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Original research

The effects of metal nanoparticles incorporation on the mechanical properties of denture base acrylic resin

Secil Ozkan Ata[1], Canan Akay[2,4,5], Emre Mumcu[3,4,5]

Purpose
The aim of this study was to examine the flexural strength of acrylic resin base material incorporated with iron, copper, and titanium nanoparticles.

Materials and Methods
Seventy bars of samples (65x10x2.5 mm^3) were divided into seven groups. Acrylic samples were prepared according to the manufacturer's instructions. Fe_2O_3, CuO and TiO_2 nanoparticles were manually added in a proportion of 1wt% and 3wt% to the heat-polymerized acrylic resin. The Universal Testing Machine was used for 3-point flexural test of 5 mm/min force. ANOVA and Weibull analyses were used for the statistical analyses.

Results
A statistical difference was found between the nanoparticle-added group and the control group. The highest mean value was observed for the 1wt% TiO_2 added group, (84.99 MPa) and the lowest value was for the 3wt% CuO added group (71.32 MPa) (p<0,001). The 3wt% Fe_2O_3 and CuO added groups showed lower values than the control group.

Conclusion
The incorporation of TiO_2 nanoparticles into acrylic resin in a proportion of 1wt% increased the flexural strength values of the resins. Within the limitations, the nanoparticle addition to acrylic resins could improve the mechanical properties; however, when the percentage of nanoparticle addition increases, the flexural strength values of the acrylic resins decrease.

Keywords: *Acrylic resin, nanoparticle, flexural strength, prosthodontics, weibull analysis*

Introduction

Acrylic resins are commonly used denture base materials. It is preferred especially due to its easy laboratory processes, low weight, reasonable price as well as convenient aesthetic and color matching properties (1,2). However, high thermal expansion coefficient, low thermal conductivity and denture fractions are among the disadvantages of this material (1,3). Especially denture fractions that occur by mistakenly dropping, or fatigue due to recurring stretches caused by chewing activities, are still serious problems (4,5). There are studies focusing on the methods to prevent acrylic resin fractures and improve its mechanical features by reinforcing with different amounts of metal, metal oxide particles, carbon and fibers such as glass aramid (6,7).

Recently, the use of nanoparticles has become increasingly common because of their anti-corrosion features, biocompatibility and resistance to fatigue and rupture. There are some studies in the literature examining the mechanical effects of denture base acrylic resins incorporated

ORCID IDs of the authors: S.O.A. 0000-0003-1756-4390; C.A. 0000-0003-2781-8710; E.M. 0000-0002-3791-6472

[1]Department of Prosthodontics, Faculty of Dentistry, Osmangazi University, Eskisehir, Turkiye

[2]Department of Prosthodontics, Faculty of Dentistry, Osmangazi University, Eskisehir, Turkiye

[3]Department of Prosthodontics, Faculty of Dentistry, Osmangazi University, Eskisehir, Turkiye

[4]Advanced Material Technologies Application and Research Center, Osmangazi University, Eskisehir, Turkiye

[5]Translational Medicine Research and Clinical Center, Osmangazi University, Eskisehir, Turkiye

Corresponding Author: Secil Ozkan Ata

E-mail: secilozkanata@gmail.com

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with nanoparticles such as copper oxide (CuO), iron oxide (Fe_2O_3), zinc oxide (ZnO), titanium dioxide (TiO_2), aluminum oxide (Al_2O_3) and silicon oxide (SiO_2) (7,8,9). According to these studies, the concentration, size, and shape of nanoparticles might affect the endurance and the fracture resistance of materials (8). It was reported that adding nanoparticles in a proper concentration might improve the mechanical properties of a material (9).

Based on the literature review, few studies examined iron and copper nanoparticle incorporation into heat-polymerized acrylic resins. Therefore, the aim of this study was to examine the flexural strength of heat-cure acrylic resins incorporated with 1wt% and 3wt% CuO, Fe_2O_3 and TiO_2 nanoparticles. The null hypothesis was that the nanoparticle added groups would not have higher flexural strength values.

