

Development and Performance of Composite from Modified Nano Filler with Plasma Treated Fiber and Heat Cured Acrylic Denture Base Material on Some of Its Properties – In Vitro Study

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Abstract: *There are alternative materials to what is used now a day in various prosthetic appliances that have proved their success. However, there are still high need for more development of a new material because of problems and defect in mechanical properties of such alternatives which is not satisfied. (1) There are demands on efficient materials that cost less, substitute material. Investigation a new concept of combining salinized zirconium oxide (ZrO₂) nano filler and plasma treated polypropylene (pp) with PMMA, aiming to improve the performance of dental materials. **Materials and Methods:** Zirconium oxide nanoparticles had salinized within a layer of (trimethoxysilyl)propyl methacrylate (TMSPM) before mixing with the monomer methylmeth acrylate in a percentage 2.0% by weight depending on the pilot study , and oxygen plasma treated polypropylene fibers also 2.0% which mixed homogeneously with powder (PMMA), then mixed together using general procedure. According to the performed tests, four group had divided and prepared as samples, each one of these groups consisted of (14.0) samples and these were subdivided into 2.0 groups, the control and experimental groups .The tests which are conducted were tensile strength, water sorption and solubility test, surface roughness, and wettability. **Results:** Highly and sufficiently increasing in tensile strength (62.1143 N/mm²) happened with the incorporation of 2.0% Wt. salinized zirconium oxide nanofiller and 2.0% modified by plasma pp fibres. A significant increase (p = 0.22) in water sorption (0.15349 mg/cm²), but the water solubility (0.0278133 mg/cm²) were significantly decreased with the addition of silanized (ZrO₂) Nano particles and oxygen plasma treated pp fibers group compared with the control one. A significant difference (p = 0.22) in surface roughness appeared, and highly significant decrease in contact angle of wettability test by using independent samples t-test for statistical analyses. **Conclusions:** The addition of silanized ZrO₂ nanoparticles and oxygen plasma treated pp fibers to heat cure acrylic resin material improves the tensile strength, solubility and wettability ,although this addition increases water sorption and surface roughness but within limited range.*

Keywords: Nano ZrO₂ Filler, PMMA, PP Fiber, Nano Composite

1. Introduction

Organic polymers such as polymethyl methacrylate and its copolymers have insufficient mechanical strength. Various strategies have been adopted to improve PMMA denture base material. The adopted strategies have focused mainly on improving properties of the material or the biological activity. Many studies had suggested that many of polymer properties could be refined into highly range by changing the type of polymer or chain length, and by copolymerization or blending of two or more polymers^[2].

Within all polymeric denture base materials, PMMA still has significant popularity. This is mostly due to its favourable, and it none ideally characteristics^[3]. More than averages of dental material researches have been focused onto the development of materials with much low levels of residual monomer after processing, higher strength, more radiopacity, better dimensional stability, and increased resistance to candida albicans^[4].

Various techniques focused on strengthen of PMMA with addition of other materials had illustrated^[5- 7]. Kevlar and carbon fibres strengthen had been used . These were found to be aesthetically unacceptable, however a complex method is required to enhance their addition, preparation, and positioning of the fibre layers, it's been found that both technique time consuming^[8].

In the recent years the incorporation of PP fibres to strengthen PMMA is found effective to enhance the mechanical properties which illustrated in the literature^[9-11], they are also publishable, aesthetic, and manipulated easily^[5,11].

Hybrid fiber reinforcing composite such as glass fiber and polyethylene fiber, presented that they shows highly effective in strengthen the denture base material^[11].

Along the significant development of nanotechnology and nanoparticles materials, the work conducted in relation to the use of Nano sized particles to reinforce the denture base material thus producing nanocomposite with enhanced properties as compared to those adding to them micro-scale fillers^[12].

The resultant polymer nanocomposite will had mechanical properties depend mainly on the less aggregation of the particles at the filler matrix interface ,so to improve the adhesion in the interface between the Nano particles and the matrix , the saline coupling material is used^[13,14].

Polyethylene fiber and ZrO₂ Nano filler have several mechanical and physical properties which are differ ,make the polymer may gain these properties to enhance its action oxygen plasma which is away accepted and could be used to take place instead of other methods, with oxygen plasma treatment for the pp fibres, surface chemistry and texture

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may be changed which increased the adhesion with the matrix [15].

