



The flexural strength of acrylic resin repairs reinforced with different nanoparticles

Secil Ozkan Ata^{a,*}, Canan Akay^a, Emre Mumcu^a

^aOsmangazi University, Faculty of Dentistry, Department of Prosthodontics, Eskisehir, Türkiye

ARTICLE INFO

Keywords:

3-point flexural test
Denture fracture
Nanoparticles
Auto-polymerized acrylic resin

Received: Feb 26, 2022

Accepted: Oct 14, 2022

Available Online: 22.10.2022

DOI:

[10.5455/annalsmedres.2022.02.080](https://doi.org/10.5455/annalsmedres.2022.02.080)

Abstract

Aim: The purpose of this study is to assess the flexural strength of heat-cure acrylic resins repaired with auto-polymerized acrylic resins modified with 1%, 3% TiO₂, Fe₂O₃, CuO nanoparticles.

Materials and Methods: Fifty-six samples (65x10x2.5 mm) were prepared with heat-cure acrylic resins and divided into 7 groups (8 samples each) to make repair procedure: Specimens were repaired with unmodified (control group) and 1%, 3% TiO₂, Fe₂O₃, CuO added auto-polymerized acrylic resins. For 3-point flexural test, the force was loaded at 5 mm/min crosshead speed directly to the middle part of the repaired acrylic resin with a Universal Testing Machine. Data were analyzed statistically by ANOVA followed by Tukey test ($p < 0.05$).

Results: Control group showed lower strength values than the other groups. 1% TiO₂ nanoparticle added group showed significantly higher flexural strength values than 3% added groups and control group ($p < 0.05$). The highest strength value was measured in 1% TiO₂ group.

Conclusion: Acrylic resins reinforced with 1% TiO₂ developed higher strength than resins reinforced with 3% TiO₂, Fe₂O₃, CuO nanoparticles. Adding 1% nanoparticles to resins could improve the fracture strength of materials.



Copyright © 2022 The author(s) - Available online at www.annalsmedres.org. This is an Open Access article distributed under the terms of Creative Commons Attribution-NonCommercial-NoDerivatives 4.0 International License.

Introduction

Acrylic resins are the most widely used removable denture base materials in prosthetic dentistry. Unfortunately, the long-term success of acrylic resins is unsatisfactory, and it appears as acrylic prosthesis fractures. These fractures mostly occur due to acrylic resin manufacturing problems, poorly balanced occlusion, insufficient mechanical properties of the repair resin [1, 2]. As a result of clinical use, stress occurs at the denture over time and this stress decreases the resistance of the resin and results with fractures.

After denture fractures, denture repair is considered as an alternative, as the construction of a new denture is time-consuming and expensive.

However, the method for denture repair should be cheap and easily applicable, and the repaired dentures should show sufficient mechanical and dimensional strength, and color match [3-6]. Auto-polymerized acrylic resins have important advantages for fracture repair due to its easy handling, no need for technical precision, and shortening

the prosthesis repair time [4,6]. However, the long-term strength of prostheses is unsatisfactory, and fractures may occur repetitively [4,5,6].

Nanoparticles are progressively utilized in materials science due to their wear and tear resistance and anti-corrosion capabilities. There are some researches to enhance the mechanical strength of repaired denture resin by adding nanoparticles such as copper oxide (CuO), iron oxide (Fe₂O₃), zinc oxide (ZnO), titanium dioxide (TiO₂), aluminum oxide (Al₂O₃) and silicon dioxide (SiO₂) [7,8]. Despite these studies, the effect of reinforcement with CuO and Fe₂O₃ nanoparticles has not been fully clarified.

Therefore, the aim of this study was to assess the flexural strength of the heat-cured acrylic resins repaired with auto-polymerized resins reinforced with 1%, 3% TiO₂, Fe₂O₃, CuO nanoparticles. The null hypothesis was that the flexural strength of the acrylic resin in repair process would not be influenced by the inclusion of nanoparticles to auto-polymerized resin.

Materials and Methods

Materials used in this study are listed in Table 1.