Material and Methods

Sample preparation

Materials used in this study were listed in Table 1. For sample preparation, stainless steel molds were used according to the ISO 1567 standards (2.5 mm thick, 10 mm wide and 65 mm long) (10). Pink Wax (Cavex Set Up wax; Cavex, Holland) was put into the molds, after 70 wax samples were prepared, they invested in dental stone (Fujirock EP; GC) by using a metal denture mold. Wax was eliminated by conventional methods.

The heat-cure acrylic resin samples (Panacryl, Arma Dental, İstanbul, Turkey) were prepared by mixing powder and liquid at a 25 gr/10 ml ratio according to the manufacturer's recommendation. As for the groups with nanoparticle incorporation in a proportion of 1wt% and 3wt%, Fe_2O_3 nanoparticles (99.9 % purity 50 nm particle size), CuO nanoparticles (99.9 % purity 20 nm particle size) and TiO_2 nanoparticles (99.9 % purity 30 nm particle size) were added to acrylic resin using a digital precision scale (Nanografi Nanotechnology, Ankara, Turkey). The acrylic resin powder with nanoparticles (1 wt% and 3 wt%) were thoroughly homogenized in a mixer (President Dental, Germany) in a 2900 rpm cycle for 30 seconds.

For polymerization, the resins were put into the mold, were kept for 8 h at 74±1 ºC in water, after 8 h, and they were boiled for 2 h. After the resin samples were retrieved from the molds, they were polished with 200, 400 and 600 grits respectively to obtain a standard surface (Waterproof silicon carbide paper, English Abrasives Ltd., London, England) for 5 minutes under the water-cooling (Figure 1). The sizes of the samples were measured to ensure standardization for the test processes. The samples were kept in distilled water for one week.

Flexural strength test

Universal Testing Machine (Lloyd-LRX, Lloyd Instruments, Fareham, UK) was used to determine the flexural resistance. A load was applied in the center of the samples at the crosshead speed of 5 mm/min until a fracture occurs. The maximum force (N) and the flexural strength values (mm) of each sample were automatically recorded in the processing unit of the machine.

Statistical analysis

Statistical package software (SPSS Version 24.0; SPSS Inc., Chicago, IL, USA) was used for data analysis. The data obtained in the study displayed a statistically normal distribution. Since the data had normal distribution within the groups, the means and the variations among the groups were examined by using the One-Way Variance Analysis (ANOVA) and the post-hoc Tukey test. The confidence interval was set to 95% and $p < 0.05$ was considered statistically significant. Variations within groups were examined by calculating the Weibull module (m). The fracture values were classified from minimum to maximum, and line graphics were obtained by using the median regression method.

Results

The values obtained from the flexural strength tests were compared by using ANOVA and Tukey test. The means, stan-

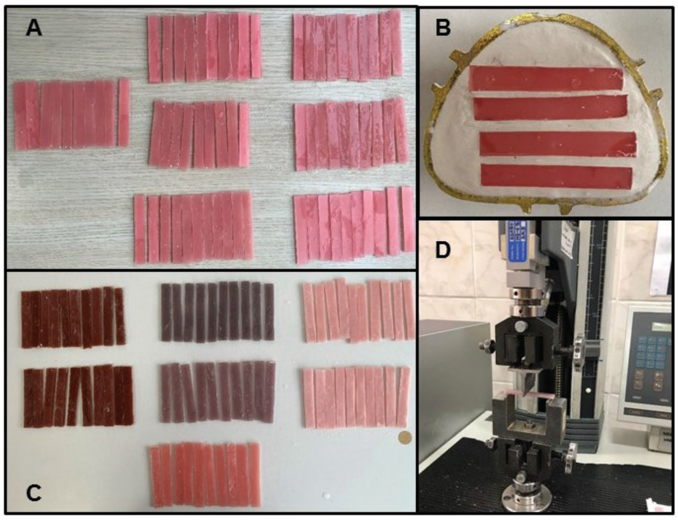

Figure 1. *Experimental design of the study (A: 70 waxes, B: Waxes placed in muffles, C: Control and modified heat-cure acrylic resins, D: Flexural strength test).*

