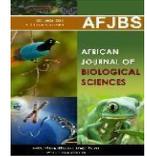




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Evaluation of The Flexural Strength of Heat-cured Acrylic Resin Modified with Titanium Dioxide/Quercetin-Nanocomposite Particles

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ABSTRACT

Objective: evaluation of the flexural strength of heat cured acrylic resin that incorporated with different concentrations of (Titanium dioxide/Quercetin) nanocomposite particles by comparing with conventional heat cured acrylic resin.

Materials and methods: flexure strength test is applied to a total of twenty specimens that are divided into four main groups according to the percentage of the added Nano material used, then Scanning of the fractured surface by electron microscope.

Results: Regarding the flexural strength there was a statistically remarkable decrease for all modified groups compared with the control group.

Conclusion: The concentration of nano (TiO₂/Quercetin) added to PMMA significantly affected the mechanical properties of PMMA. Flexure strength has not significantly improved in the modified groups when compared to the control group.

KEY WORDS: heat cure resin, PMMA, flexure strength, Nano, TiO₂, Quercetin, electronic microscope

INTRODUCTION

Acrylic resin (PMMA) has been widely used in dental field since it developed by Walter in 1936, due to its good characteristics as low cost, ease of construction, polishability, acceptance of repair, excellent aesthetics, and biocompatibility. Despite that, it has some drawbacks such as low surface hardness, insufficient flexural strength, and poor antimicrobial activity that causing denture stomatitis and abutment caries.(1)

Complete denture is one of the important dental prostheses for completely edentulous patients. It enhances mastication, phonetic problems and aesthetic that improves the psychological condition of the patient.(2)

the fillers size, shape, distribution, and adhesion at the PMMA matrix, has an important role in the mechanical properties of the PMMA.(3)

Reinforcement of PMMA with metal oxides also improved the physical properties of the material, as well as patients' sensation of hot and cold stimuli that cause better food sensation and healthier oral mucosa. (4)

Recently, incorporation of natural organic substances in denture base acrylic resin improves inflammation treatment with varying success. (5)

Nanofillers incorporation mostly improve PMMA properties. huge surface area, and homogenous distribution of nanoparticles improving the physical and mechanical characteristics of PMMA compared with pure PMMA. (4)

TiO₂ nanoparticle has recently been used due to their high stability, availability, white colour, and low cost. TiO₂ NPs also are chemically inert and non-toxic and have corrosion resistance as well as good hardness and antibacterial activity under a wide microbial spectrum.(6)

It was found that adding TiO₂ nanoparticles has improved flexure strength, hardness, and thermal conductivity of PMMA. As well as increase in impact strength and a significant decrease in water sorption were found upon addition of TiO₂ to PMMA.(4)

On the other hand, other studies found that TiO₂ has decreased the flexure strength of denture acrylic resin, that may be due to clustering of the nanoparticles within the resin matrix, that decreasing the mechanical properties of PMMA.(4)

Quercetin, this name comes from Quercus that mean:(oak forest tree), quercetin is found in plants like apples, grapes, onions, and tea as well seeds like nuts, flowers.(7)

Quercetin displays activity against streptococcal membranes as well as Streptococcus-mutans by inhibiting acid production in biofilm. It also showing inhibitory activity against Staphylococcus aureus. (8)

Quercetin also has a good antimicrobial effect against candida that is the causative organism of denture stomatitis (9)

Quercetin is a polyphenolic flavonoid that mainly is found in fruits and vegetables. It also has antioxidant, antiatherogenic, anti-inflammatory, anti-carcinogenic, neuroprotection properties, antibacterial, and antiviral properties. (10)

Titanium surface of implant was coated with quercetin (QUE), the obtained compound (Ti-Que) was found that able to reduce the biofilm formation of Staphylococcus mutants and adhesion compared to the uncoated titanium surface. (11)

Complete dentures are subjected to high flexural stresses mainly during the mastication, which cause cyclic deformation of denture base PMMA, resulting in crack propagation and formation that caused denture fracture. So that , high flexural strength is important to denture success.(12)

Due to the irregular pattern of bone resorption, dentures are supported by uneven alveolar ridges that cause high flexural stresses in denture base material under flexural loading. (13)

According to ISO 20795-1 (international standard of denture base polymer), the denture base material (PMMA) should have flexural strength not be less than 60 MPa.(14)

Materials and Methods

1.1. Study design.

This Study was designed as an experimental in vitro controlled study. The study was carried out in the removable prosthodontic department - Faculty of Dental Medicine – al Azhar university (Assiut branch).