*Corresponding author:

Email address: secilozkanata@gmail.com (Secil Ozkan Ata)

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% Ethilenglicoldimethylacrylate (EGDMA)
Autopolymerizing acrylic resin	Birlesik Group Dental (BGD), Turkey	95% Methyl Methacrylate (MMA), 5% Ethilenglicoldimethylacrylate (EGDMA)
Fe ₂ O 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

Sample preparation

According to American Dental Association Specification no. 12, samples (n=56) were made from metal molds (65×10×2.5 mm) for flexural strength assessment [9]. Heat-cure acrylic resin samples were set up by conventional technique using metal flasks. Waxes (Cavex Set Up wax; Cavex) were put into the molds, after 56 wax samples were prepared, they were invested in dental stone (Alston Dental Stone, Turkey) using a metal denture flask. Waxes were eliminated by conventional methods, followed by using of a separator (IMICRYL Imibase, Turkey), the flasks were put to get to room temperature. The heat cure acrylic resins (Panacryl, Arma Dental, İstanbul, Turkey) were prepared by mixing powder and liquid according to the manufacturer's recommendation. For polymerization, the resins put into the mold, they were kept for 8 h at

74±1 °C in water, after 8 h, they were boiled for 2 h. After the resin samples were retrieved from the molds, they were polished with 400 and 600 grits respectively to obtain a standard surface (Waterproof silicon carbide paper, English Abrasives Ltd., London, England) for 5 minutes (Figure 1).

Repair procedure

Two lines were drawn 1.5 mm apart on either side of the sample center and cut vertically along their long axis with a high-speed diamond disc, creating 3 mm between these two lines. A pair of divided samples was put in a metal mold. The samples were distributed randomly into 7 groups (n=8) according to nanoparticle type and concentration. To prepare the auto-polymerizing resin, the amount of TiO₂ nanoparticles (99.9 % purity 30 nm particle size), Fe₂O₃ nanoparticles (99.9 % purity 50 nm particle size) and CuO nanoparticles (99.9 % purity 20 nm particle size) were added in concentrations of 1% and 3%wt. to the resin powder (Nanografi Nanotechnology, Ankara, Turkey) and thoroughly homogenized in a mixer (President Dental, Germany) in a 2900 rpm cycle for 30 seconds. Nanoparticle added resins were then mixed and filled into the repair gap in accordance with the manufacturer's recommendations. After then, the repaired samples were put in a pressure pot for 20 min at 2-bar pressure. The control group was repaired with unmodified auto-polymerizing resin. After the polymerization process was completed, the samples were removed from the molds. The excess resin was removed using a #600 silicon carbide paper under water irrigation and completed to a 10 × 65 × 2.5 mm rectangular specimen. The samples were stored in distilled water at 37 °C for 24 h (Figure 1).

Three-point flexural test

Universal testing machine (Lloyd-LRX, Lloyd Instruments, Fareham, UK) was used for 3-point flexural tests. A load of 500 N was applied at a crosshead speed of 5 mm/min until fracture occurred. Load at fracture was noted, and according to the sample's dimensions flexural strength was assessed [10]. The flexural strength (S in MPa) for a rectangular sample under a load was calculated by using the formula $S=3WL/2bd^2$, where S is the flexural strength (MPa), W is the load the sample fracture

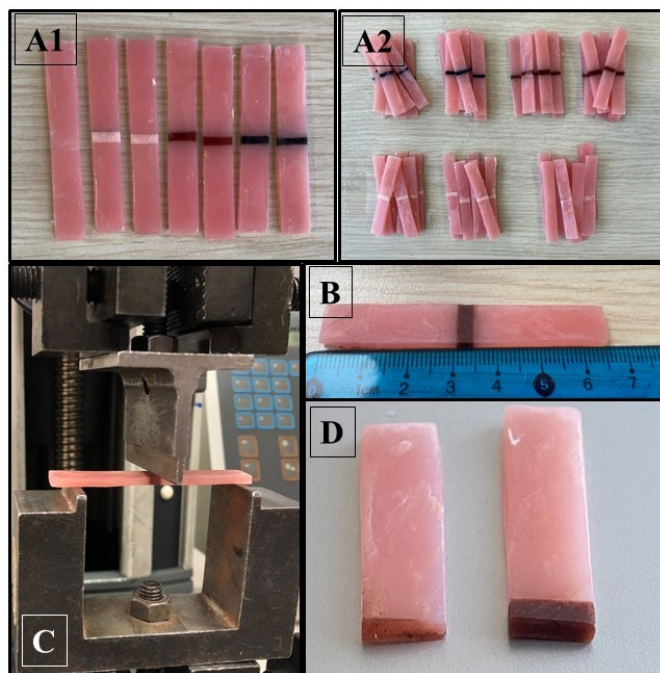


Figure 1. A1-A2: Specimens used in this study, B: Dimensional length of specimen and repair part (3mm), C: Flexural testing machine, D: Fractured specimen after testing.