Plasma treatment increase the polarity of the low surface energy of pp fibers by forming strong covalent bonds which lead to improve the adhesion with the matrix. The main purpose of plasma treatment is to by introducing polar and make roughness on the surface of the fibre and this enhance the fiber-matrix adhesion [9,16].

The present research investigates some mechanical and physical properties of PMMA polymer as a matrix after incorporation of salinized nano-ZrO₂ particles and plasma treated PP fibers aiming to increase performance of the polymer.

2. Materials and Methods

Surface modifications for the Nano particles and fibre:

Modification of Zirconium oxide (ZrO₂) nanofiller 70-80nm HWNANO (China) was utilized by adding saliane coupling agent (trimethoxysilylpropyl methacrylate TMSPM) to the zirconium oxide Nano particles. As the procedure described in a study of Al- Hiloh and Ismail (2016) [17] to introduce reactive groups.

According to the pilot study, the decision was made to choose 2.0 wt% silanized Nano zirconia filler which was used in this study, then the polypropylene fiber (Crace cemfiber) cutted to 4.0 mm length and modified by oxygen plasma, then choose 2.0 wt%. According to a study done by Mohammed and Ismail [18] in which they used 2.5 wt% as the best percentages, but here because of the loading with other type of enforcement the percentage was decreased to 2.0 wt%. PP fibers was processes treated with Oxygen plasma utilizing DC glow discharge system device with 6 minutes exposure time, the gas pressure had been limited to 0.5×10^{-1} millibar and the gas flow rate was 10 ml/min using flowmeter. Therefore 2.0 % of silanized ZrO₂ nano filler dispersed in the monomer which was weighted by using digital electronic balance of accuracy 0.0001 were added to the monomer, the fillers were significant dispersed in the monomer by ultra-sonication, using a probe sonication apparatus (120 W, 60 KHz) within period of 3 minutes to prevent aggregation [17].

2.0% oxygen plasma treated pp fibers, were mixed in mortar and pestle until uniform mixture was attained with the powder for about 4.0 minutes then mixed with the sonicated nano particles in monomer liquid to make a dough which are going to be used to make the specimens. When the dough stage was achieved when after mixing powder and liquid in ratio of 2.5:1 polymer/monomer by weight according to manufacturer's instructions, pressed in the mould of each test used, then packed, and cured for 0.5 hours at 74°C and 1.5 hours at 100°C according to the instructions.

The mould for making the specimens were prepared by using various plastic patterns which was made by cutting plastic plates with measured thickness into desired shape and dimension according to the specification of each test, using accurate laser cutting machine. Compression mold flasking technique for complete denture that was followed in the

mould preparation. Four different patterns mould of plastic were constructed. For tensile strength flat dumbbell shaped with (75mm in length, 4.0mm in width and 2.5mm in thickness) at the parallel segment. Dumbbell shaped specimens were fabricated according to ASTM specification D-638M (1986) with dimensions as appeared in figure 1.

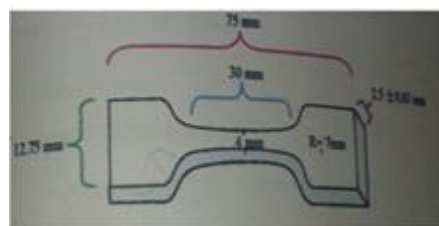


Figure 1: Dimensions of tensile test pattern

Instron universal testing machine (LARYEE, china) where used to accomplished the test with crosshead speed 1.0 mm/min and maximum loading of 20 Kg Tensile strength was calculated according to the formula

$$T.S. = \frac{\text{Force of Fracture}}{\text{Cross Section Area}}$$

For water sorption (WS) and solubility (WSL) metal pattern were made by cutting stainless steel plate with 50mm in diameter and 0.5mm in thickness. According to below equation water sorption and solubility was calculated:

Where M₁ the conditioned dried weight, M₂ the weight after immersed in distilled water and get a constant weight and M₃ was the reconditioned weight.