1.2. Samples grouping

To evaluate the effectiveness of adding TiO₂/Quercetin nanoparticles to heat cured PMMA, a total of 20 specimens were divided into four main groups according to the percentage of added nanoparticles (Five samples in each group).

Group 1:Control group –heat cured PMMA.

Group 2:Nanoparticles added to heat cured PMMA by 0.5% of weight.

Group 3:Nanoparticles added to heat cured PMMA by 1% of weight.

Group 4: Nanoparticles added to heat cured PMMA by 1.5% of weight.

2.1. Materials

The Materials that have been used in this study along with relative information (description and manufacturers) are:

- Heat cured poly methyl methacrylate (*Acrostone Dental & medical supplies, Cairo, Egypt*).
- Nanoparticles of Titanium dioxide/Quercetin (*Nano Gate company, Cairo, Egypt*)

2.2. Mixing nanoparticles with PMMA

The dried powder of (Qu loaded TiO₂ NPs) was mixed with PMMA powder at different ratios 0.5%, 1% and 1.5% by weight, typically for 1%, 1 gm of TiO₂/Quercetin NPs mixed with 99 gm of PMMA by meaning of ball mill (Retsch pm 400, Japan, Nano Gate company) for 2 h to get homogenous mixing.

3.1. Samples fabrication

To fabricate heat cured acrylic resin samples with standard dimensions according to ISO standards, a virtual design was made with the help of a computer program (Tinkercad, free web app for 3D design, Autodesk) according to ISO standard dimensions and then converted into a stereolithography (.stl) file, which then printed to resin model by 3D printer that shown in (Fig.1).

Resin models of rectangular shape were converted to heat cured PMMA samples for flexural strength test, The resins were processed by the conventional water bath technique in this research.

The specimens were cut out and trimmed to final dimensions, using finishing stones. A digital calliper (TMT331501; Total, China) was used to ensure the final dimensions of the specimens (Fig.2).



Fig.1: 3D printed model of the specimens



Fig.2: 3.3mm thickness, 64 mm length and 10 mm width are dimension of flexure strength test and hardness test specimens.

3.2. Flexural strength test:

Dimension of **64 mm length, 10.0±0.2 mm width and 3.3±0.2mm height** to conduct the flexural strength test based on the recommendation of **ISO 20795-1:2013**.(14)

A total of 20 samples were tested using a universal testing machine (INSTRON, Dental material lab, Assuit university, Egypt).

A standard three-point load flexural test was used to fracture the specimens, after mounting the specimens in the testing device apparatus with 50 mm distance between two vertical supports, the vertical load was applied midway between the supports with a 5 mm/min crosshead speed until the specimen was fractured, and the maximum load at fracture was recorded with the PC software (**Fig.3**).

After flexure strength test, features of the fractured surfaces of each test group were observed using field emission scanning electron microscope.

Flexural strength was calculated using the formula:

$$FS = 3WL/2bd^2$$

FS: flexure strength (MPa)

W: the maximum load before fracture (N)

L: the distance between supports (50 mm)

b: the specimen width (10 mm)

d: the specimen thickness (3.3 mm)

