

STRONTIUM TITANATE NANOPARTICLES MODIFIED DENTURE BASE MATERIALS

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ABSTRACT

Objectives: The current study was intended to evaluate addition of different concentrations of strontium titanate nanoparticles to denture base materials regarding: surface roughness, impact strength, water sorption and solubility.

Methods: Polyamide and heat-cured acrylic resin were used and both were modified with strontium titanate with these concentrations: 0 wt%, 0.5wt%, 1wt%, 1.5wt%. Surface roughness, impact strength, water sorption and solubility were evaluated for all specimens. Data were statistically analyzed.

Results: The findings of this study showed that for surface roughness, the highest value was for flexible resin with 1.5 wt% while the lowest value was for heat cured acrylic resin with 0 wt %, for impact strength, the highest value was for flexible resin with 1.5 wt% while the lowest value was for heat cured acrylic resin with 0 wt %, for water sorption and solubility, the highest value was for heat cured acrylic resin with 0 wt %, while the lowest value was for flexible resin with 1.5 wt%.

Conclusions: Strontium titanate nanoparticles have increased surface roughness of both resins as the concentration increase. For impact strength, water sorption and solubility, strontium titanate nanoparticles gives better effect with both polyamide and heat cured acrylic resin as the concentration increase, leading to increased impact strength, while decreased water sorption and solubility. Polyamide has higher surface roughness than heat cured acrylic resin. Polyamide has higher impact strength than heat cured acrylic resin. Polyamide has lower water sorption and solubility than heat cured acrylic resin.

KEYWORDS: Denture base materials, strontium titanate, nanoparticles, mechanical properties

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INTRODUCTION

Restoration of esthetics and function especially is of a great concern to patients, so it is mandatory for missed teeth to be replaced by artificial substitutes and prosthetic appliances of various materials, including metallic and non metallic ones.^{1,2} One of the mostly used non metallic materials is acrylic resin because of their good esthetics matching that of normal tissues, lower cost and density than metallic dentures, also ease of handling.²

However there are some drawbacks of acrylic resin dentures, as change in dimensions through and after processing, usually due to the polymerization shrinkage, also, thermal shrinkage after cooling that causes internal stresses resulting in dimensional inaccuracy, low thermal conductivity, low impact strength leading to sudden fracture when fall down and hypersensitivity to acrylic resin.^{3,4} Innovation of flexible resins as polyamide and other flexible ones for fabrication of dentures either complete or partial ones open the way for new materials for fabrication of dentures other than conventional acrylic resin.^{4,5} It provide excellent esthetics since it is translucent so, it allows natural gum to show through making it invisible, comfort and also good adaptation to undercuts and constant movement in partially edentulous patients.^{6,7}

Polyamide flexible resin (nylon) showed some disadvantages such as high surface roughness, low hardness.^{8,9} Another some drawbacks is difficulty to adjust and polish, it can't be relined or repaired.¹⁰ Also nylon tends to absorb water content and discolor often.¹¹ Smooth surface dentures and polished ones are of great significance for comfort of patient and prognosis of denture.¹² One of the goals of antimicrobial agents addition is to reduce or eliminate microbial organisms without affecting the mechanical properties of dental materials.^{13,14} Impact strength is of a great concern since because it measures the energy needed to fracture the

material suddenly under an impact force.¹⁵ Flexible resins were introduced which have a higher impact strength compared with conventional acrylic resin dentures.¹⁰ Water sorption results in dental materials dimensional changes specially polymeric ones. Molecules of water can penetrate into the spaces among the polymer chains and impulse them away from each other.¹⁶ Gradually, molecules of water can act as plasticizers, changing the polymer mechanical properties.¹⁷

Leaching out of compounds from resins due to solubility affects the biocompatibility of dentures used.¹⁸ Nanotechnology allows actual control of materials at nano scale size with different and enhanced physical, chemical, biologic and electrical properties that differ from those of structures of large-scale.¹⁹ Strontium titanate nanoparticles are a promising filler, since strontium is characterized by its radiopacity and enhancing physical properties also titania nanoparticles characterized by enhancing biological and mechanical properties.²⁰ So in this study we will evaluate the effect of its addition.

