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Altering Water Effect and Surface Properties of Heat Cured Poly Methyl Methacrylate by Adding Surface Treated Titanium Di Oxide Nano Particles

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Abstract

Titanium dioxide (TiO_2) is extensively used part of our regular life. It can be observed in various commonly utilized goods & customer imports for instance drugs, plastics, cosmetics, food and paper. Universally, the quantity of titanium oxide manufactured in 2009 was 4.68 million tons for that reason the tenacity of the revision is controlling the amount of change on the denture base physical features such as the heat treatment in denture processing (PMMA)through the implementation of modified outer surface titanium di oxide Nano particles. Its properties are comprised of water solubility, surface roughness, water sorption and surface hardness. The controlled TiO_2 powder added to heat cured acrylic material according to the main weight of PMMA in concentration of 3%, 5% and 7%, and then it's mixed regularly by engine of probe ultra-sonication. Titanium oxide Nano fillers were merged in denture based material with the help of more free radical polymerization in bulk. The nanoparticles were surface treated by a layer of trime thoxysilypropylmethacrylate (TMSPM). 120 specimens were created and separated into 3 sample sets affording to the examination (each sample sets consist of 40 samples) and each sample sets were sectioned in four subsets according to the TiO_2 added ratio and ultimately containing 10 samples in each subset. The tests which conducted were indentation hardness (shore D), surface roughness properties as well as water sorption and solubility. For each test four of the sub groups (one controlled and three with added Nano TiO_2). Important to mention that extremely elevation in surface hardness score and roughness were detected at the ratio of 3%, 5%and7%. Thus escalation of added TiO2 nan-fillers considerably reduced the water solubility and sorption. The accumulation of the powder of TiO2 nanoparticles which have been surface treated to the PMMA acrylic resin thus changing the PMMA properties for example surface rigidity and surface coarseness. In which it's increased in direct proportional relation with the raise of TiO_2 concentration; at the same time addition of surface treated TiO_2 Nano particles decrease water sorption and solubility of (PMMA) also in direct proportional relation with the raise of TiO_2 concentration.

Introduction:

Regardless of the common use of acrylic resin in prosthetic dentistry the surface roughness and surface cracks are the most collective causes of faller in the cures of prosthodontic which is still an unsolved issue. To improve properties of polymers it had been strengthened by additives. Up to the present time, up to 95% dental prostheses are self-possessed of poly (methyl meth acrylate), important advantages counting its biocompatibility, properties, adequate strength and visual make it useful [(1)]; however, this resin inherit some flaws which used a meager power especially a worse size under exhaustion disaster intraoraly wares abrasion resistance surface roughness an hardness as well as water sorption and solubility [(2)]. Surface treated Nano particles might be a quality material for strengthening denture based polymers as for their well acknowledged development in mechanical & physical properties and an adequate appearance as well [(3)]. Polymer nanotechnology characterizes a fresh view in Nano science, attracted great attention by the polymer Nano composite especially because of their unexpected hybrid properties that are consequent from the two components [(4)]. Modify the properties of PMMA by reinforcing surface treated TiO_2 to the PMMA being very important. It's a convenient technique to lessen the energy of surface, to elevate the polymer medium compatibility & scattering resemblance. Therefore, improving the polymer inorganic particle's properties by the surface alteration of an inorganic element with an organic selected material. The non-viable Nanoparticles were suffered surface dealing with saline well coupler agent and well entrenched into PMMA.[(5,6)] Eliminate accumulation of TiO_2 elements and advance its self-compatibility with organic polymer by the treatment of the TiO2 nanoparticle's' surface with trimethoxysilypropyl methacrylate (TMSPM) [(7,8)]

Materials and Methods:

Titanium di Oxide (TiO_2) Nano filler Silanated by a layer of trimethoxysilypropylmethacrylate (TMSPM), tin foil substitute as separating media, type III hard dental stone and heart curing acrylic liquid & powder were utilized. Almost eighty samples were prepared for this research. All these specimens were distributed in two groups conferring the selected tests. These groups comprised of forty samples each while were further subdivided rendering titanium di oxide Nano filler (TiO2) concentration in four subgroups as given below:

1-Group (A) control group (30 specimens of acrylic resin without TiO_2).