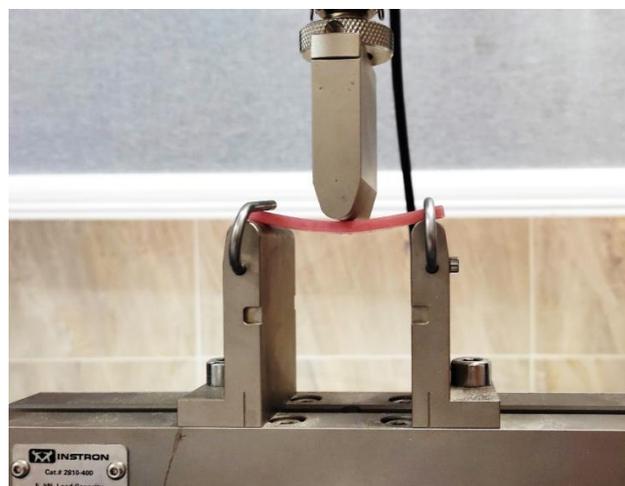


Fig.3: samples under three-point loading during flexural strength test

4.1. Electron microscope scanning (SEM)

Fractured surfaces of the specimens of control, 0.5%, 1%, and 1.5% groups after flexure strength test were randomly selected, gold-sputter coated by (JEOL JFC-1100) and observed using scanning electron microscope SEM (JEOL Scanning electron microscope JSM-5400) under magnification of (350x, 1000x) at unit of Electron Microscope Scanning, Assiut University.

The fractured surface of the specimens was studied to explain the failure mechanism. Specimens were mounted onto aluminium stubs and sputter-coated with gold to overcome the non-conductive nature of the material.

Images were taken at different magnifications (350x, 1000x) for better visual inspection, and to determine the nature of the failure.

Smooth surface fields present brittle fracture modes, while a rough appearance presents intermediate (brittle to ductile) fracture modes.

4.2. SEM analysis displayed:

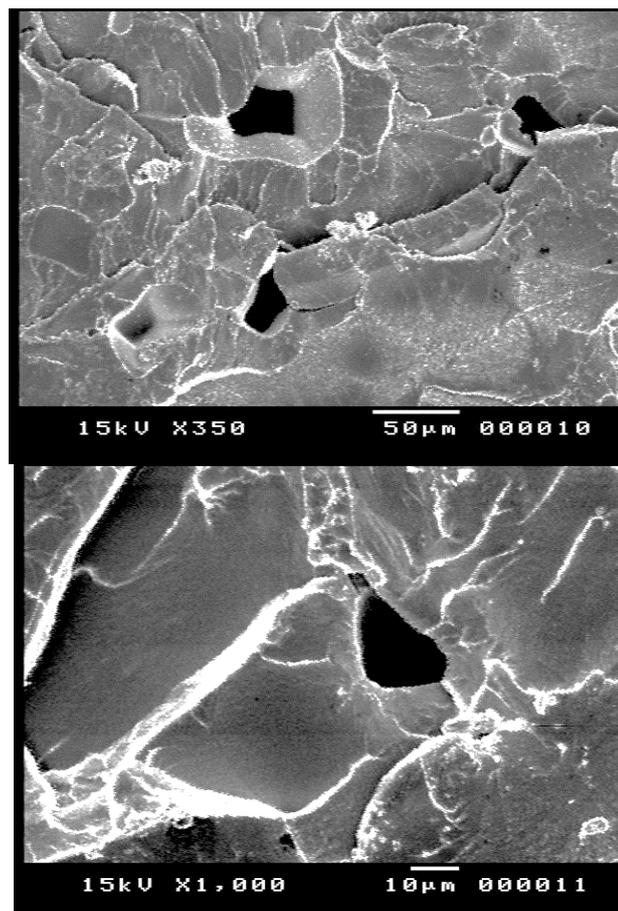


Fig.4: control sample scanned image by electron microscope at 350x and 1000x

shows a compact morphology containing broad scattered voids with irregular compact surface and show flake or sheetlike structure of certain thickness.

