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Original article

The Impact of Incorporating Silicon Dioxide Nanoparticle Fillers into Polymethyl Methacrylate Denture Base Material on Flexural Strength

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Abstract
Several previous studies have incorporated NPs-SiO2 at various concentration percentages in heat-cure PMMA to increase the mechanical properties of the denture base. Many researchers found out that adding NPs-SiO2 at low concentrations has shown enhancements in mechanical properties compared to higher concentrations percentages. This study was conducted to examine the impact of adding Silicon Dioxide as a nanoparticle filler at diver's concentrations in heat-cured PMMA denture bases on flexural strength. Fifty specimens with dimensions (65 x 10 x 2.5 mm3) were fabricated from heat-cure-PMMA, and then specimens were divided into 5 groups according to different concentrations of NPs-SiO2. Each group consisted of 10 specimens. Moreover, 10 samples were prepared as a control group without any additives of NPs-SiO2 to PMMA. Three-point bending test was carried out using a universal testing machine to measure the flexural strength of specimens. Data analyses were conducted through analysis of variance and Tukey's post hoc test (a = 0.05). The one-way ANOVA showed that there were statistically significant differences among groups (p<0.05). The highest flexural strength value was observed for the group (S1), with concentrations 0.5% by weight of NPs-SiO2 into PMMA. The control group showed a lower value of flexural strength than other groups. Low concentration percentages of NPs-SiO2 added to PMMA could increase the flexural strength of the PMMA denture base. According to the current investigation, 0.5% was the optimal concentration percentage for adding NPs-SiO2 to PMMA while maintaining an appropriate flexural strength value.
Keywords. *Silicon Dioxide, Nanoparticles Filler, Polymethyl Methacrylate, Flexural Strength, Denture Base.*

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Introduction

Polymethyl Methacrylate (PMMA) has been utilized in dentistry since the 1930s and conventionally is available in the form of a powder and liquid system [1,2]. The powder contains a Benzyl Peroxide initiator, a plasticizer (dibutyl phthalate), opacifiers (Titanium and Zinc oxide), fibers, and pigments. The liquid component contains Methyl Methacrylate (MMA) Monomer, Ethylene Glycol Di methacrylates, a cross-linking agent, and hydroquinone as an inhibitor [3].

PMMA is widely used for several dental applications such as removable applications and orthodontic appliances [1]. PMMA obtained its popularity in dentistry not only for the construction of denture bases but also for dental maxillofacial prostheses, temporary crowns, and bridges. Moreover, its superior advantages include low cost, lightweight, good aesthetics, ease of manipulation and handling, dimensional accuracy, biocompatibility, satisfactory physical and mechanical properties, well adhesion to artificial teeth, and ease to repair [4-6].

On the other hand, there are various disadvantages associated with using PMMA for dental applications, such as the fracturing of dentures which is commonly reported in dental clinics and laboratories [5,7]. Denture base materials are exposed to repeating stresses of the mastication process in the oral cavity and dropping causes the fracturing of denture due to the weakness of PMMA's mechanical properties, especially flexural strength, hardness, and impact strength [8]. This leads to the failure of PMMA appliances [9].

The mechanical properties of PMMA are still insufficient to meet the ideal characteristics required to construct denture bases and other applications as well [10]. For that reason, PMMA denture bases should have further improvement in their mechanical properties to withstand the forces of

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mastication without breaking the denture base. *Hassan, M., et al 2019* stated that the denture base should have a high flexural strength to withstand the mastication forces without permanent deformation or fracture [4]. Therefore, many researchers have attempted a variety of modifications to reinforce and overcome the limitations of PMMA's properties by adding nano particular fillers into PMMA denture bases such as silicon dioxide, fibers, Zirconium Oxide, titanium dioxide, zinc oxide, or hydroxyapatite [9-11]. Therefore, nanofillers can improve the flexural properties of PMMA, as well as provide resistance to an oral environment that causes fatigue and cracking of denture base [12]. Furthermore, the mechanical properties of denture base PMMA can be affected by many factors, such as the curing method, chemical composition, degree of polymerization including nano particular filler's size, shape, concentration, and distribution into resin matrix [12,13]. Also, the salinization of the surface of the particular filler by a coupling agent increases the bonding strength between the inorganic fillers and the organic matrix [9]. The applied load must be transferred from the matrix to the fillers via this interfacial connection, as this bonding could greatly enhance the mechanical qualities of dentures. Moreover, the joined interface between the inorganic filler and the organic matrix could decrease the water sorption and increase stability under wet and humid conditions [14].