2-Group (B) 3% modified group (30 specimens of acrylic resin + 3% by weight TiO_2)

3-Group (C) 5%modified group of (30 specimens of acrylic resin+5% by weight TiO_2).

4-Group (D) 7% modified group of (30 specimens of acrylic resin + 7% by weight TiO_2).

Table (1) show mixing ratio of TiO_2 with PMMA according to the manufacturer instruction

Plastic model preparation:

The highly precise laser cutting machine was used to cut deferent gage (2.5 & 4mm) plastic plate in chosen dimension and shape for the construction of two plastic models Fig. (1).

1. Bar shaped samples having dimensions of length 65mm x width 10mm x thickness 2.5 ± 0.1mm were tested with Shore. D hardness test and Surface roughness test.

2. Similarly, disc dimension such as 0.5 mm thickness and 50mm diameter were utilized in water solubility & sorption tests.

Mould preparation:

Among the process of mould preparation, a conservative flasking practice was followed for complete dentures. The cold mold seal or separating medium was coated on the plastic model and dried. After that, the lower part of the metal flask was invested which was full of dental stone. All of this was mixed according to appropriate instructions of manufacturer, thus, the mixing ratio of powder to water used was 100g per 25ml. Moreover, vibration was used to release the stuck air and was left to set. Approximate insertion of the plastic model was about one half of the depth of models for easy removal after stone setting Fig.(2).

Addition of Titanium di oxide Nano fillers:

The modified Nano filler powder of TiO_2 was added in four groups by weight such as the accumulation to monomers was carried out to be as 7%, 3%, 0% and 5%. For this, an electronic balance having an accuracy of 0.001 g was utilized. After adding the Nano fillers of TiO_2 to monomers, the dispersion of fillers in monomers is carried out with the aid of ultra-sonication mixing type while utilizing apparatus of probe sonication (120W, 60 KHz). Thus their breakage in Nano crystals was done only in 3 minutes Fig. (3). The probability of phase separation and aggregation of particles was reduced by mixing the acrylic powder to the suspension of TiO2 Nano filler & monomers.

Packing of acrylic resin:

After indication of perfect parting of the resin from mixing glass jar or simply reaching to the dough stage, the packaging was started. All the resin was extracted from the jar, rolled & packed into the mold which was already coated with polythene sheet and a separating medium. The flask's two portions were sealed together and a hydraulic pressure was slowly put on the flask which led the dough to even move all though the space of mould. After releasing the pressure, the flask was opened up and a sharp scalpel was used to remove all extra material. Then a second trail closure was executed. The surface of stone was coated once more with separation medium and dried. While removing the polythene sheet. Lastly, flasks two sections were closed again to obtain the intimate contact among them. I was left for five minutes under press of 20 bars and then shifted to the water bath immediately.

Curing:

The clamped flask was placed in water bath for this purpose and heat treatment was done for one and half hour at 74 °C. Thus, the increase in temperature occurred to the boiling point for thirty mins.

Finishing and polishing:

A lathe polishing machine was used to polish and finish each specimen excluding those which have to be used for test of surface roughness. All samples were removed from flasks cautiously & cleaned. Acrylic bur and W& H laboratory engine was utilized to remove the acrylic flashes. Whereas, stone bur was utilized to get a smooth surface after usage of 120 grain sand paper along with constant cooling which was provided by immersing in rubber bawl cold water. Further, specimens were polished with lathe polishing machine having ruge wheel along pumice stone and bristle brush. The surface gloss was attained with polishing swap & chamois buff. Their dimensions were calculated using a digital Vernier and polishing was achieved at low speed of

1500 rpm of the machine. A constant cooling was provided as to protect the specimens as extreme heat might distort them.

1- Surface hardness testing

A-Specimen design: The dimensions for the preparation of specimens were 65mm x 10mm x 2.5mm. To test the surface hardness, 10 specimens of each concentration along with the control made up a total of forty specimens. Before testing, all specimens were absorbed in distilled water for about 48 hours.

