

**EFFECT OF ADDING TIO2 NANOPARTICLES ON** THE MECHANICAL PROPERTIES OF HEAT CURED **ACRYLIC RESIN BEFORE AND AFTER THERMOCYCLING** 

Bayiumy Ali Sayed<sup>\*</sup> <sup>(D)</sup> and Elsokkary Mohammad Mahmoud<sup>\*\*</sup> <sup>(D)</sup>

#### ABSTRACT

Objective: The aim of this study was to evaluate the effect of different concentrations of titanium dioxide nanoparticles on the mechanical properties of heat polymerized acrylic resin before and after thermocycling. The tested parameters were flexural strength and hardness. Material and Methods: 120 specimens were prepared according to the specific dimension for each test (flexural strength and micro hardness) and distributed into: - Before thermocycling (control, (TiO2) nanoparticles with concentration (0.1% and 0.2%). - After thermocycling (control, (TiO2) nanoparticles with concentration (0.1% and 0.2%). Thermal cycles were performed in a thermocycling machine consisted of 5000 cycles at 5°C and 55°C. Results: Flexural strength and micro hardness were tested for both groups. The data was collected and statistically analyzed. Both Flexural strength and micro hardness were decreased by adding TiO2 nano particles concentrations. Conclusion: adding (TiO2) nanoparticles to methyl methacrylate adversely affects the flexural strength and micro hardness before and after thermocycling.

KEYWORDS: Poly methyl methacrylate, TIO2 Nano particles, thermocycling, mechanical properties

#### **INTRODUCTION**

The plentiful benefits of acrylic resin make it the most widely used polymer in removable prosthetic dentistry. Acrylic resin as a prosthetic material has different benefits such as simple processing technique, economic, low density (which enhances retention in oral cavity and patient comfort) and brilliant optical properties<sup>(1)</sup>. Conversely, acrylic is not superlative in all aspects. Acrylic resin is (poly methyl methacrylate) PMMA, which is resin denture base material with weak surface and mechanical properties such as impact and flexural

Article is licensed under a Creative Commons Attribution 4.0 International License

<sup>\*</sup> Lecturer of Prosthodontics, Faculty of Dentistry, Assiut University

<sup>\*\*</sup> Lecturer of Dental Materials, Faculty of Dentistry, Assiut University

strengths <sup>(2)</sup> consequently, a lot of trials have been suggested to improve the strength of acrylic. Lately, nanotechnology attacked the dentistry and introduced multiple ideas and approaches to improve dental applications. It is essential to recognize the technique behind this technology to know how to use it to our benefit. Nano composite polymers are made of matrix and fillers at the Nano scale<sup>(3)</sup>. In dentistry, a lot of researches have been introduced to generate recent versions of acrylic with the addition of different shapes and sizes of nano fillers<sup>(4)</sup>. Currently, international researches showed several progresses in the nanocomposites field after the widespread research on mechanical and physical properties of these nanocomposites<sup>(5)</sup>. Multiple types of nano particles have been added to the matrix of acrylic resin, such as TiO2NP with its properties which are thought to improve mechanical properties<sup>(5,6)</sup>.

Titanium dioxide is a semiconductor material with excellent optical properties and popular in nanotechnology applications.<sup>(7)</sup> Titanium dioxide (TiO<sub>2</sub>) has distinct optical, electrical and chemical properties.<sup>(7,8)</sup> So it has a lot of applications such as pigments, ultra violet protection skin creams and inorganic membranes, etc.<sup>(9,10)</sup> Its important phases include tetragonal, (orthorhombic), and oxygen deficient  $\lambda$ -Ti3O5, having a monoclinic crystal structure and photo-reversible characteristics <sup>(9)</sup>.

TiO2NP has antibacterial effect against multiple bacterial species such as gram-positive and gramnegative bacteria, in addition to activity against viruses <sup>(11-13)</sup>. Adding some materials to improve the antimicrobial properties of the acrylic should not have side effects on the mechanical properties. In reverse, it is extremely favored to add materials that advance both biological and mechanical properties<sup>(14)</sup>.