Table 1: *Experimental materials used in this study.*

Material Name	Manufacturer	Composition
Panacryl Heat-cure acrylic resin	Arma Dental, İstanbul, Turkey	95% Methyl Methacrylate (MMA), 5% Ethilenglicoldimethilacrylate (EGDMA)
Fe_2O_3 nanoparticles	Nanografi Nanotechnology, Ankara, Turkey	99.9 % purity 50 nm particle size
CuO nanoparticles	Nanografi Nanotechnology, Ankara, Turkey	99.9 % purity 20 nm particle size
TiO_2 nanoparticles	Nanografi Nanotechnology, Ankara, Turkey	99.9 % purity 30 nm particle size

dard deviation values, minimum -maximum and Weibull analysis results are shown in Table 2. Boxplot graph and Weibull graph are shown in Figures 2 and 3. According to the flexural strength test results in this study, the significantly highest flexural strength value was found for 1wt% TiO_2 added acrylic resin. 3wt% CuO added acrylic resin had lower flexural strength values than those of the control group. Similarly, flexural resistance values, Weibull modulus and Weibull characteristics of 3wt% Fe_2O_3, CuO and TiO_2 added acrylic resins were lowest than those of the other groups.

Discussion

In the present study, 1wt% TiO_2 added group had significantly higher flexural strength value than all other groups, while 3wt% Fe_2O_3, CuO and TiO_2 added groups had lower flexural strength value than the other groups. Thus, the null hypothesis of the study was rejected.

Some recent studies in the literature examined the effects of adding nanoparticles in different concentrations to enhance the resistance of denture base material against fractures and improve its mechanical properties. According to these studies, the addition of nanoparticles in various amounts ranging between 1wt% and 5wt% strengthens the mechanical properties of materials; however, these properties weaken when the amount exceeds 5wt% (11, 12). Therefore, 1wt% and 3wt% nanoparticle incorporation were examined in the present study. The flexural strength values obtained in this study also showed a significant decrease in flexural strength values for 3wt% concentration added groups when compared to other groups. Compared to monomers and polymers, nanoparticles might negatively affect the properties of a material by clustering in large sizes (13, 14). When nanoparticles are inappropriately distributed in an acrylic resin matrix, monomer reaction decreases, and the amount of non-reactive monomers increases (15). It is also possible that the stress concentrations caused by filler particles alter the elasticity module and crack the growth mode of resin (14). Therefore, if the nanoparticle can be uniformly distributed in the resin, the stress concentration can be avoided, and the mechanical properties of the resin can be improved (16). Therefore, in this study, the nanoparticles were incorporated into resin with a mixing machine for 30 minutes to ensure homogenization.

Toodehzaeim *et al.* (17) added 0.01wt, 0.5wt and 1wt% CuO nanoparticles to nanocomposites It was reported that the shear bond strength values did not reveal a significant difference between the control group and CuO particle added groups. The results of the present study showed a decrease in strength values when compared to the control group in the case of CuO particle addition. Poosti *et al.* (18) examined the mechanical properties of TiO_2 added resins by using the shear bond test and did not find a difference between TiO_2 added group and the control group. The difference in our study might be explained by using different material and mechanical tests as well as different nanoparticle proportions.