B-Testing method: a tester used to test was durometer hardness tester or also termed as Shore D hardness which was fabricated by an appropriate design of acrylic material HARTIP 3000compant. This apparatus comprised of a 1, 6 mm diameter cylinder in which a blunt pointed intender of 0.8mm diameter was present. Further this intender was attached to a 0-100 unit gradual digital scale. The normal process involved was to compress the intender quickly & firmly and noting the maximum values as Shore D hardness. All the measurements were noted directly from the digital reading scale. From the various areas (particular selected areas) of every specimen, five values were documented and calculation of the all five readings was done by taking average Fig.(4).

2- Surface roughness tests

A-Test specimens: To test the surface roughness, specimens of dimensions 65mm x 10mm x2.5mm (11) were utilized. For this purpose of roughness measurement, 10 specimens from each group while making a total of 40 specimens, were taken. All these specimens were put into distilled water before testing for about 48 hours at 37 °C.

B-Test equipment and procedure: the surface roughness tester known as profilometer device was utilized to observe the SiO2 Nano fillers' effects of strengthening on the test surface micro geometry. This device has a sharp stylus composed of diamond or surface analyzer to find out the profile for irregularities of surface Fig.(5). The maximum 11mm of distance was traveled.

3- Water sorption and solubility test:

A-Test Specimens: plastic model of dimension 0.5 mm ± 0.1 mm thickness and 50mm ± 1 mm diameter were used for preparation of acrylic disc specimens. To measure the water solubility & sorption, 10 specimens with each concentration forming a total of 40 were utilized.

B-Equipment and procedure: all the specimens were dried out suing a desiccator which comprised of fresh dry silica gel Fig. (6). After that all the desiccators were kept in incubator for twenty four hours at 37°C ± 2 °C and then all specimens were placed in room temperature. Again all specimens were weighed using a digital balance of an accuracy of 0.0001 g and all the cycle was reiterated to attain a constant mass. Therefore, in 24 hours of time, all discs showed a weight loss not more than 0.5 mg. After a time period of five days, all groups showed a 'Conditioned Mass (MI)' and then these were absorbed in distilled water at 37°C ± 2°C for seven days [(19)]. A clean dry towel was utilized to wipe the dental tweezer and after the surety of no visible moisture tweezers were utilized to take out the discs from water. These discs were weighed after removal of one minute. All these showed a mass M2 which was the disc mass subsequent to the distilled water immersion.

WSP=M2-M1/S

For the attainment of solubility value, the reconditioning of discs was done in a desiccator at a constant mass and 37 °C ± 2°C. Thus, at sorption test for first time, the M3 was the mass reconditioning recorded. The entire group reached at M3 in time period of five days. The solubility of each disc among the immersion was determined by equation given below:

WSL = M3-M1/S

Results:

1-Surface hardness: Table (2) indicates the standard error of means, standard deviation, means, standard deviation, maximum and minimum experimental specimen's values for measurement of hardness of surface in various TiO_2 Nano filler concentration.

2-Table (2) descriptive data of Surface hardness parameters analysis.

Thus, the uppermost mean value for group D was observed to be of 85.111 whereas, the lowest mean was detected in (Control) group A having mean of 82.711. Therefore, the means surface hardness of all 4 groups were conspired in Fig. (7).

The increase might be because of the random dispersion of Titanium di oxide (hard material) in the acrylic matrix medium. The slight upsurge of Nano composite hardness at low concentration of Nano particles will be dominated by network density. Although, the increase in Nano composite hardness at7% & 5& may be due to the TiO_2 particles' accumulation on the surface of acrylic matrix.

3-Surface roughness

The standard error of means, standard deviations, means, specimens' maximum & minimum experimental values of surface roughness measurement at various concentration of Nano fillers.

Table (3) Surface roughness parameters analysis

It presented the highest mean value in group D which was 2.457 and lowest in group A which was 2.331. Thus, the graph of all 4 mean values have been plotted as indicated in Fig.(8).

This might be because of the reason that the variance in matrix of acrylic denture base and Titanium particles' roughness or perhaps may be due to the variations in form of particles & characteristics of microstructures.

4-Water sorption

The table 4 represents the means, standard error of means, standard deviation & range of experimental specimen's values which measured the water sorption in numerous concentration of Nano fillers of TiO_2.