The outcome of adding TiO2NP into acrylic was reported and stated a decrease in flexural strength. It was mentioned that there is a reverse relation between the concentration of the filler and the flexural strength of reinforced acrylic<sup>(15-18)</sup>. In dissimilarity to preceding studies, researches have reported an increase in flexural strength by using different concentrations of TiO2NP added to acrylic<sup>(19-22)</sup>. Many previous studies have confirmed that TiO2NP enhanced the hardness of heat cure acrylic resin <sup>(19, 20)</sup>. Results of multiple studies concluded that filler content in the range of 2wt% would improve the mechanical properties including the hardness <sup>(23)</sup>

Thermal cycling in a wet environment may affect adversely the mechanical properties of the denture polymers <sup>(31)</sup>. The heat stress may increase water sorption because of increasing the distance between the polymers chains, so absorbed water can act as a plasticizer and soften the denture decreasing strength and hardness, thus reducing the mechanical properties of the material <sup>(32)</sup>.

Therefore, the present study was carried out to investigate the effect of adding titanium dioxide nanoparticles on flexural strength and hardness of heat cured acrylic resin before and after thermo cycling.

#### MATERIAL AND METHODS

An in vitro study was conducted to assess the outcome of adding Titanium dioxide (TiO2) nanoparticles with concentration (1% and 2%) by weight of the acrylic resin polymer powder on the three point flexural strength test, and micro hardness of heat-polymerized acrylic resin before and after thermo cycling.

Anatase subdivisions were set by precipitation from homogeneous standardized solution by means of titanium (IV) isopropoxide as precursor in aqueous solution acidified with nitric acid to pH 2 with a water-to-titanium mole ratio of about 200.<sup>(24-28)</sup>

#### Preparation of PMMA + TiO, NPs

The dried powder was mixed with PMMA powder at different ratios 1 and 2 w/w, typically

for 1%, 1 gm. of TiO2 NPs mixed with 99 gm. of PMMA by meaning of ball mill for 2h to get homogenous mixing. For 2% 2gm TiO2 NPs was mixed with 98 gm. PMMA as described previously.

TABLE (1) Materials used in the current study.

	Material	Specification	Manufacturer
1.	Idobase	PMMA heat cured acrylic resin	Tielmes (Madrid, Spain)
2.	• TiO <sub>2</sub> NPs	• TiO <sub>2</sub> NPs	Nano-gate Company, Egypt

## Sample grouping:

120 specimens were prepared according to the specific dimension for each test (flexural strength and micro hardness) and distributed according to the following table

		Control		T1		T2	
Flexural St.	No TC	10	10	10	10	10	10
	TC	10	10	10	10	10	10
Micro hardness	No TC	10	10	10	10	10	10
	TC	10	10	10	10	10	10

\* TC (Thermocycled) T1 (1% TiO<sub>2</sub> NPs) T2 (2% TiO<sub>2</sub> NPs)

The sample dimensions were selected according to specific dimension of each test, (65 x10x2.5mm) for flexural strength, and micro hardness tests then the wax pattern with this dimensions was used to form the mold. The samples were processed according to manufacture directives (3:1 by volume or 2.5:1 by weight) until reaching the dough stage. Conventional packing method for 1.5 hours at 74°C. After polymerizations were completed, the flasks were gradually bench cooled at room temperature for 30 minutes and then placed for 15 minutes below running water beforehand opening. Specimens were inspected for any irregularity. Defective specimens were removed.

#### **Thermocycling:**

Thermal cycles were performed in a thermocycling machine (SD mechatronic thermocycler D-83620 Feldkirchen-Westerham GERMANY) and consisted of 5000 cycles at 5°C and 55°C with a 30-second dwell time.



Fig. (1) Thermocycler.

## Three point Flexural strength test:

Samples were examined by 3-point bending assessment on Instron universal testing machine (Model 3366, UK) the specimens were loaded then compressed till breakage at a crosshead speed of 10 mm/min. The stress-strain curves were documented with computer software (Instron Bluehill 3 testing software). By this software, the flexural strength was obtained.



Fig. (2) Specimen in Universal testing machine for measuring flexural strength

# Micro hardness test:

Micro-hardness of the samples surface was determined using digital presentation Vickers microhardness tester (Wilson Tukon 1102, Buhler, Germany) with a Vickers diamond indenter and a 10X objective lens. A load of 500g was applied to the surface of the samples. Three indentations not closer than 0.5 mm to the neighboring indentations were prepared on the exterior of each sample. The diagonals lengths of the indentations were measured automatically by scaled microscope.