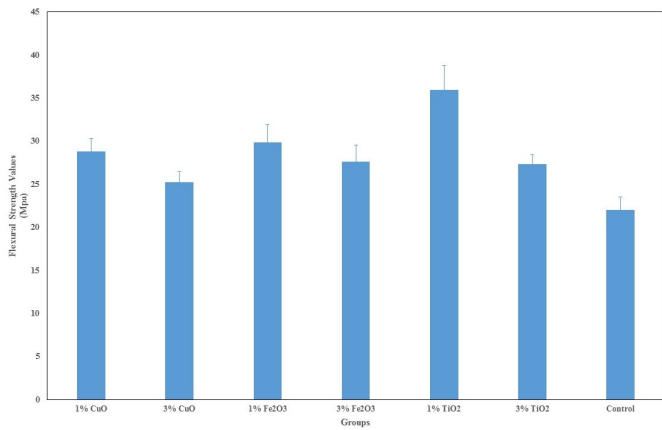


Figure 2. Bar graph of flexural stress values with SD's.

(N), L is the distance measured between the two supports, b is the sample width, and d signifies the sample thickness (Figure 1) [11,12] .

Statistical analysis

Statistical package software (SPSS Version 24.0; SPSS Inc., Chicago, IL, ABD) was used for the statistical analyses of the results. 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 doing using One-Way Variance Analysis (ANOVA) and the post-hoc Tukey test.

Results

The flexural strength values, standard deviation, and min-max values were shown in Figure 2 and Table 2.

The findings stated that control group showed lower strength values than the other groups. 1% TiO₂ nanoparticle added group showed significantly higher flexural strength values than 3% added groups and control group (p < 0.05). Moreover, highest strength value was at the 1% TiO₂ added group and the lowest strength value was at the control group. 3% added CuO group showed lower values than other nanoparticle added groups. The results of statistical analysis is shown in Table 3.

Discussion

In this study, the auto-polymerized resin reinforcement was conducted by incorporating 1%, 3% TiO₂, Fe₂O₃,

Table 2. Number of specimens, the mean flexural strength values, SDs, min and max values of each group.

Groups	CuO		Fe ₂ O ₃		TiO ₂		Control
	1%	3%	1%	3%	1%	3%	
N	8	8	8	8	8	8	8
Mean	28.80	25.22	29.85	27.61	35.91	27.30	21.97
SD	1.49	1.25	2.10	1.92	2.88	1.10	1.54
Min	21.99	20.11	21.59	21.49	23.46	22.39	15.28
Max	34.71	28.91	36.31	35.59	45.18	31.89	26.81

Table 3. P values between groups.

Groups	1% CuO	3% CuO	1% Fe ₂ O ₃	3% Fe ₂ O ₃	1% TiO ₂	3% TiO ₂	Control
1% CuO	-	0.812	1.000	0.999	0.111	0.997	0.141
3% CuO	0.812	-	0.568	0.968	<i>0.003</i>	0.984	0.871
1% Fe ₂ O ₃	1.000	0.568	-	0.977	0.251	0.956	0.056
3% Fe ₂ O ₃	0.999	0.968	0.977	-	<i>0.037</i>	1.000	0.331
1% TiO ₂	0.111	<i>0.003</i>	0.251	<i>0.037</i>	-	<i>0.027</i>	<i>0.000</i>
3% TiO ₂	0.997	0.984	0.956	1.000	<i>0.027</i>	-	0.399
Control	0.141	0.871	0.056	0.331	<i>0.000</i>	0.399	-

Statistical significant values between groups shown in italic numbers.

CuO nanoparticles and their effects were measured with the flexural strength test. According to the results, all concentrations of nanoparticle addition to auto-polymerized resin increased the flexural strength values of the repaired resin. Therefore, the null hypotheses were rejected.

Nanoparticles were added at 1%, 3% by weight to auto-polymerized resin in this study. It was reported that low content addition of nanoparticles into acrylic resin polymers between 1% and 5% improve mechanical properties of resins [13]. It was also stated that the content level more than 5% could have negative impact on mechanical properties [13,14]. This study also showed that mechanical properties of resins decreased by increasing nanoparticle proportion.