Although previous studies have been modified by the addition of silicon dioxide as nano particular fillers (NPs-SiO2) with different concentrations to PMMA, *Alnamel and Mudhaffer, (2014)* stated that the addition of (NPs-SiO2) into PMMA could improve the surface hardness, thermal and mechanical properties, as well as transverse and impact strengths [15]. The mechanical properties of adding NPs-SiO2 into PMMA depend on the shape, size, and concentrations and the distribution of particles filler in the resin matrix [16,17].

Emad Azmy., et al 2022 and al *Balos S., et 2014* discovered that adding (wt 3%) NPs-SiO2 greatly improves the PMMA acrylic resin's flexural, impact, and wear resistance. Otherwise, it is advised to add NPs-SiO2 to PMMA at low concentrations to increase durability, strength, and the elastic modulus of a PMMA denture base resin [18,19]. *Alzayyat., et al 2022* concluded that the ideal concentration of NPs-SiO2 to incorporate into PMMA in order to potentially improve the flexural and elastic modulus capabilities of modified denture base resins is at low concentrations [20]

The purpose of this study to evaluate the effect of adding Silicon Dioxide (SiO2) as a Nanoparticle filler material with different concentrations on the flexural strength of heat-curing PMMA.

Methods

Fifty specimens were fabricated according to ISO specification (1567:1999), the specimen's size measurements were (65 x 10 x 2.5 mm3) in length, width, and thickness. The specimens were divided into (5 groups) according to concentrations of NPs-SiO2 (0%, 0.5%, 1%, 1.5%, and 2%) and each group consisted of 10 specimens. Moreover, 10 samples were prepared as a control group without any additive of (NPs-SiO2) in PMMA as shown in Table 1. All materials that are used in this study are listed in table 2.

Table 1. The specimen groups according to concentrations of NPs-SiO2

Groupe	Number of samples	Concentrations of SiO2
Control	10	0%
S1	10	0.5%
S2	10	1%
S3	10	1.5%
S4	10	2%

Table 2. Materials used in the study

Material	Material content	Manufacturer
(Acrylic denture base)	PMMA: Powder400 gm, (Heat-curePolymethyl Methacrylate)	Pyrex, India
Monomer	Liquid 200 ml, MMA, methacrylate Mixing ratio: (2.5 g powder/1 ml liquid)	Pyrex, India
SiO2 nanoparticles	(White; 98.5% purity; average size: 15±3 nm; specific surface area: 150–550 m2/g; and density: 2.2 g/cm3)	NanoGATE, Ciro, Egypt
Silane coupling agent	Sigma-Aldrich ChemieGmbH Riedstrasse2, Purity 98%, Ethanol 99.7%, lot no. 440159, Acetone solvent 95% CAS no 67641.	Germany

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Silanization of NPs-SiO2

In the current study, NPs-SiO2 was silanized with 5% silane coupling agent 3-(tri methoxy silyl) propyl methacrylate, (98%; γ-MPS) and acetone solvent 95% to enhance the chemical bonds between the surface of NPs-SiO2 and the PMMA matrix, following the method and steps which were described by previous studies [21,22].

Mixing of different ratios of NPs-SiO2 with PMMA and MMA Monomer

A digital electronic balance (Wedo Picco1000, Electronic Digital Scales Lab, China) was used to weigh the NPs-SiO2 and PMMA powder. NPs-SiO2 was incorporated at 0.5%, 1%, 1.5%, and 2% by weight to (PMMA) powder. Each concentration of nano-SiO2 was separately mixed with the PMMA powder at 400 rpm for 20 minutes to distribute it within the PMMA powder, then the mixture of PMMA powder and NPs-SiO2 with different concentrations was independently mixed with monomer in a ratio of (2.5ml:1g) according to the manufacturer's recommendation as shown in table 3. After that, the conventional flashing procedure for complete dentures was done, packing method and heat-polymerization by immersing all the flasks in a water bath for 8 hours at 75°± 1°C, and then the deflating procedure was done. Finally, the finishing and polishing of specimens done. All the specimens were stored in water at 37°C for one week before flexural strength testing.

Table 3. Mixing of NPs-SiO2, MMA monomer, and PMMA with different ratios by weight

SiO2 powder	PMMA powder	MMA monomer
0%	50g	20ml
0.5%	49.5g	20ml
1%	49g	20ml
1.5%	48.5g	20ml
2%	48g	20ml

The testing procedure of Flexural strength

The sample thickness and width were measured with a micrometer device with dimensions of (65 x 10 x 2.5mm3) before the flexural strength test. The distance between supports is set to 50 mm apart. Three-point bending test was carried out using a universal testing machine (Tokyo Testing Machine, Japan), applying a load to the specimen at a cross-head speed of 1mm/min until the specimen fractures. The maximum load exerted on the specimen is recorded. Flexural strength is calculated according to the following equation:

$$\sigma = 3FL/2BD2$$

Where

σ is the flexural strength (MPa), F is the load at the fracture point (N), L is the length of the support span (mm), b is the width of the specimen (mm) and d is the depth or thickness of the specimen (mm) [23].