## **Statistical analysis:**

Data offered as mean and standard deviation (SD). Data explored for familiarity using Kolmogorov-Smirnov and Shapiro-Wilk tests. Oneway ANOVA used to compare between tested groups followed by Tukey's HSD test for pair wise comparison. Independent t-test used to compare between thermocycling (TC) in each group.

The significance level was set at  $P \le 0.05$ .

Statistical analysis was performed with IBM SPSS Statistics for Windows, Version 26.0. Armonk, NY: IBM Corp.



Fig. (3) Specimen in hardness tester for measuring micro hardness.

### RESULTS

#### A. Flexural strength

		Control		T1		T2		
		Mean	SD	Mean	SD	Mean	SD	- p-value
El	No TC	118.7	15.9	115.6	12.1	108.2	26.9	0.687 NS
Flexural St.	TC	106.6	23.8	92.9	18.1	97.8	18.1	0.569 NS
p-value		0.357 NS		0.048*		0.494 NS		

TABLE (1) Mean and SD for Flexural strength for different tested groups

NS= Non-significant, \*= significant Different lowercase letters within each row indicates significant difference.

- Statistical analysis results revealed that Flexural strength was nonsignificantly decreased for ratio of 1% and 2% titanium dioxide nanoparticles before thermo-cycling and after thermo-cycling in comparison to control.
- There are a significant decrease in flexural strength in the Thermocycled T1 in comparison to the non Thermocycled T1

# **B. Hardness**

		Control		T1		T2		1	
		Mean	SD	Mean	SD	Mean	SD	— p-value	
Hardness	No TC	36.6	3.1	34.3	3.6	35.0	4.2	0.615 NS	
	TC	32.9 <sup>ab</sup>	1.5	30.8ª	1.5	33.4 <sup>b</sup>	0.5	0.016*	
p-value		0.04	0.042*		0.079 NS		0.425 NS		

TABLE (2) Mean and SD for Hardness for different tested groups

NS=Non-significant, \*= significant

Different lowercase letters within each row indicates significant difference.

Statistical analysis results revealed that Micro hardness strength was non-significantly decreased for ratio of 1% and 2% titanium dioxide nanoparticles before thermo-cycling and significant decrease after thermo-cycling in comparison to control.

## DISCUSSION

Numerous studies have been performed to advance the assets of poly methyl meth acrylate, which comprise adding of supporting material as fibers, hybrid reinforcement, fillers and lately, nanoparticles. However, the most effective strengthening is not obvious, and investigation academics are disordered about evaluating such assistances. An additive has two significant dedications on prosthesis. The first resolution is to improve the strength and prevent fracture, and the second resolution is to advance the dimensional accuracy to avoid residual ridge resorption. (29) Lately, nanotechnology attacked the dentistry and introduced analytical exploration plans to realize the possible applications and probable profits in dentistry. Currently, international researches displayed several progresses in the nanocomposites field later the prevalent exploration on mechanical and physical possessions of these nanocomposites <sup>(5)</sup>. Many nanoparticles have been incorporated into the acrylic medium. In this study TiO2NP were

designated to estimate the outcome of strengthening on the flexural strength and micro hardness of acrylic resin before and after thermocycling.

TiO2NP have been known to be effective alongside an extensive variety of microbes <sup>(13)</sup>. The adding of some material to advance the antimicrobial belongings of the acrylic resin should not have an adversarial outcome on the mechanical assets.

By reviewing the previous studies it was founded that low percentages of TiO2NP give rise to enhanced assets if compared to greater ratios. Humble adding of 1-2wt% proportions showed better-quality assets, whereas increasing the packing contented extra than 5wt% expressively weakened the finishing nanocomposites <sup>(30)</sup>.

This investigation designed to evaluate probable fluctuations in the mechanical assets of acrylic resin denture base material, in specific, the flexural strength, and micro-hardness by including of Tio2 Nano-elements with two altered applications 1%, and 2% before and after thermocycling. Built on flexural strength values gained in the current study, it has been established that flexural strength decreased by addition of TiO2NP to acrylic resin. This result agrees with the previous studies concluded that the combination of TiO2 Nano-particles into PMMA can unfavorably affect its flexural strength<sup>(15-18)</sup>. In distinction to this experiment, greater flexural properties were conveyed with different concentrations of TiO2NP additional to PMMA than those of normal PMMA <sup>(19,20,22,23)</sup>. This advance may be ascribed to the outcome of salinization of the nanoparticles <sup>(18)</sup> or the better diffusion of the nanoparticles inside the medium.