Table (4) descriptive data of Water sorption parameters (mg/cm^2).

Water sorption seems to decrease in a similar manner in group B (3%) and group C (5%) concentrations of the added TiO_2 Nano filler (0.336), but for a limited extent. After reaching a concentration of 7% group D water sorption seems to decrease (0.304) Fig (9).

With the increase of TiO_2, the water sorption value decreases. This might be due to the replacement of hydrophilic resin with TiO_2 percentage which results in the decrease of water uptake.

5-Water solubility

The standard means' error, means, standard deviation as well as min. & max. Values of specimen evaluating the solubility of water in TiO2 Nano filler concentration variants.

Table (5) Water solubility parameters analysis (mg/cm^2)

The greatest mean value was shown in group A having a mean of 0.015 and lowest was observed in group D as 0.011, thus, the means of all groups was plotted in Fig. (10).

These reductions can be because of the insolubility of TiO_2 in water as the

accumulation of TiO2 to the specimen's mass performs as additive. Thus, their occurrence will further reduce the acrylic resin solubility.

Discussion:

Surface coarseness of acrylic denture base considerably increased as TiO_2 was added. This might be because of the variance among the matrix of denture acrylic base and titanium particles roughness or perhaps ascribed to the particles' form and material micro structural characteristics variance [10, 9]. Thus, the outcomes resembled the results attained by (Nabil, et al 2011) [11] because of addition of Nano fillers to acrylic resin. A greatly significant increase indicated an increased surface hardness because of the increased percentage of TiO_2 powder. Which might be ascribed as the random distribution of hard material's particles (Titanium di oxide) in the matrix of acrylic media. Network density dominates a slight upsurge in Nano composite hardness at lower concentration of about 3% nanoparticles whereas, the TiO_2 particles' addition on acrylic matrix surface cause an increase in Nano composite hardness at 7% & 5 %[12]. Our current study differed with results of Abdul Ameer [14] while same outcomes were presented by AlMomen, 2002[13]. With an upsurge in TiO_2 percentage, the water sorption value decreased which was because of the statement that hydrophilic resins are substituted by titanium particles. Which resulted in reduction of water uptake as the diffusion of water molecules via this material highly lowered as compared to that of matrix [15]. This research agreed with Duraid, et al, 2012[16] who specified an increased in ZrO_2 Nano fillers' percentage due to water sorption decrease. A great substantial decrease was observed in the specimen's water solubility values while comprising TiO_2 with various percentages. This reduction might be because of the insolubility of TiO_2 in water, thus further addition of TiO2 o the samples' mass will turn as additives and their incidence will cause reduction in acrylic resin's solubility [15]. Its outcomes were in accordance to the Duraid, et al 2012[16] who indicated a decrease in water solubility due to the increased ZrO2 Nano filler's percentage.

Conclusions:

Inside the boundaries of this research, the drawn conclusions are given below:

1- The increased percentage of TiO_2 caused a greatly substantial decrease in acrylic resin's water solubility & sorption.

2- The addition of various percentages (7%, 5% and 3%) of TiO_2 to heat cured acrylic resin caused a significantly increased surface hardness.

Over all conclusions from the result appear in the study the 5% reinforcement of TiO_2 Nano filler advisable because of the highly significant decrease in the total amount of water uptake, the significant decrease of the solubility of reinforced acrylic, non-significant increase in the roughness of the surface and finally the surface hardness was highly significant increased.

Fig. (1): Laser cutting machine (CNC).

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Fig. (2): Mould preparation.

Fig. (3): Probe sonication apparatus.

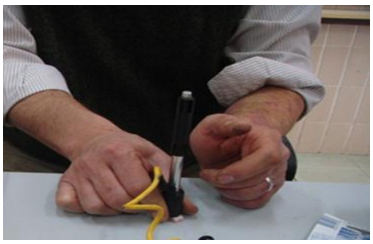

Fig. (4): Durometer hardness tester.

Fig. (5): The Profilometer device.

Fig. (6): Desiccator.