MATERIALS AND METHODS

Grouping of specimens:

120 specimens have been prepared and divided into two main groups: Group A: 60 specimens fabricated from the polyamide resin (Dentiflex, Multipress injection system, Rokodent, Poland), Group B: 60 specimens were fabricated from heat-cured acrylic resin (Vertex-Dental.Johan van. Olden-bamevelten, Netherlands). The groups A and B were modified with strontium titanate nanoparticles (Prepared in national research center-Cairo-Egypt). Each group was divided into four subgroups according to strontium titanate nanoparticles concentration as follows: 0 wt%, 0.5 wt%, 1 wt%, 1.5wt%.

Preparation and characterization of strontium titanate nanoparticles

In the present study, strontium titanate (SrTiO₃) nanoparticles were synthesized by a single-step direct hydrothermal process under the strong alkaline condition using crystalline anatase titanium dioxide and strontium hydroxide octahydrate (Sr(OH)₂·8H₂O) as the starting materials at 220 °C.²¹ Strontium titanate nanoparticles were evaluated using scanning electron microscope (SEM) (JEOL, JSM-6510LV, Japan) conducted at an accelerating voltage of 30 kV as shown in figure 1. Also Fourier transform infrared spectroscopy (FTIR) was used. The chemical structures such as bonds between the strontium and titanate are analyzed by the FTIR

spectrum in the range of 400– 4000 cm⁻¹ are shown in figure 2 and its assignment is shown in Table 1.

Mixing of strontium titanate nanoparticles with heat cured acrylic resin and polyamide.

For heat cured acrylic resin strontium titanate nano powder with different concentrations was mixed by weight with the polymer powder of resin.²² For polyamide resin strontium titanate nano powder with different concentrations have been added by weight to grains of polyamide cartridges. All cartridges containing grains of polyamide resin with the strontium titanate nanopowder were subjected to a ball mill technique to obtain acceptable homogeneity.²³

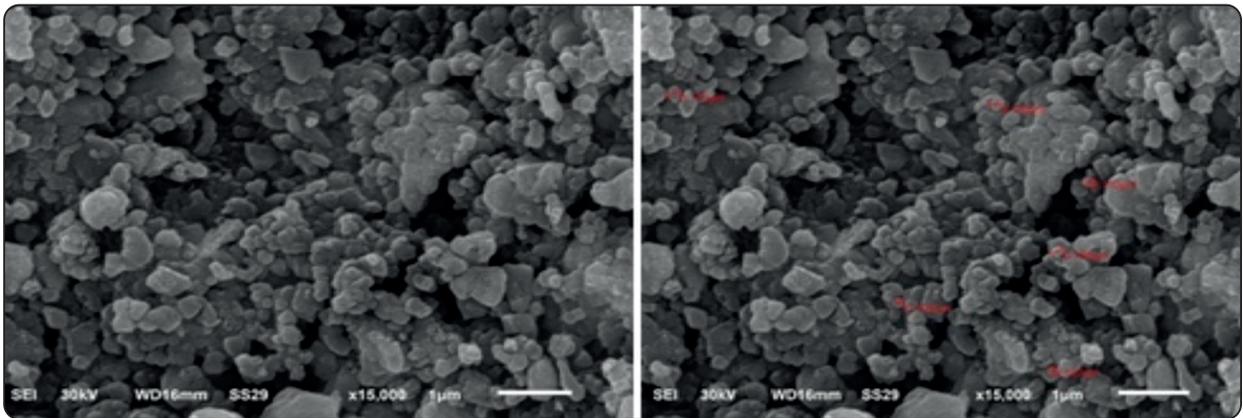


Fig. (1) SEM of strontium titanate nanoparticles

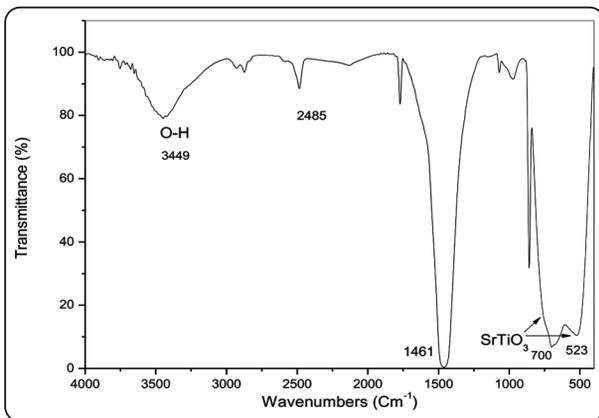


Fig. (2) FTIR of strontium titanate nanoparticles

TABLE (1) FTIR assignment

Wave number (cm ⁻¹)	Assignment
3449	OH stretching vibration of TiO(OH) ₂
2485	C-H mild stretching of butyl group in n-butyl titanate
1461	Instrumental artifact
700	SrO stretching vibration
523	TiO ₂ stretching vibrations