Micro-hardness of polymethyl methacrylate reinforced with titanium dioxide nanoparticles in different concentrations demonstrated decrease more than conventional polymethyl methacrylate. This result disagrees with study that concluded that the adding of titanium dioxide nanoparticles to heat cured PMMA improve its surface hardness <sup>(20, 21, 23)</sup>.

It was suggested the necessity of addition 5% TiO2NP to improve surface micro hardness of acrylic resin to significant standards <sup>(19)</sup>.

#### CONCLUSIONS

Within the limitation of this study we could concluded that:

Flexural strength and microhardness of acrylic resin material reinforced with TiO2 nanoparticles are adversely affected before and after thermocycling.

#### REFERENCES

- R.L. Sakaguchi and J.M. Powers, Craig's Restorative Dental Materials, Elsevier Health Sciences, Philadelphia, PA, USA, pp. 163-176, 2012.
- N. Murakami, N. Wakabayashi, R. Matsushima, A. Kishida, and Y. Igarashi, "Effect of high-pressure polymerization on mechanical properties of PMMA denture base resin," Journal of the Mechanical Behavior of Biomedical Materials, vol. 20, pp. 98–104, 2013.
- R. Adhikari and G. H. Michler, "Polymer nanocomposites characterization by microscopy," *Polymer Reviews*, vol. 49, no. 3, pp. 141–180, 2009.
- P. Chaijareenont, H. Takahashi, N. Nishiyama, and M. Arksornnukit, "Effect of different amounts of 3-methacryloxypropyltrimethoxysilane on the flexural properties and wear resistance of alumina reinforced PMMA," *Dental Materials*, vol. 31, no. 4, pp. 623–628, 2012.

- M.M. Gad, S.M. Fouda, F. A. Al-Harbi, R.N<sup>a</sup>p<sup>a</sup>ankangas, and A. Raustia, "PMMAdenture basematerial enhancement: A review of fiber, filler, and nanofiller addition," *International Journal of Nanomedicine*, vol. 12, pp. 3801– 3812, 2017.
- F. Li, S. Zhou, B. You, and L. Wu, "Kinetic study on the UVinduced photopolymerization of epoxy acrylate/TiO2 nanocomposites by FTIR spectroscopy," Journal of Applied Polymer Science, vol. 99, no. 6, pp. 3281–3287, 2006.
- Dalvandi, M.; Ghasemi, B. Journal of Sol-Gel Science and Technology. 2013, 66: 68-72.
- Shahruz, N.; Hossain, M. Journal of World Applied Sciences. 2011, 12: 1981-1986.
- 9. Chaudhary, V.; Srivastava, A.; Kumar, J. *Mater. Res. Soc.* Symp. 2011, 1352.
- Thangavelu, K.; Annamalai, R.; Arulnandhi, D. International Journal of Emerging Technology and Advanced Engineering. 2013, 3: 636-639.
- G. Venkatesh Anehosur, R. D. Kulkarni, M. G. Naik et al, "Synthesis and Determination of Antimicrobial Activity of Visible Light Activated TiO2 Nanoparticles with Polymethyl Methacrylate Denture Base Resin Against Staphylococcus Aureus," *Journal of Gerontology&Geriatric Research*, vol. 01, no. 01, pp. 103–111, 2012.
- L. Ghahremani, S. Shirkavand, F. Akbari, and N. Sabzikari, "Tensile strength and impact strength of color modified acrylic resin reinforced with titaniumdioxide nanoparticles," *Journal of Clinical and Experimental Dentistry*, vol. 9, no. 5, pp. e661 e665, 2017.
- H. K. Aziz, "TiO-Nanofillers Effects on Some Properties of Highly- Impact Resin Using Different Processing Techniques," *The Open Dentistry Journal*, vol. 12, no. 1, pp. 202–212, 2018.
- S. Rashahmadi, R. Hasanzadeh, and S. Mosalman, "Improving the Mechanical Properties of Poly Methyl Methacrylate Nanocomposites for Dentistry Applications Reinforced with Different Nanoparticles," *Polymer—Plastics Technology and Engineering*, vol. 56, no. 16, pp. 1730– 1740, 2017.
- A. Sodagar, A. Bahador, S. Khalil, A. Saffar Shahroudi, and M. Zaman Kassaee, "The effect of TiO2 and SiO2 nanoparticles on flexural strength of poly (methyl methacrylate) acrylic resins," *Journal of Prosthodontic Research*, vol. 57, no. 1, pp. 15–19, 2013.
- 16. I. M. Hamouda and M. M. Beyari, "Addition of glass fibers and titanium dioxide nanoparticles to the acrylic resin

denture base material: comparative study with the conventional and high impact types," *Oral Health and Dental Management*, vol. 13, pp. 107–112, 2014.