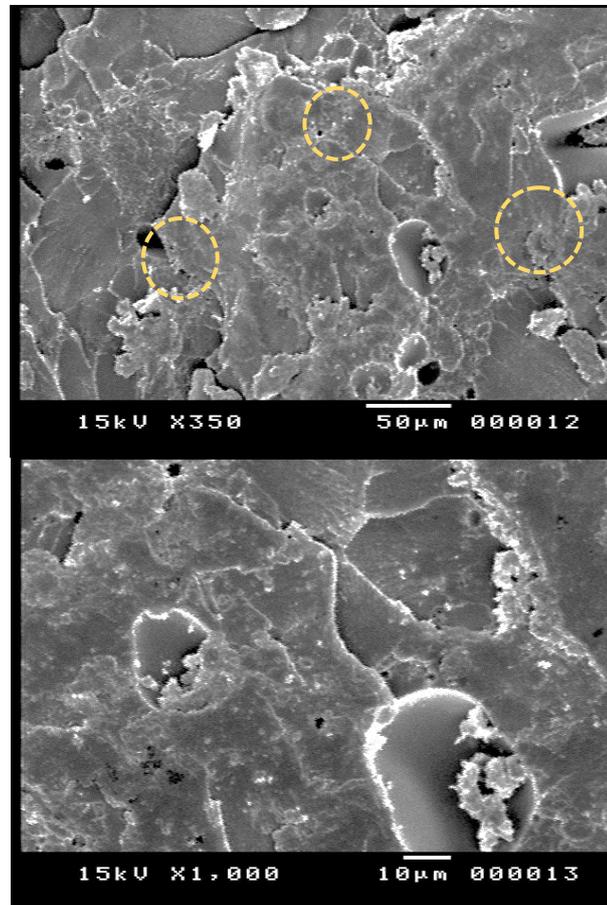
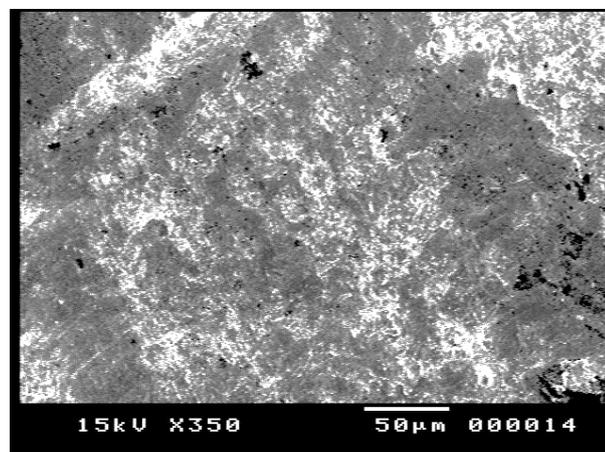


Fig.5:(0.5%) scanned image by electron microscope at 350x and 1000x

shows a compact morphology with diminutive pores preoccupied by nanomaterials (yellow circles).



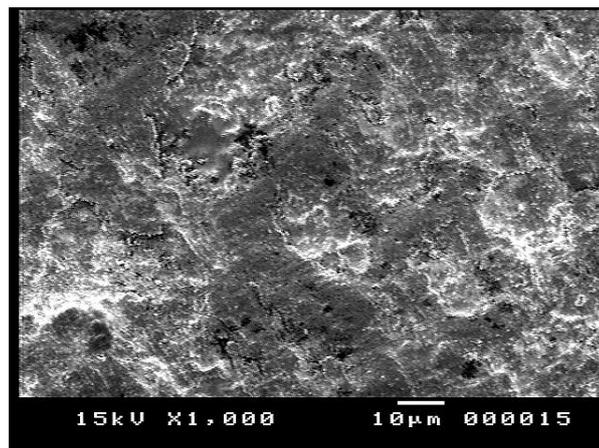


Fig.6:(1%) scanned image by electron microscope at 350x and 1000x shows a smooth surface resulting from the role of nanoparticles filling the voids.