Surface roughness

A whole number of 40 specimens that are disk shaped with dimensions 8 mm x 2 mm were fabricated (20 specimens for each resin). Two dimensional profilometer (Mitutoyo SJ-201) was used. Roughness average (Ra) of each specimen was measured in three different directions, and then the mean (Ra) was calculated for each specimen.²⁴

Impact strength

A whole number of 40 specimens that are rectangular shaped 50 mm x 6 mm x 4 mm were fabricated (20 Specimens for each resin). The impact strength was assessed using impact test machine (Zowek Roell Amsler /RKP 450- Germany) using Charpy method with a pendulum, in which the specimens were horizontally positioned with a distance of 40 mm between the 2 fixed supports, the charpy impact strength of un notched specimens was measured in KJ/m³.²⁵

Water sorption and solubility

A whole number of 40 specimens that are disk shaped with dimensions 8 mm x 2 mm were fabricated (20 specimens for each resin). The specimens were weighed after preparation (M1) on four digits electronic balance. All specimens were stored in distilled water for 7 days in eppendorf in a plastic box and were weighed on four digits electronic balance until a constant weight was achieved (M2). The specimens were dried by being kept on filter paper then they were put again in the plastic box inside a desiccator containing calcium chloride at room temperature and they weighed again after removal from the desiccator (M3) on four digits electronic balance. Volume (V) of each specimen was calculated. Water sorption (Wsp) values and solubility (Wsl) values in µg/mm³ for each specimen were calculated using the following equations:²⁶ $Wsp = M2 - M3 / V$ $Wsl = M1 - M3 / V$

RESULTS

Regarding surface roughness test, Table 2 indicated that the highest value (1.02 µm) was for flexible resin with 1.5 wt% concentration and the lowest value (0.45 µm) was for heat cured acrylic resin with 0 wt% concentration. t test presented significant difference between heat cured acrylic resin and flexible one related to all concentrations. LSD test for heat cured acrylic resin presented significant difference between all concentrations. LSD test for flexible resin presented significant difference between 0 wt%, 0.5 wt% on aside and 1 wt%, 1.5 wt% on the other side, however presented no significant difference between 0 wt% and 0.5wt%.

Regarding impact test, Table 3 indicated that the highest value (3.83 KJ/m²) was for flexible resin with 1.5wt% and the minimum one (0.9 KJ/m²) was for heat cured acrylic resin with 0wt%. t test indicated significant difference between heat cured acrylic resin and flexible related to all concentrations. For heat cured acrylic resin LSD test indicated no significant difference between 0wt% and 0.5 wt% concentrations, also between 1 wt% and 1.5 wt% concentrations, however there was significant difference between 0wt%, 0.5 wt% on` aside and 1 wt%, 1.5 wt% concentrations on the other side. LSD test for flexible resin presented no significant difference between 0wt% and 0.5 wt% concentrations, also between 1 wt% and 1.5 wt% concentrations, however there was significant difference between 0wt%, 0.5 wt% on` aside and 1 wt%, 1.5 wt% concentrations on the other side.

Regarding water sorption test, Table 4 indicated that the highest value (0.0015 µg/mm³) was for heat cured acrylic resin with 0wt% while the lowest value (0.00003µg/mm³) was for flexible resin with 1.5 wt%. t test presented significant difference between heat cured acrylic resin and flexible one in all concentrations, LSD test indicated significant difference between all concentrations in heat cured

acrylic resin. For flexible resin LSD test indicated no significant difference between 0 wt% and 0.5 wt% concentrations, also between 1 wt% and 1.5 wt% concentrations. However there was significant difference between 0wt%, 0.5 wt% concentrations on aside and 1 wt%,1.5 wt % concentrations on the other side.

Regarding solubility test, Table 5 indicated that the highest value (0.0016 µg/mm³) for heat cured acrylic resin with 1.5 wt% concentration while the lowest value (0.00003µg/mm³) for flexible resin with

1.5 wt% concentration. t test presented significant difference between heat cured acrylic resin and flexible one in all concentrations. LSD test presented significant difference between all concentrations in heat cured acrylic resin. For flexible resin LSD test presented no significant difference between 0 wt% and 0.5 wt% concentrations, also between 1 wt% and 1.5 wt% concentrations. However there was significant difference between 0 wt%, 0.5 wt% concentrations on aside and 1 wt%, 1.5 wt % concentrations on the other side.