The 3-point bending test is a commonly used and well-recognized method to measure the bending resistance of denture base polymers in conformity with international ISO 1567 standards (10). The samples were applied 5 mm/min force as suggested in Barbosa's study (19). According to ISO 1567, the bending resistance of an acrylic resin polymerized by using any known methods should not be lower than 65 MPa (10). The

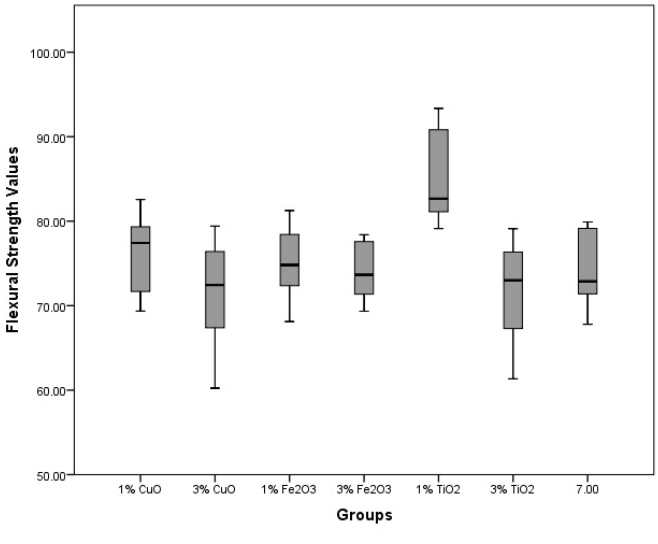

Figure 2. *Boxplot graph of flexural stress values and statistical significant differences.*

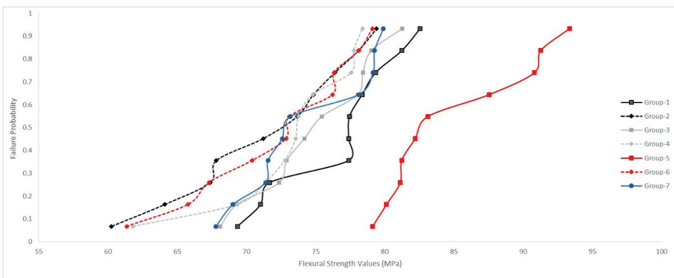

Figure 3. *Weibull plot of failure probability against stress to failure (MPa) for each group.*

Table 2: *The mean flexural strength values, SDs, min and max values of each group.*

Groups	Mean ± SD	Min	Max	Weibull Modulus	Weibull Characteristics
1% CuO	76.58±1.40	69.35	82.56	18.31	78.68
3% CuO	71.32±1.99	60.24	79.41	12.26	74.16
1% Fe_2O_3	74.91±1.37	68.11	81.26	18.88	76.90
3% Fe_2O_3	73.12±1.56	61.81	78.40	15.38	73.99
1% TiO_2	84.99±1.66	79.12	93.35	16.53	87.52
3% TiO_2	72.07±1.84	61.34	79.11	13.38	74.72
Control	74.20±1.43	67.79	79.90	17.33	76.35

group with the lowest bending resistance in our study was the 3% CuO added group (71.32 Mpa), which might be explained by the fact that acrylic resin failed to complete the chain reaction and the full polymerization due to metal nanoparticles. Similarly, the literature review showed that the effect of incorporating nanoparticles such as Fe_2O_3, TiO_2 and SiO_2 on the mechanical properties of acrylic resins is directly related to the nature of "concentration". The strength of a material decreases as nanoparticle concentration increases (7, 11). In contrast, a study found that the addition of 1wt% TiO_2 decreases the micro hardness of an acrylic resin while adding 5wt% TiO_2 causes an increase in this type of hardness. Since such an increase causes higher fatigue resistance in acrylic resin, it can be used in certain equipment restorations such as occlusal splints (20, 21). On the other hand, since higher concentrations (5wt% TiO_2) are likely to deteriorate the impact resistance of a resin, any attempt to add nanoparticles after reaching saturation during polymerization might lead to interruptions in the sustainability of resin chains (22). In addition, it was observed that nanometals added in high concentrations might cause great changes in the color of the acrylic resin.

Weibull analysis was included in the present study because it provides information about the variations in the results as well as the reliability and endurance of the material used and makes a more accurate clustering of fracture tension values and indicates. The highest Weibull characteristic value in this study was found in 1wt% TiO_2 added group. A high Weibull characteristic value means that the material is durable and displays better mechanical properties. On the other hand, the lowest Weibull characteristic values were calculated for the groups in which 3wt% nanoparticles were added. These findings imply that acrylic resins might get weaker and have lower fracture resistance in time. The Weibull results in our study are consistent with the flexural resistance test results. Thus, Weibull distribution can be considered an alternative method in evaluating the fracture possibility of materials (23).