$$WS = (M_2 - M_1) / \text{Surface area}$$

$$WSL = (M_1 - M_3) / \text{Surface area (ADA Specification NO.12, 2000)}.$$

Surface roughness test specimens with (65mm x10mm x 2.5+ 0.1mm) dimensions made to be used for the test. All specimens were without polishing with only slight remove to the flash were done and stored in distilled water at 37°C for 48 hours before being tested [21]. A contact profilometer (Taylor Hobson Form TalysurfPGI-840, USA) was used to study the effect of pp fiber and salinated ZrO₂ nanoparticles addition on the topography of the surface. The device has stylus for surface analyser to examine the profile of the surface by recording of all the peaks and recesses which is found by its scale. Each specimen of the control and experimental group had put on its location of the tested area passing over three lines on the tested area, calculation for mean was done to the three readings, and statistical analysis used the average values.

Wettability and Contact Angle

Wetted surface is described if liquid had spreads within that surface evenly without the formation of droplets. Figure(2) below shows the hydrophilic surface that's defined as a wetted surface with water as a liquid. In order to determine the wettability contact angle measurement is used, the angle is in the middle of the sessile drop base and the tangent line that contacting the solid, liquid, and air simultaneously. Dispersing of 10µL as distilled water on the specimen centre

using pipette, dimensions of the sample (8mm× 30mm× 2mm) width, length and thickness respectively. This study has utilized the static sessile drop method; as the water dispersed, five minutes waiting in order to allow drops to be in equilibrium status. Contact angle measured within both sides and the mean was taken, by adopting digital microscope known as (Dino lite digital microscope pro Taiwan) at 45x magnification adjusted with high resolution camera at (1024 × 768 pixel) and digitised with a software (Dino capture 2.0 Version 1.03.03); an accurate measurement to the contact angle was done as shown in figure (3).

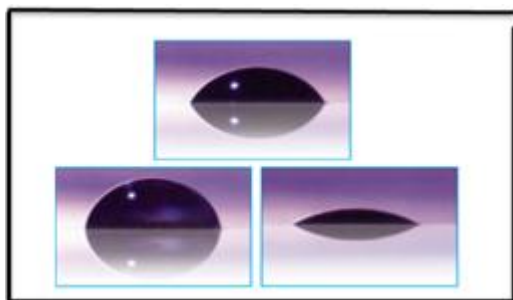


Figure 2: Surface wetting types (22)

A. Ordinary surface typical wetting ,B. Hydrophilic poor wetting ,C. Hydrophilic good wetting



Figure 3: Measuring contact angle by Digital microscope with specimen

Statistical analysis

SPSS (Statistical Package for Social Science) version 20.0 a statistical software program was used for data analysis. Both control and experimental group properties had been analysed by independent test at 95% confidence level to assess the statistical significance.

3. Results

This In vitro study was conducted to compare and evaluate the tensile strength, water sorption and solubility, surface roughness and wettability of conventional, and modified hot cured acrylic resin.

The characterization FTIR test has performed to samples of zirconium oxide nanofiller before Silanation which mean without modification, and (trimethoxysilyl) propyl methacrylate (TMSPM) saline coupling agent to indicate the difference in the spectra, as seen in figure (4) and figure (5) below. Additionally other FTIR has performed to zirconium oxide nanofiller after Silanation to indicate the differences in in the peaks, as seen in figure (6). MPS absorption bands could be assigned to functional groups presence.

