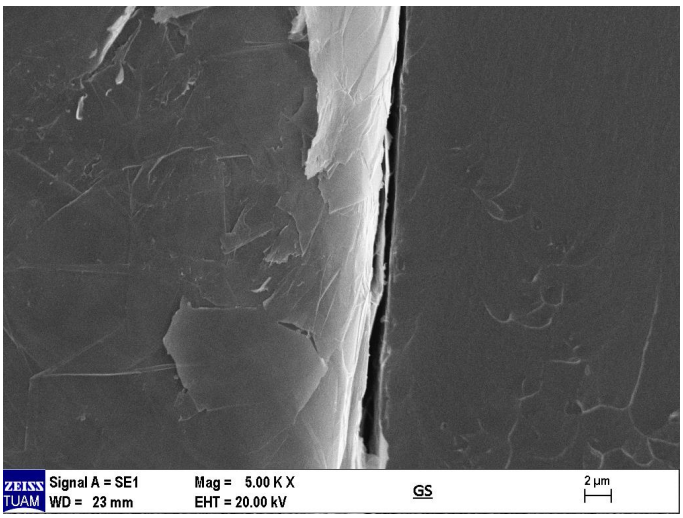

Figure 9. SEM image x5000 magnifications for GS group

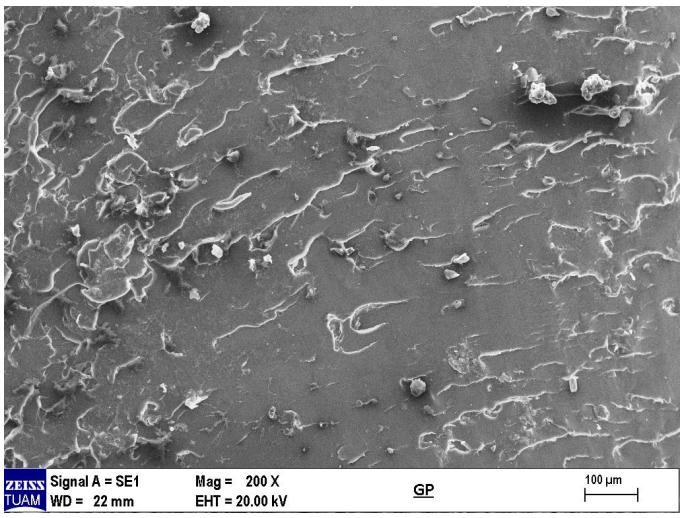

Figure 10. SEM image x200 magnifications for GP group

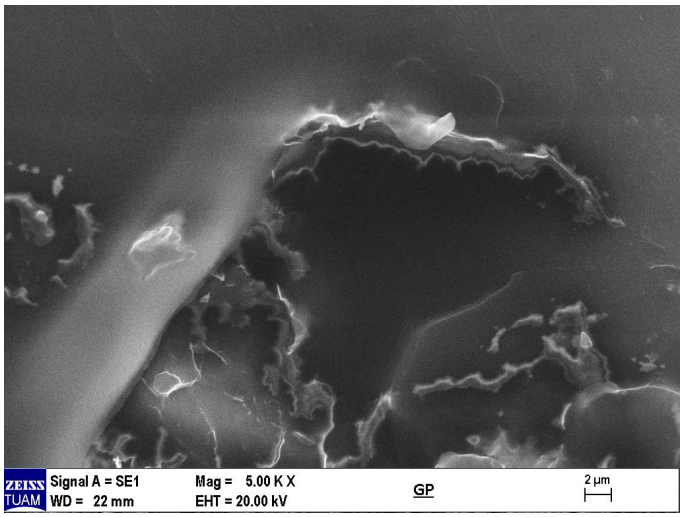

Figure 11. SEM image x5000 magnifications for GP group

Discussion

In this study, PVP nanoparticles, PVP mats, graphene nanoparticles, and graphene sheets were added to heat-polymerized PMMA denture base material, and the effects of the material type and physical form on the PMMA's strengthening were evaluated. The null hypothesis that different physical forms of the same material would not show the same bending resistance effect was rejected, as no statistically significant differences emerged according to the type and physical form of the strengthening material used to enhance the PMMA's bending resistance.