Statistical Analysis

Calculated values for the parameters test were statistically analyzed using IBM SPSS Statistics version 26.0 software (IBM Corporation, Armonk, NY, USA). Furthermore, the Shapiro-Wilk test was used to analyze, whether the variables were normally distributed or not. Then, the homogeneity of variance F-test (one-way ANOVA) to test the significant differences between groups and within groups on flexural strength at different concentration ratios.

Results

The current study includes one major hypothesis, which is the addition of Silicon Dioxide (SiO2) as a Nanoparticle filler material with different concentrations increases the flexural strength of the heat-curing PMMA denture base.

According to table 4, the variables were normally distributed because the values of the Shapiro-Wilk test were not significant ($p > 0.05$).

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Table 4. Means, Medians, Standard Deviations, and Test of Normality

Variables	Mean	S.D.	Shapiro Wilk value	Df	Sig.
%0	70.47	2.60	.968	10	.874
%0.5	97.15	1.27	.919	10	.351
%1	93.57	1.38	978	10	.877
%1.5	88.41	1.86	.944	10	.596
%2	83.01	1.12	953	10	.699

The researcher used the F-test one way (ANOVA) as shown in table 5, to analyze the significant differences between groups at low concentration ratios of (NPs-SiO2) on flexural strength. The result of one-way ANOVA test showed statistically significant differences among groups on flexural strength because the value of ($F = 254.344$, $p < 0.001$). Tukey's post hoc test was used to compare the differences between groups as shown in table 6.

Table 5. Result of One-Way ANOVA Test (The Effects of Groups on Flexural Strength)

Source of variance	Sum of Squares	Df	Mean Square	F	Sig
Between Groups	4363.3	4	1090.8	361.9	.000
Within Groups	135.6	45	3.0		
Total	4498.9	49			

The comparisons of the differences between specimen groups on flexural strength were estimated with Tukey's post hoc tests, generally, there were significant differences between the values for the control group and SiO2 nano particles groups, whereas p-values were less than (0.05). The means, standard deviations, and significance levels of flexural strength for low (NPs-SiO2) concentrations were stated in Table 6. The results showed that all groups got higher significance on flexural strength compared with the control group ($p < 0.001$).

The control group showed the lowest values of flexural strength than other groups had, with mean and standard deviation (70.47 ± 2.60 MPa), the highest flexural strength values were observed in group (S1), with concentrations 0.5% by weight of NPs-SiO2 in PMMA, with mean and standard deviation (97.15±1.27 MPa), the group which got the second higher values of flexural strength was (S2), with concentration 1% by weight of NPs-SiO2, with mean and standard deviation (93.57 ±1.38 MPa), after that, the group (S3, with concentration 1.5% by weight of NPs-SiO2, with mean and standard deviation (88.41 ±1.86 MPa), and the last group was (S4, with concentration 2% by weight of NPs-SiO2, with mean and standard deviation (83.01 ±1.12 MPa).

Table 6. The Result of the Post Hoc Test to check the differences Among Groups

Groups	0.5	1%	1.5%	2%	Control
0.5%	—	3.57	8.73	14.14	-26.68
1%	-3.57	—	5.16	10.56	-23.10
1.5%	-8.73	-5.16	—	5.40	-17.94
2%	-14.14	-10.56	-5.40	—	-12.54
Control	-26.68	-23.10	-17.94	-12.54	—

Discussion

The results of this study found that the conventional heat-cure PMMA denture base cause agglomeration of NPs-SiO2 in resin matrix. Thus, it might influence and reduce the flexural strength value in heat-cure acrylic resin.

Al-Thobity and Gad 2021 reported that the addition of low concentrations of nano-SiO2 improved the flexural strength of the heat-cure PMMA denture base. In addition, they have been suggested that the addition of lower concentrations of less than 1% of nano-SiO2 significantly increases the flexural strength of the PMMA resin base. Therefore, the increase in flexural strength may be imputed to the distribution of homogeneous distribution of SiO2 at low concentrations [27]. Moreover, *Salman et al 2017* stated that the increasing concentrations percentage from 1% and up caused agglomeration of NPs-SiO2 due to the influence affinity of NPs to aggregate, however, nano-fillers are hardly scattered in resin matrix with conventional techniques to be