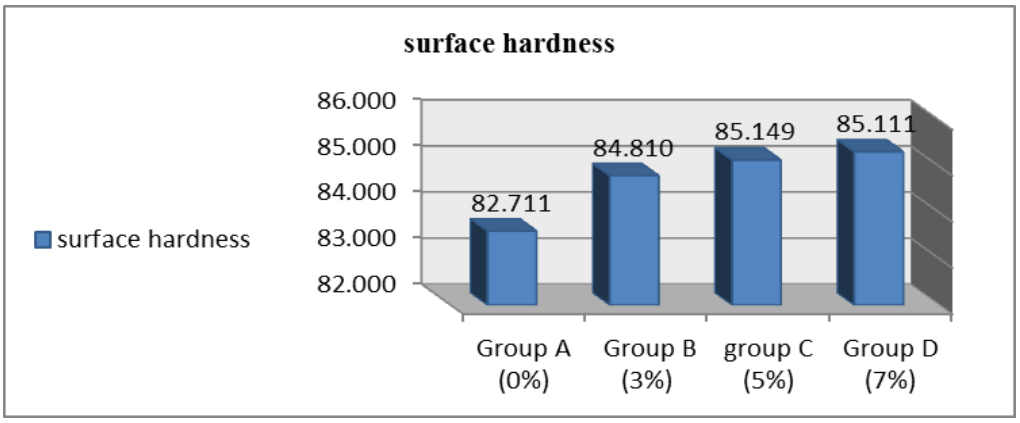

Fig. (7): Bar chart showing the mean of changes in the surface hardness.

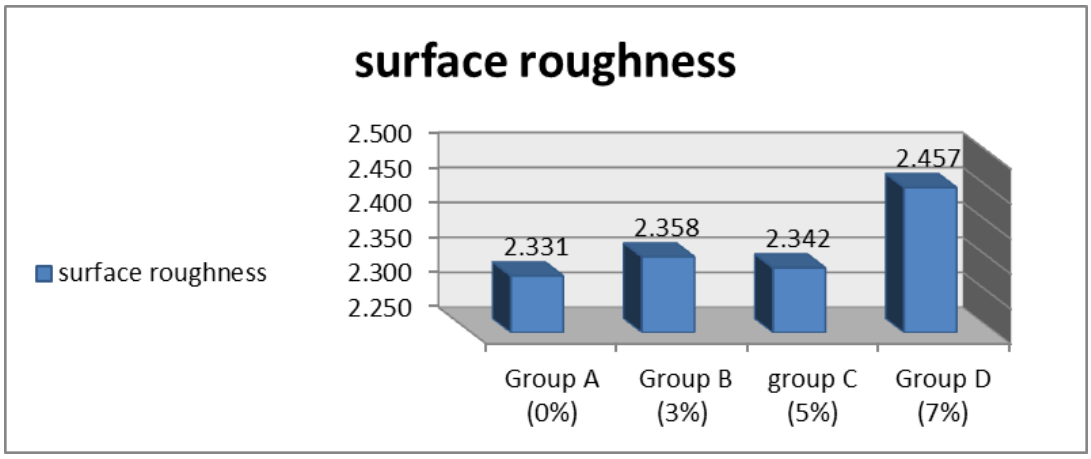

Fig. (8): Bar chart showing the mean of changes in the surface hardness..

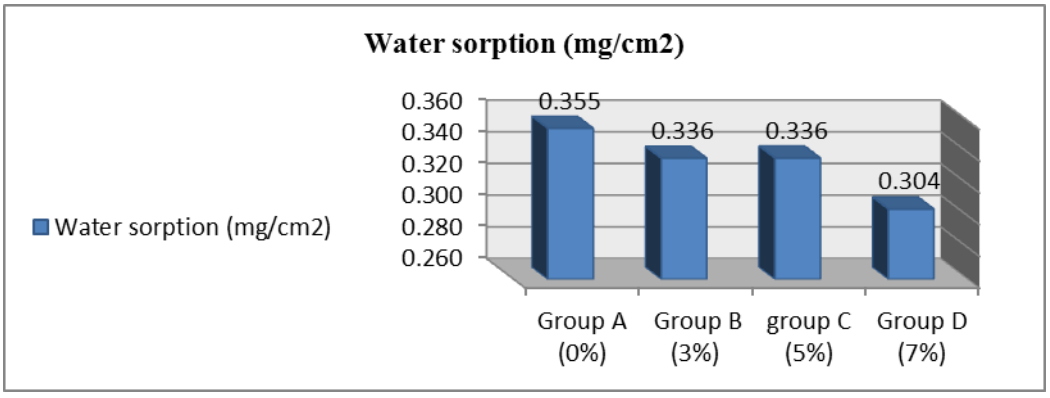

Fig. (9): Bar chart showing the mean of changes in the surface hardness.