This invitro study is limited, as it does not accurately simulate real conditions of the oral cavity. When it is considered the fact that the acrylic resin might be affected by factors such as temperature and Ph changes in the oral cavity, it might be difficult to predict the material's original clinical reactions by interpreting the in vitro reactions. Obtaining microscopic views of fractured surfaces might provide more detailed information about fatigue resistance by allowing an effective examination of surface structure and some possible voids inside the resin interface. Due to the mentioned limitations, it is necessary to conduct more advanced clinical and in vitro studies examining the effects of incorporating various nanoparticles into acrylic resin.

Conclusion

It can be concluded that adding 1wt%TiO_2 nanoparticles to acrylic resin improved the mechanical properties of acrylic resins. Despite some limitations, nanoparticle addition to acrylic resins might improve its mechanical features; however, flexural resistance values of acrylic resin decreases as the percentage of incorporated nanoparticles increases.

Türkçe özet: *Akrilik Rezinlerin Metal Nanopartikülleri İlavesi Sonrası Eğilme Mukavemetinin Değerlendirilmesi. Amaç: Bu çalışmanın amacı, demir, bakır, titanyum nanopartikülleri eklenmiş ısı ile polimerize olan akrilik rezinin eğilme mukavemetini incelemektir. Gereç ve Yöntem: Üreticinin talimatlarına göre hazırlanmış yetmiş adet örnek (65x10x2.5 mm³) yedi gruba ayrıldı. Isı ile polimerize edilmiş akrilik rezine ağırlıkça %1 ve %3 oranında* Fe_2O_3*, CuO ve* TiO_2 *nanopartiküleri manuel olarak eklendi. 3 noktalı eğilme testi için 5 mm/dk süreye ayarlanan Universal Test Cihazı kullanıldı. İstatistiksel analizler için ANOVA ve Weibull analizleri kullanıldı. Bulgular: Nanopartikül eklenen grup ile kontrol grubu arasında istatistiksel olarak fark bulundu. En yüksek ortalama değer ağırlıkça %1 TiO2 eklenen grup için (84.99 MPa) ve en düşük değer ağırlıkça %3 CuO eklenen grup için gözlendi (71.32MPa) (p<0.001). Ağırlıkça %3* Fe_2O_3 *ve CuO ilaveli gruplar kontrol grubuna göre daha düşük değerler gösterdi. Sonuç: Akrilik rezine* TiO_2 *nanopartiküllerinin ağırlıkça %1 oranında katılması rezinlerin eğilme mukavemeti değerlerini arttırmıştır. Limitasyonlar dahilinde, akrilik rezinlere nanopartikül ilavesi mekanik özellikleri iyileştirebilir; ancak nanopartikül ilave yüzdesi arttığında akrilik rezinlerin eğilme mukavemeti değerleri düşmektedir. Anahtar Kelimeler: Akrilik rezin, nanopartikül, eğilme mukavemeti, protetik diş tedavisi, weibull analizi*

Ethics Committee Approval: Not required.

Informed Consent: Not required.

Peer-review: Externally peer-reviewed.

Author contributions: SOA, CA, EM participated in designing the study. SOA, CA, EM participated in generating the data for the study. SOA, CA, EM participated in gathering the data for the study. SOA, CA, EM participated in the analysis of the data. SOA, CA, EM wrote the majority of the original draft of the paper. SOA, CA, EM participated in writing the paper. SOA, CA, EM have had access to all of the raw data of the study. SOA, CA, EM have reviewed the pertinent raw data on which the results and conclusions of this study are based. SOA, CA, EM have approved the final version of this paper. SOA, CA, EM guarantee that all individuals who meet the Journal's authorship criteria are included as authors of this paper.

Conflict of Interest: : The author had no conflict of interest to declare.

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