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homogeneous [26]. *Supova M., et al 2010*, concluded that the addition of high concentrations of NPs-SiO2 in PMMA denture base could potentially lead to agglomeration of fillers into resin matrix due to the reaction caused by the van der Waals forces and then effective the physical and mechanical characteristics [28].
Salman et al., 2017 obtained a limited increase in flexural strength through the addition of low concentrations of nanoparticles. Otherwise, the higher concentrations of SiO2 decrease the flexural strength value [26]. *Alabadi., et al 2023* specified that the best value percentages in order to obtain the highest strengths for NPs-SiO2 were (0.05wt%) for impact strength and (0.1wt%) for flexural strength. As result, more agglomerations on the surface of the specimens caused lower mechanical properties of nano-SiO2 reinforced groups. For instance, the higher concentrations of nano-SiO2 indicate a higher volume and lower density of nano-SiO2 in the resin matrix with more agglomeration. Therefore, lower concentrations of nano-SiO2 were recommended for NPs-SiO2 than higher concentrations [24].
Moreover, the silanization of nano-fillers with coupling agents is commonly used in denture base applications. Coupling agents act as chemical bonds between the NPs-SiO2 and polymer matrix resin. These bonds significantly affect the mechanical properties and transfer the applied load from the matrix to fillers. The joined interface between NPs-SiO2 and polymer matrix could decrease the water sorption and increase stability under wet and humid environment [14]. *Al-Thobity and Gad 2021* found that the values of the flexural strength of SiO2 nano particle-reinforced heat-polymerized acrylic resin depend on the concentration by weight, dimension, geometry, and silanization of nanoparticles [27].

Conclusion

The flexural strength value of the PMMA denture base could be improved by means of the addition of NPs-SiO2 at low concentration percentages into PMMA; In other words, the flexural strength values of PMMA significantly decrease when higher concentrations percentages of NPs-SiO2 added into PMMA. The current study finds that the ideal concentration percentage of addition of NPs-SiO2 to PMMA with acceptable flexural strength value is 0.5%.

Conflict of Interest

There are no financial, personal, or professional conflicts of interest to declare.

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المستخلص

قامت العديد من الدراسات السابقة إضافة جسيمات نانوية بتراكيز مختلفة من ثاني أكسيد السيليكون كمادة حشو مع راتنج بي إم إم أي المعالج بالحرارة لتصنيع قاعدة طقم الأسنان، بهدف تحسين خواصه الميكانيكية. كما أظهرت نتائج العديد من الدراسات، أن إضافة جسيمات الحشو النانوية منخفضة التركيز من ثاني أكسيد السيليكون تؤدي إلى تحسن ملحوظ في الخواص الميكانيكية لمادة الراتنج بي إم إم أي مقارنة بالتراكيز الأعلى. هدفت هذه الدراسة إلى فحص تأثير إضافة جسيمات الحشو النانوية من ثاني أكسيد السيليكون بتراكيز مختلفة إلى مادة راتنج بي إم إم أي المستخدمة في صناعة قواعد أطقم الأسنان المعالجة بالحرارة، على قوة الانثناء. حيث تم تحضير 50 عينة بأبعاد (65 × 10 × 2.5 مم^3) من مادة راتنج بي إم إم أي المعالجة بالحرارة، وتم تقسيم العينات إلى خمس مجموعات حسب التراكيز المختلفة لجسيمات الحشو النانوية من ثاني أكسيد السيليكون. كل مجموعة اشتملت على 10 عينات، بالإضافة إلى 10 عينات كمجموعة ضابطة خالية من أي إضافات من ثاني أكسيد السيليكون. تم إجراء اختبار الانحناء ثلاثي النقاط لقياس قوة الانثناء للعينات. كما تم تحليل البيانات إحصائيا باستخدام تحليل التباين واختبار Tukey's post hoc ($\alpha = 0.05$). أظهر تحليل التباين الأحادي (ANOVA) وجود فروق ذات دلالة إحصائية بين المجموعات ($P<0.05$) وسجلت أعلى قيمة لقوة الانثناء في المجموعة (S1) التي تحتوي على تركيز 0.5% من جسيمات الحشو النانوية من ثاني أكسيد السيليكون المضافة إلى راتنج بي إم إم أي. في المقابل، أظهرت المجموعة الضابطة قيمة أقل لقوة الانثناء مقارنة بالمجموعات الأخرى. تشير النتائج إلى أن النسب المئوية المنخفضة من جسيمات الحشو النانوية من ثاني أكسيد السيليكون يمكن أن تحسن قوة الانثناء لقاعدة طقم الأسنان المصنوعة من راتنج بي إم إم أي. ووفقاً للبحث الحالي، فإن النسبة المثلى لتركيز الجسيمات الحشو النانوية من ثاني أكسيد السيليكون كانت 0.5%، حيث تحققت عندها قيمة قوة انحناء مناسبة.