In this study, the control group showed less flexural strength values than the nanoparticle added groups. Polyzois et al. [15] found higher flexural strength values in samples repaired with glass-fiber reinforced auto-polymerized acrylic resins. Vikram et al. [16] reported that the incorporation of nanoparticles improved the surface hydrophobicity and decreased the accumulation of molecules. These results are parallel with our findings, as the reinforcement repair material with nanoparticles increased the flexural strength values. In this study 3% concentration performed less flexural strength than 1% concentration added nanoparticles. Sodagar et al. [7] stated that adding 0.5% and 1% of TiO₂ and SiO₂ nanoparticles to acrylic resin decreased the mechanical properties of the resin. They also reported that increasing concentration of TiO₂, reduced the mechanical strength values of the material, and nanoparticles might have negative effects on polymerization of acrylic resins [7]. On the other hand, Zhang et al. [17] studied the effect of TiO₂ nanoparticles on different concentrations of acrylic resin. They stated that the highest strength value was seen in resins modified with 3% TiO₂. In our study 3% TiO₂ added group showed less strength values than 1% TiO₂ group. Toodehzaeim et al. [18] evaluated the effects of CuO nanoparticles on the mechanical strength of orthodontic adhesives. They concluded that the shear bond strength of adhesives was not affected by adding CuO nanoparticles. These studies are not in line with our findings. These variations may be explained by the differences in material type, mechanical test, and different experimental procedures.

The most used and recognized method to measure flexural resistance of denture base polymers in conformity with international ISO 1567:1999 standards is 3-point flexural

test [19]. The samples were applied at 5 mm/min force as suggested in Barbosa's study [20]. According to ISO 1656, bending resistance of an acrylic resin should not be lower than 65 MPa [21]. It is known that the flexural strength values of auto-polymerized acrylic resin are lower than that of the heat-cure resin; hence, more likely to fracture due to strength inconsistency [22,23]. The previous studies reported that the flexural strength of auto-polymerized resin is 18 to 81% lower than those heat-cure acrylic resins [22-25]. In this study, the highest strength was 45.18 MPa less than 65 MPa. More studies are needed to strengthen the mechanical properties of the repaired denture base resins for longer clinical use.

In this study, the fractures were observed mostly at the bonding areas between auto-polymerized and heat-cure acrylic resins. Therefore, reinforcing repair material is not enough for repaired material's mechanical strength, but also it is needed to increase the strength of bonding areas by roughening mechanically or chemically to ensure mechanical interlocking. The present study is limited due to being performed under laboratory conditions, which are different from the oral environment. In addition, further in vitro studies are needed including storage of the samples in saliva and/or application of mechanical and thermal cyclic stresses, adjusting bonding interface of repaired resins, different surface treatment procedures, or incorporating different nanoparticles. Clinical studies are also required to fully understand resins reinforced with the nanoparticles.

Conclusion

It can be concluded that adding nanoparticles, especially 1% TiO₂ reinforced the mechanical properties of resins. As most of the failures in this study occurred between the bonding area, more research is needed as the interface properties of the bonded material to the prosthesis are important for mechanical interlocking and strength.

Ethics approval

In our study, animals and humans were not used, only laboratory materials were used.