TABLE (2) Means and standard deviations (SD) of roughness (µm) of both heat cured acrylic and flexible resins with different concentrations of strontium titanate nanoparticles.

Strontium titanate Conc.	Materials		T value	P value
	Heatcured	Flexible		
0	0.45 ^{Bd} ±0.0100	0.85 ^{Ac} ±0.015	38.58	0.0001
0.5wt%	0.46 ^{Bc} ±0.0110	0.88 ^{Ac} ±0.015	37.69	0.0001
1 wt%	0.50 ^{Bb} ±0.0057	0.95 ^{Ab} ±0.015	48.08	0.0001
1.5 wt%	0.57 ^{Ba} ±0.0057	1.02 ^{Aa} ±0.00005	12.16	0.0003
LSD	0.0163	0.0654		

Means with different letters in one row and column are significantly different (P <0.05).

TABLE (3) Means and standard deviations (SD) of impact strength (kJ/m²) of both heat cured acrylic and flexible resins with different concentrations of strontium titanate nanoparticles.

Strontium titanate Conc.	Materials		T value	P value
	Heatcured	Flexible		
0	0.93 ^{Ba} ±0.11	3.53 ^{Aa} ±0.05	34.88	0.0001
0.5 wt%	1.03 ^{Ba} ±0.03	3.63 ^{Aa} ±0.05	64.84	0.0001
1wt%	1.07 ^{Ab} ±0.03	3.80 ^{Ab} ±0.1	44.48	0.0001
1.5 wt%	1.08 ^{Ab} ±0.028	3.83 ^{Ab} ±0.05	74.68	0.0001
LSD	0.1217	0.170		

Means with different letters in one row and column are significantly different (P <0.05).

TABLE (4) Means and standard deviations (SD) of water sorption ($\mu\text{g}/\text{mm}^3$) of both heat cured acrylic and flexible resins with different concentrations of strontium titanate nanoparticles.

Materials Strontium titanate Conc.	Materials		T value	P value
	Heatcured	Flexible		
0	0.0015 ^{Aa} ±0.00005	0.0003 ^{Ba} ±0.00011	15.5	<0.0001
0.5 wt%	0.0013 ^{Ab} ±0.00005	0.0003 ^{Ba} ±0.00005	20.5	<0.0001
1 wt%	0.0011 ^{Ac} ±0.00005	0.0001 ^{Bb} ±0.0001	15.5	<0.0001
1.5 wt%	0.0007 ^{Ad} ±0.00005	0.00003 ^{Bb} ±0.00005	20.1	<0.0001
LSD	0.0001	0.0002		

Means with different letters in one row and column are significantly different ($P < 0.05$).

TABLE (5) Means and standard deviations (SD) of solubility ($\mu\text{g}/\text{mm}^3$) of both heat cured acrylic and flexible resins with different concentrations of strontium titanate nanoparticles.

Materials Strontium titanate Conc.	Materials		T- value	P value
	Heatcured	Flexible		
0	0.0016 ^{Aa} ±0.00015	0.0004 ^{Ba} ±0.00005	13.08	<0.0001
0.5 wt%	0.0014 ^{Ab} ±0.00005	0.0002 ^{Ba} ±0.00005	26.16	<0.0001
1 wt%	0.0011 ^{Ac} ±0.0001	0.00006 ^{Bb} ±0.00005	15.5	<0.0001
1.5 wt%	0.0008 ^{Ad} ±0.00005	0.00003 ^{Bb} ±0.00005	17.67	<0.0001
LSD	0.0002	0.0001		

Means with different superscript capital letter in one row and different superscript small letter in one column are significantly different ($P < 0.05$).
LSD = Least Significant Difference

DISCUSSION

Nanotechnology could enhance the properties of different materials used.²⁷ Prosthetic discoloration and microbial colonization due to rough surface considered a source of discomfort to patients.²⁸ Regarding surface roughness strontium titanate nanoparticles have increased it for all specimens of both polyamide and heat cured acrylic resin specimens and this may be due to filler incorporation that increase denture surface roughness.²⁹ Polyamide was shown to have higher values of surface roughness than heat cured acrylic resin, that may be a result of easy and good performing of finishing and polishing

of heat cured acrylic resin.^{29,30} These results was in agreement with **Menaka, Abuzar, et al.**²⁹ and **de Freitas Fernandes, et al.**³⁰ who concluded that conventional acrylic dentures have a significantly smoother surface than that of polyamide. Polyamide surface roughness are affected by some degree of degeneration of the surface of that is heated to a greater temperature and subjected pressure during injection than that with conventional heat cured acrylic one. Difficulty of finishing and polishing of polyamides have been reported since it have low melting temperature.²⁹ Throughout polyamide polishing, margins fraying was sometimes arised as a result of excess heating and exposure of fibers.²⁹