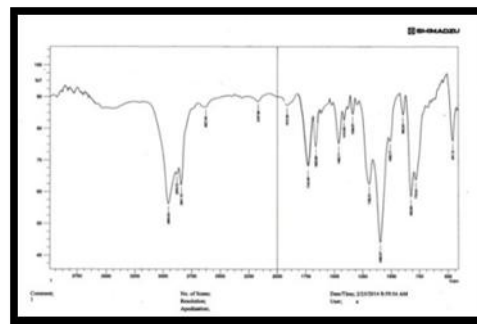


Figure 4: FTIR spectrum of TMSPM

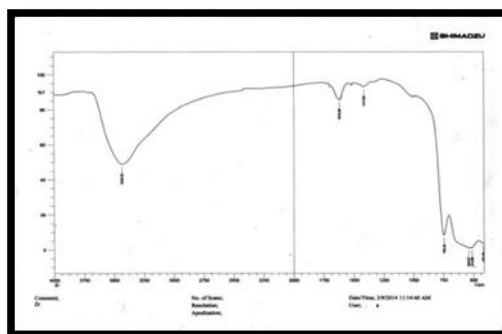


Figure 5: FTIR spectrum of non silanized zirconium oxide nanoparticles

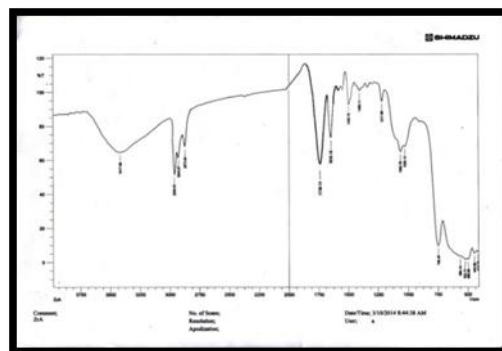


Figure 6: FTIR spectrum of zirconiananoparticles after silanization

The polypropelene fibre before and after oxygen plasma treatment as shown in figure (7) and Figure (8) which indicate (C=O,1719) and (O-H ,3382) functional groups.

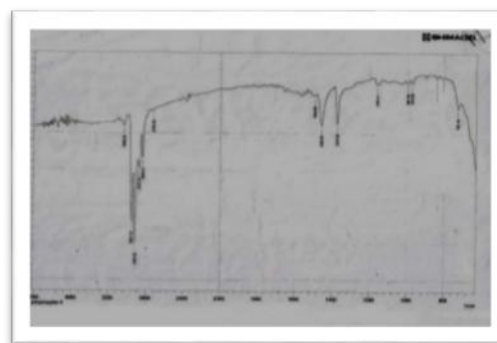


Figure 7: FTIR of untreated PP fibers

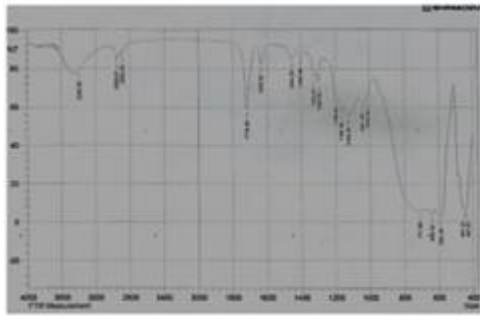
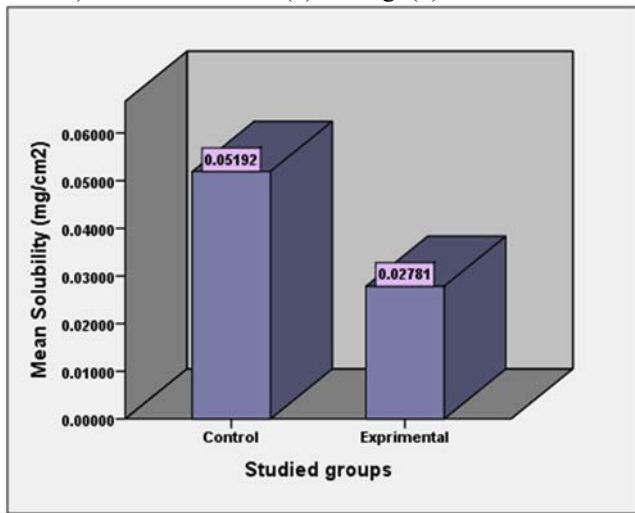


Figure 8: FTIR of oxygen plasma treated

Table (1) show the descriptive data of mean, standard deviation, standard error of all the tested properties, while the t-test analyses are listed in table (2) for all the tested groups.