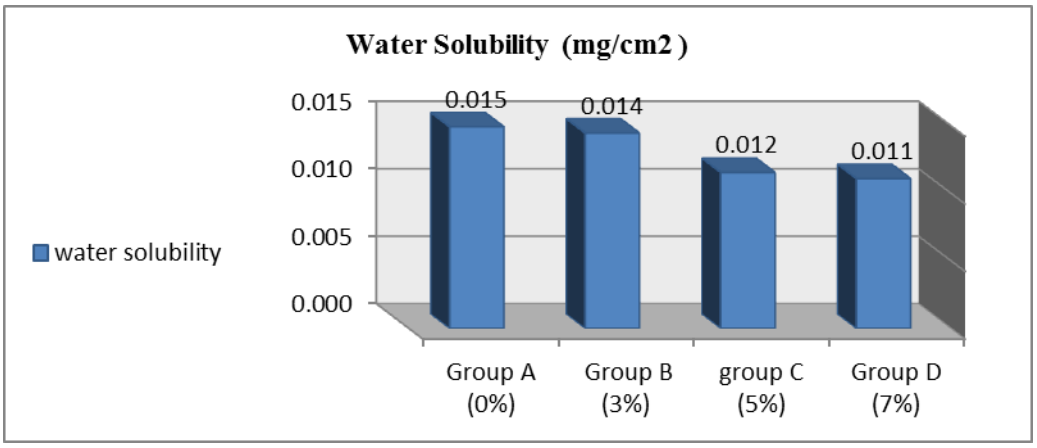

Fig. (10): Bar chart showing the mean of changes in the surface hardness.

Table (1): Mixing ratio of TiO_2 with PMMA according to the manufacturer instruction.

TiO_2	***Gram Wight***	***Polymer***	***Monomer***
0%	0 g	100 g	40 ml
3%	3 g	97 g	40 ml
5%	5 g	95 g	40 ml
7%	7 g	93 g	40 ml

Table (2): Descriptive data of Surface hardness parameters analysis.

		Group A	***Group B***	***Group C***	***Group D***
N		10	10	10	10
Mean		82.711	83.811	84.140	85.111
SD		0.723	0.923	0.555	0.321
SE		0.223	0.267	0.199	0.1
range	Min.	81.44	82.01	83.24	84.4
	Max.	83.48	85.011	86.22	85.345

Table (3): Surface roughness parameters analysis.

		Group A control	***Group B***	***Group C***	***Group D***
N		10	10	10	10
Mean		2.331	2.358	2.342	2.457
SD		0.01	0.031	0.001	0.007
SE		0.003	0.009	0.003	0.002
range	Min.	2.303	2.338	2.341	2.451
	Max.	2.337	2.41	2.344	2.471

Table (4): Descriptive data of Water sorption parameters (mg/cm^2).

		Group A control	***Group B***	***Group C***	***Group D***
N		10	10	10	10
Mean		0.355	0.336	0.336	0.304
SD		0.0001	0.005	0.001	0.004
SE		0.0005	0.0016	0.0023	0.0013
range	Min.	0.355	0.331	0.336	0.3
	Max.	0.356	0.348	0.338	0.311

Table (5): Water solubility parameters analysis (mg/cm^2).

		Group A	***Group B***	***Group C***	***Group D***
N		10	10	10	10
Mean		0.015	0.014	0.012	0.011
SD		0.005	0.0002	0.009	0.0001
SE		0.0014	0.0007	0.0005	0.0002
range	Min.	0.002	0.014	0.011	0.011
	Max.	0.017	0.015	0.012	0.011

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