Impact strength of both polyamide and heat cured acrylic resin have been increased as strontium titanate nanoparticles increased. This is may be due to the combination SrTiO₃ nanoparticles and resin, enhance the internal resistance due to force exchange between matrix and filler, also the high interfacial shear strength between them. The applied pressures are also transferred to the nanoparticles, increasing the impact strength.^{31,32} This was in agreement with **Shihab et al.**,³³ who have assessed some properties of heat activated resin reinforced with strontium titanate nano powder and have found that the impact strength of heat cured acrylic have been increased with increasing strontium titanate nanopowder.³³ The addition of strontium titanate nano fillers to the resin may result in limitation of motion and deformation of the matrix by introducing a mechanical restriction. Also increased impact strength may be resulted from reduction of the segment motion which result from form efficient network of filler and PMMA.³⁴

These results were in agreement with **Mohamed et al.**,³⁵ who have studied the consequence of titanium dioxide nano particles incorporation on mechanical and physical properties on two different types of acrylic resin denture base and concluded that adding 1.5 wt% TiO₂ have increased the impact strength of acrylic resin. **Asar et al.**,³⁶ have revealed that impact strength of heat cured acrylic resin have been increased at both 1wt% and 2wt% titania nanoparticles. Impact strength of polyamide was higher than that of heat cured acrylic resin. This might be as a result of polyamide chemical structure properties, permitting it to absorb forces in a better sequence. The findings of this study was in agreement with **Koray, et al.**³⁷ and **Hamanaka, et al.**³⁸ who presented that heat cured acrylic resin had lower impact strength than thermoplastic resins.

Water sorption can cause discoloration and dimensional changes results in internal stresses, cracks or failure of the denture. Excessive water sorption can lead to undesirable effects on the properties of the material and accordingly diminish

the longevity of a denture within the oral cavity. Thus, it is critical to use materials with low possible rates of water sorption.³⁹ Solubility of dental materials is of clinical importance since it influence both rate of degradation and their biologic compatibility due to the nature of the eluents. Solubility contributes to surface properties and esthetics, resulting in prosthetic and restoration failure.⁴⁰ In the present study strontium titanate nanoparticles have decreased the water sorption and solubility of both heat cured acrylic resin and polyamide specimens as the concentration increased. Results of this study was in agreement with the study that was done by **Asar et al.**³⁶ who have founded that TiO₂ have decreased water sorption and solubility and he explained that result may be due to polymer porosity high level or microvoids that lead to fluid transport into and out of polymer. And since the filler to polymer ratio increased, the water sorption and solubility decreased. Results of the current study revealed that water sorption and solubility of polyamide were less than that of heat cured acrylic resin and these results were in agreement with the study which was done by **Shah et al.**⁴¹ who evaluated water sorption, solubility of heat cured acrylic resin and flexible denture base resins, and he claimed that repellency of water of flexible resin was high and they have low surface free energy and the contact angle between water and flexible resin was high with, and all of this caused lower values of water sorption. As well, it was showed that there was a strong hydrogen bonding among amide groups and a lesser attachment areas for water molecules; consequently, the water sorption amount in flexible resin was lower than acrylic ones. The residual monomer of heat cured acrylic resin was indicated as a reason for the greater solubility than that of polyamide.⁴¹ **Nguyen et al.**,⁴² studied water sorption and solubility of polyamide denture base materials compared with PMMA denture bases, and he found that both Polyamide materials had lower water sorption and solubility than the PMMA.

CONCLUSIONS

Within this study limitations and based on the results. Strontium titanate nanoparticles have increased surface roughness of both resins as the concentration increase. For impact strength, water sorption and solubility, strontium titanate nanoparticles gives better effect with both polyamide and heat cured acrylic resin as the concentration increase, since as the concentration increase impact strength increased, while water sorption and solubility decreased. Polyamide has higher surface roughness than heat cured acrylic resin. Polyamide has higher impact strength than heat cured acrylic resin. Polyamide has lower water sorption and solubility than heat cured acrylic resin.

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