There are different approaches to strengthening the physical properties of PMMA, such as changing the chemical structure of the resin or adding supplementary materials (20-23). Zirconium oxide, glass fibers, titanium oxide, aluminum oxide, polyethylene fibers, and carbon nanotubes are some of these materials. Studies have reported that in addition to the type of strengthening material, various other factors such as the size, shape, concentration, adhesion, and distribution of the filling particles in the polymer matrix, along with strong adhesion in the interface, have important effects on PMMA reinforcement (20, 24-26).

Due to the advantages of nanomaterials such as excellent biocompatibility, toughness, strength, abrasion and corrosion resistance, good esthetics, ability to improve the mechanical properties of acrylic resins, withstand crack propagation; several studies have remarked on their benefits in strengthening PMMA (27). As electrospun PVP nanofiber, in particular has not been previously studied, and there are few studies on fiber mats and graphene, this study examined PVP and graphene as supplementary materials. Studies have examined the same physical form of strengthening material (12, 18, 28) though very few have assessed different physical forms of the same material (29). As such, in the current study, the effects of physical mat and sheet PVP forms and graphene on PMMA reinforcement were examined. In the PVPP and PVPM groups, powder and mat PVP forms were used, and a statistically significant difference was not determined between these groups and the control group.

Electrospinning is a multi-aspect technique for the production of ultrafine fibers. The electrospinning method can be applied to most organic polymers as long as they can be dissolved in appropriate solvents to obtain solutions or melted without decomposition (30). In a study by Uyar et al., a polyvinyl alcohol (PVA) nanofiber mat produced in three different designs with the electrospinning method was combined with PMMA. The researchers reported that nanofibers developed with different designs provided significant improvements to the mechanical properties of PMMA (12). In the current study, there was no statistically significant difference between the PMMA strengthened with a PVP fiber mat (PVPM group) and the control group. However, the PVPM group showed numerically higher bending resistance values than the control group (115.8487 ± 17.15440 vs. 108.2407 ± 24.23104 Mpa).

Several studies have found that zirconia nanoparticles significantly increase the bending resistance of PMMA (19, 31). In a study by Wei Yu et al., zirconia in nanotube form had a greater strengthening effect than that in nanoparticle form, with the polymer chains forming a three-dimensional mesh structure by wrapping around the zirconia nanotubes (29). In the current study, the mat form of PVP and the sheet form of graphene had higher numerical bending resistance values than the particle forms. Per the SEM images, the fibers within the PVP mat better combined with the PMMA than its PVP powder counterpart. This shows that the polymer chains could have formed a three-dimensional mesh structure by wrapping around the PVP fibers within the mat.

The use of graphene as a supplement within nanocomposites is similarly advantageous, as it provides an important development in production methods and has low processing costs. In the current literature, there are few studies that have analyzed the

properties of PMMA-based nanoparticles strengthened with graphene (15). Paz et al. reported that low GO loading on PMMA could improve mechanical performance, such as fatigue resistance and breaking capacity, for as the GO percentage increased in their investigation, the breaking force decreased (32). In the current study, while the powder and sheet forms of graphene showed higher numerical values than the control group, bending resistance was higher in the sheet form than in the powder form. Even a small amount of graphene powder is visible in PMMA and causes a discomforting color change almost to black. However, if a graphene sheet is placed between the PMMA, this causes less discoloration. When considered in this respect, it is easier to place a graphene sheet in the hard palate part of a dental prosthesis where there is less visibility, although there is a high risk of fracture on the denture base.

The observation of a planar fracture model in the control group revealed the fragile structure of PMMA (33). From the multiple fracture planes, together with gaps seen in the GP group, it can be said that graphene powder brings PMMA into a plastic state. Still, the plasticization change in resin polymers may cause the polymer chains to fragment, which can reduce deformation resistance (3). Previous studies have also shown that a rational increase in the ratio of materials used to strengthen PMMA significantly affects the PMMA's properties, but the mechanical resistance of PMMA decreases with increasing concentrations and causes visible color changes (20, 26, 34).

Conclusions

The results of this study demonstrate that the mat form of PVP and the sheet form of graphene strengthen PMMA more than their respective powder forms. That these material forms' polymer chains may create a three-dimensional mesh structure proves a valuable research contribution. There is a need for further studies that examine different polymers, including different PVP mat thicknesses and designs.

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