- M. Ashour Ahmed, M. El-Shennawy, Y. M. Althomali, and A. A. Omar, "Effect of Titanium Dioxide Nano Particles Incorporation on Mechanical and Physical Properties on Two Different Types of Acrylic Resin Denture Base," *World Journal of Nanoscience and Engineering*, vol. 06, no. 03, pp. 111–119, 2016.
- G. Nazirkar, S. Bhanushali, S. Singh, B. Pattanaik, and N. Raj, "Effect of Anatase Titanium Dioxide Nanoparticles on the Flexural Strength of Heat Cured Poly Methyl Methacrylate Resins: An In-Vitro Study," *Journal of Indian Prosthodontist Society*, vol. 14, pp. 144–149, 2014.
- P. Harini, K. Mohamed, and T. V. Padmanabhan, "Effect of Titanium dioxide nanoparticles on the flexural strength of polymethylmethacrylate: An in vitro study," *Indian Journal of Dental Research*, vol. 25, no. 4, pp. 459–463, 2014.
- S. A. Alwan and S. S. Alameer, "The Effect of the Addition of Silanized Nano Titania Fillers on Some Physical and Mechanical Properties of Heat Cured Acrylic Denture Base Materials," *Journal of Baghdad College of Dentistry*, vol. 27, no. 1, pp. 86–91, 2015.
- M. A. Ahmed, A. A. Omar, M. El-Shennawy, M. I. Ebrahim, and Y.M. Althomali, "Influence of addition of different types of nano-fillers on the microstructure and mechanical properties of PMMA based denture resin," *Kasmera Journal*, vol. 45, pp. 48–59, 2017.
- W. Wang, S. Liao, Y. Zhu, M. Liu, Q. Zhao, and Y. Fu, "Recent Applications of Nanomaterials in Prosthodontics," *Journal of Nanomaterials*, vol. 2015, Article ID 408643, 11 pages, 2015.

- S. Rashahmadi, R. Hasanzadeh, and S. Mosalman, "Improving the Mechanical Properties of Poly Methyl Methacrylate Nanocomposites for Dentistry Applications Reinforced with Different Nanoparticles," *Polymer–Plastics Technology and Engineering*, vol. 56, no. 16, pp. 1730–1740, 2017.
- Anderson, M. A.; Gieselmann, M. J.; Xu, Q. J. Membr. Sci. 1988,39, 243.
- 25. O'Regan, B.; Gra<sup>-</sup>tzel, M. Nature 1991, 353, 737.
- Vorkapic, D.; Matsoukas, T. J. Colloid Interface Sci. 1999, 214,283 - 291.
- 27. Penn, R. L.; Banfield, J. F. Geochim. Cosmochim. Acta 1999, 63,1549.
- Barbe´, C. J.; Arendse, F.; Comte, P.; Jirousek, M.; Lenzmann, F.; Shklover, V.; Gra¨tzel, M. J. Am. Ceram. Soc. 1997, 80, 3157. Disadvantage is the liability of an acrylic resin denture to break during service (1).
- Gad MM, Fouda SM, Al-Harbi FA, Näpänkangas R, Raustia A. PMMA denture base material enhancement: A review of fiber, filler, and nanofiller addition. Int J Nanomedicine. 2017;12:3801–12. (PMC free article) (PubMed) (Google Scholar)
- Mohammed M. Gad 1 and Reem Abualsaud Behavior of PMMA Denture Base Materials Containing Titanium Dioxide Nanoparticles: A Literature Review International Journal of Biomaterials Volume 2019, Article ID 6190610, 14 pages. https://doi.org/10.1155/2019/6190610
- Franklin P, Wood DJ, Bubb NL. Reinforcement of poly (methyl methacrylate) denture base with glass flake. Dent Mater. 2005;21:365-70.
- 32. Durkan RK, Özdemir T, Pamir AD, Usanmaz A. Water absorption of two different denture base resins reinforced with dental fibers systems. J Appl Polym Sci. 2010;117:1750-3.