References

1. Khalaf H, Salman T. The Influence of Adding of Modified ZrO₂-TiO₂ Nanoparticles on Certain Physical and Mechanical Properties of Heat Polymerized Acrylic Resin. *Journal of Baghdad College of Dentistry*. 2021;27: 33-39.
2. Zappini G, Kammann A, Wachter W. Comparison of fracture tests of denture base materials. *J Prosthet Dent*. 2003;90:578-85.
3. Minami H, Suzuki S, Kurashige H et al. Flexural strengths of denture base resin repaired with autopolymerizing resin and reinforcements after thermocycle stressing. *J Prosthodont*. 2005;14:12-8.
4. Beyli MS, Von Fraunhofer JA. Repair of fractured acrylic resin. *J Prosthet Dent*. 1980;44:497-503.
5. John J, Gangadhar SA, Shah I. Flexural strength of heat-polymerized polymethyl methacrylate denture resin reinforced with glass, aramid, or nylon fibers. *J Prosthet Dent*. 2001;86:424-7.
6. Arioli Filho JN, Butignon LE, Pereira Rde P et al. Flexural strength of acrylic resin repairs processed by different methods: water bath, microwave energy and chemical polymerization. *J Appl Oral Sci*. 2011;19:249-253.
7. Sodagar A, Akhouni M, Bahador A et al. Effect of TiO₂ nanoparticles incorporation on antibacterial properties and shear bond strength of dental composite used in Orthodontics. *Dental Press J. Orthod*. 2017; 22: 67-74.
8. Agnihotri R, Gaur S, Albin S. Nanometals in Dentistry: Applications and Toxicological Implications- a Systematic Review. *Biol Trace Elem Res*. 2020;197:70-88.
9. Revised American Dental Association specification no. 12 for denture base polymers. *J Am Dent Assoc* 1975;90:451-8.
10. Revilla-León M, Al-Haj Husain N, Ceballos L, Özcan M. Flexural strength and Weibull characteristics of stereolithography additive manufactured versus milled zirconia. *J Prosthet Dent*. 2021 Apr;125:685-690.
11. Alkurt M, YeşilDuymuş Z, Gundogdu M. Effect of repair resin type and surface treatment on the repair strength of heat polymerized denture base resin. *J Prosthet Dent* 2014;111:71-78.
12. Gad MM, Rahoma A, Al-Thobity AM, ArRejaie AS. Influence of incorporation of ZrO₂ nanoparticles on the repair strength of polymethyl methacrylate denture bases. *Int J Nanomedicine*. 2016;11:5633-43.
13. Alnamel HA, Mudhaffer M. The effect of Silicon dioxide Nanofillers reinforcement on some properties of heat cure polymethyl methacrylate denture base material. *Journal of Baghdad College of Dentistry*. 2014;26:32-36.
14. Omer RA, Ikram FS. Effect of Addition of Silver Nanoparticles on Flexural and Impact Strength of Heat Cure Acrylic Resin. *Erbil Dental Journal*. 2019;2:243-50.
15. Polyzois GL, Tarantili PA, Frangou MJ, Andreopoulos AG. Fracture force, deflection at fracture, and toughness of repaired denture resin subjected to microwave polymerization or reinforced with wire or glass fiber. *J Prosthet Dent*. 2001;86:613-9.
16. Vikram S, Chander NG. Effect of zinc oxide nanoparticles on the flexural strength of polymethylmethacrylate denture base resin. *Eur Oral Res* 2020; 54: 31-5.
17. Zhang JG. Study on friction and wear behavior of PMMA composites reinforced by HCl-immersed TiO₂ particles. *Journal of Thermoplastic Composite Materials*. 2014: 603-10.
18. Toodehzaeim MH, Zandi H, Meshkani H et al. The Effect of CuO Nanoparticles on Antimicrobial Effects and Shear Bond Strength of Orthodontic Adhesives. *J Dent (Shiraz)*. 2018;19:1-5.
19. Reis JM, Vergani CE, Pavarina AC et al. Effect of relining, water storage and cyclic loading on the flexural strength of a denture base acrylic resin. *J Dent* 2006;34:420-6.
20. Barbosa DB, de Souza RF, Pero AC et al. Flexural strength of acrylic resins polymerized by different cycles. *J Appl Oral Sci* 2007; 15: 424-8.
21. Uzun G, Hersek N. Comparison of the fracture resistance of six denture base acrylic resins. *J Biomater Appl* 2002; 17: 19-29.
22. Rached RN, Powers JM, Del Bel Cury AA. Repair strength of auto polymerizing, microwave, and conventional heat-polymerized acrylic resins. *J Prosthet Dent*. 2004;92:79-82.
23. Faot F, da Silva WJ, da Rosa RS et al. Strength of denture base resins repaired with auto- and visible light-polymerized materials. *J Prosthodont*. 2009;18:496-502.
24. Heidari B, Firouz F, Izadi A et al. Flexural strength of cold and heat cure acrylic resins reinforced with different materials. *J Dent (Tehran)*. 2015;12:316-323.
25. Yadav NS, Khare S, Mishra SK et al. In-vitro evaluation of transverse strength of repaired heat cured denture base resins without surface treatment and with chemical and mechanical surface treatment. *J Int Oral Health*. 2015;7:89-92.