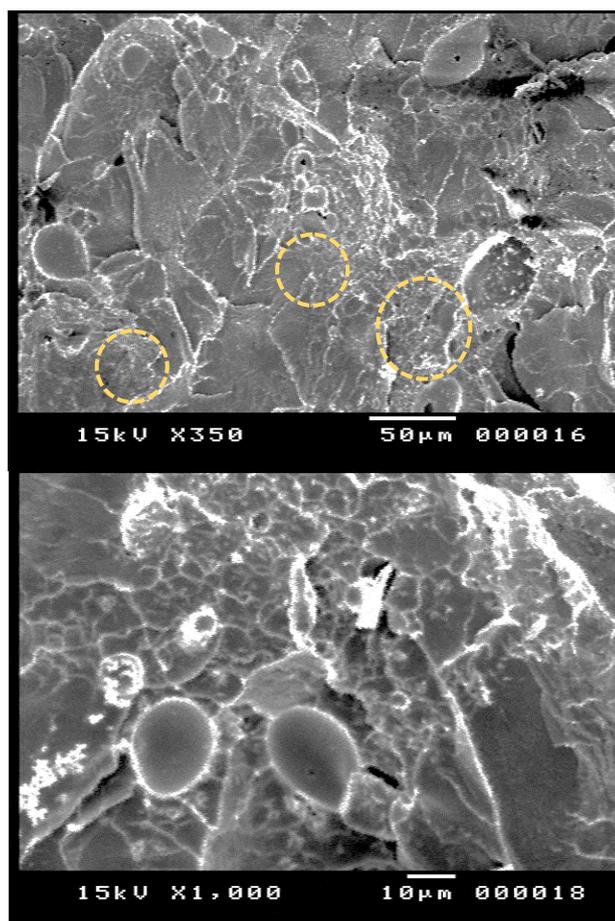


Fig.7:(1.5%) scanned image by electron microscope at 350x and 1000x shows loosely attached clusters of nanoparticles on the surface of specimens. the distribution

of the nanoparticles was not uniform, and the fillers tended toward agglomeration (*yellow circles*).

- The nanoparticles appeared as bright points in the acrylic resin matrix.
- Void formation shown in **Fig.4** may be due to entrapped air and moisture, incomplete wetting of the fillers by the resin, difficulty of obtaining good adhesion between fillers and the resin matrix and the fact that nanoparticles act as an interfering factor in the integrity of the polymer matrix.
- Microporosities may be resulting from an inadequate polymer monomer ratio, distinct stages of the mass during the packing process, and inadequate pressure on the mass inside the flask during the polymerization procedure.
- Although the nanoparticles were in the nanoscale, the electron microscope images showed that some nanoparticles had some agglomeration and were still in the nanoscale.
- This agglomeration may be due to the tendency of nanoparticles to decrease their contact surface with PMMA. Good distribution of nanoparticles in PMMA matrix help improving the nanocomposite properties.

Results

One WAY ANOVA showed a significant difference between tested groups at $p=0.002$. The highest flexure strength resulted for control which was insignificantly higher than 0.5% concentration only and significant with other tested groups.

Table (1): Descriptive statistics of flexure strength test results showing mean values, standard deviations, and statistical significance.

Groups	Mean	Sd	Range		P value
			Min	Max	
Group 1 (control)	99.81	5.69	93.1	108.3	<0.001*
Group 2 (0.5% wt Nano)	93.42	7.84	85.7	106.2	
Group 3 (1% wt Nano)	82.44	5.35	77.3	91.1	
Group 4 (1.5% wt Nano)	75.76	4.01	71.3	79.7	

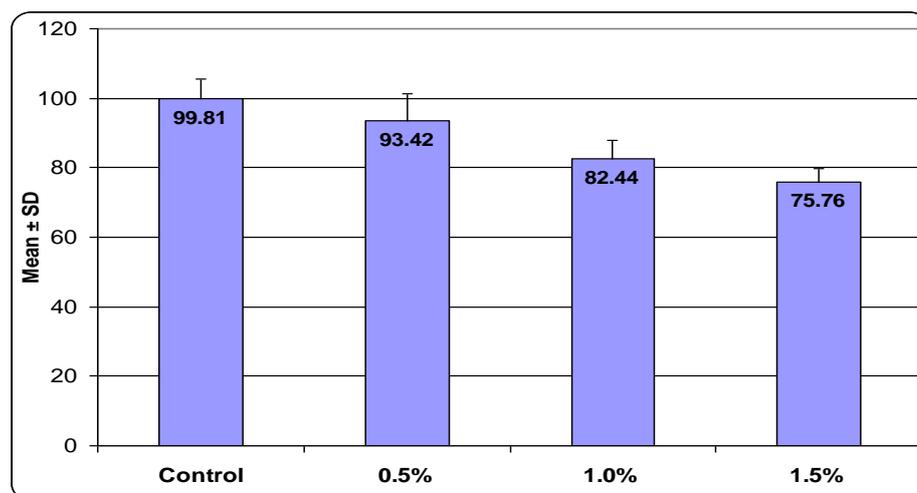


Fig.8:Column chart showing flexure strength mean values and standard deviation for nano modified groups compared to control group.

Discussion

different materials have been used for construction of denture base, but polymethyl methacrylate (PMMA) still used for more than 80 years. PMMA has several advantages as ease of manipulation and repair, accurate fit, ease of polishing, aesthetic appearance, and stability in the oral environment. However, PMMA also has disadvantages, as low hardness and poor wear resistance.⁽¹⁵⁾

Literature has found that reinforcement by nanoparticles of Titanium dioxide brings new optical, physical and mechanical properties at small TiO₂ concentrations, which makes PMMA-TiO₂ nanocomposites a promising new material.⁽¹⁶⁾

Moreover, TiO₂ nanoparticles have been used as additives to biomaterials to improve antimicrobial properties.⁽¹⁶⁾

Quercetin exists in many vegetables and fruits including apples, tea, onions, red grapes, and tomatoes as it is the most abundant flavonoid. Quercetin has good activity against wide spectrum of bacteria, such as *Staphylococcus aureus*, *Escherichia coli* and *Pseudomonas aeruginosa*.⁽¹⁷⁾

Previous studies demonstrated that quercetin could affect the bacterial cell surface properties to decrease biofilm formation. As well as, it may be beneficial to decrease periodontal tissue destruction by inhibiting activity of *P. gingivalis*.⁽¹⁷⁾

As nanoparticles have a high surface energy, uniform distribution at the polymer matrix can be a difficult challenge, due to its tendency to aggregate.⁽¹⁸⁾

The results of this study showed a not significant decrease in flexural strength by adding (0.5% TiO₂/Quercetin nanocomposite) to PMMA resin and significant decrease in the other groups from control.

Denture base is subjected to compressive, tensile, and shear stresses during function, and fractures may develop due to repeated masticatory force. Increase in flexural strength is required to prevent the fracture of denture resulting from long term use or heavy occlusion.⁽¹⁵⁾

The concentration of nanoparticles was inversely proportional to the flexural strength of PMMA, It was noted that all the groups showed flexural strength more than the minimum requirement of flexural strength for denture base according to the ISO specification i.e., 65 MPa.⁽¹⁹⁾

Incorporated nanoparticles can also aggregate and agglomerate, that creating a stress concentration centre and lead to poor mechanical properties.⁽¹⁹⁾

On the other hand, higher concentrations of nanoparticles may cause agglomeration, cluster formations, and loss of homogeneity.⁽²⁰⁾

As well as, Residual monomers may act as a plasticizer, and negatively influence the mechanical properties of the denture base PMMA.⁽²¹⁾

The surface of the nanoparticles must be modified with coupling agents, which creates a strong bond with the resin matrix. A silane coupling agent is required to fix the fillers in the resin matrix⁽¹⁸⁾

A silane coupling agent is required to retain fillers in the resin matrix and to form chemical bonds with inorganic fillers and organic matrix .⁽¹⁸⁾

Conclusion

Based on the findings of this in vitro study, the following conclusions indicate that: The flexural resistance property did not improve in the modified groups when compared to the control group.

Recommendation

The authors recommend further studies assessing the use of different acrylic resins, different concentration of loading the nanomaterial, adding nanofillers to PMMA using silane coupling agent.

limitations

The flat surface of the tested specimens, which did not mimic the actual denture anatomy completely but would allow for comparison with previous reports.

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