Tensile strength test:

The result of this test showed that experimental group exhibited the highest tensile strength mean value (62.1143 N/mm²); while the control group exhibited the (42.9571 N/mm²) as shown in table (1) and fig. (9)



Statistical analysis of independent t test indicated a highly significant difference between the studied groups (P< 0.01)

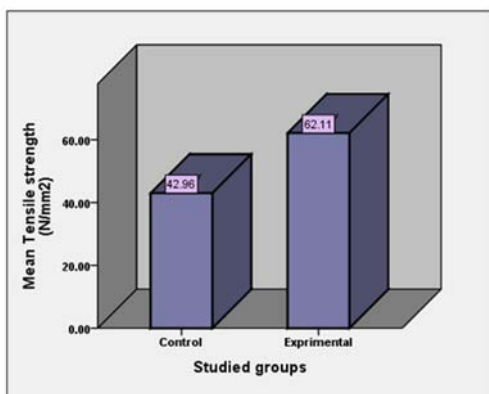


Figure 9: Histogram for the mean value of tensile strength of the control and experimental groups reinforced PMMA

Water sorption test

The result of this test showed that experimental group had the highest mean value (0.153714mg/cm²); while the control group had the lowest one (0.086171mg/cm²) as shown in

table (1) and figure (10), Statistical analysis of independent t test indicated a significant difference between the studied groups (P< 0.05 but > 0.01) as appeared in table (2).

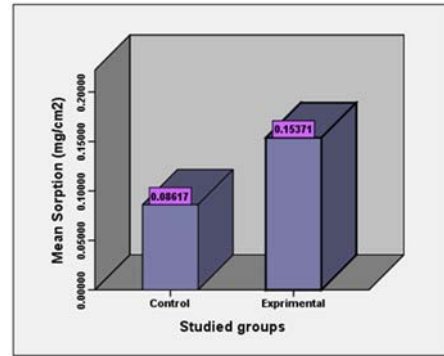


Figure 10: Bar chart for the mean value of Water Sorption test of the control PMMA and experimental groups

Water solubility test

Descriptive data of mean, standard error and deviation, are listed in Table 1. Water solubility seems to be decreased in experimental group with mean value (0.013943 mg/cm²) when it was compared with the control group which was (0.05471mg/cm²) as appeared in table (1) and figure (11) .T test was not significant with a P value of (0.06) suggested a none significant statistical difference between the two groups as seen in table (2).

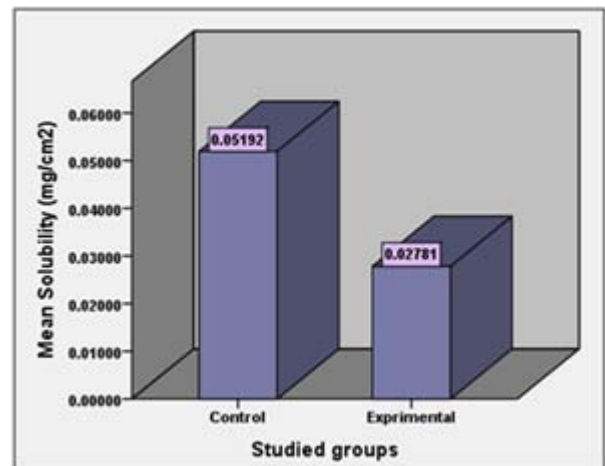


Figure 11: Bar chart for the mean value of Water Solubility test of the control PMMA and experimental groups

Surface roughness

Examination for the surface roughness of the specimens had conducted for each group. Table (1) shows the mean, standard deviation (S.D), and standard error (S.E), of surface roughness for control and experimental groups of acrylic resin. As illustrated within table (1) and figure (12), the higher mean value (1.50467 μm) is the experimental group comparing to the control group which is (1.33592um). T-test was indicated significant difference with a P-value (0.022) between the two groups as present in table (2).

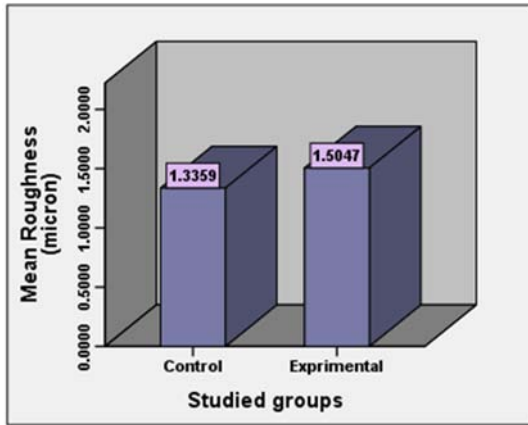


Figure 12: Bar chart for the mean value of Surface roughness test of the control PMMA and experimental groups

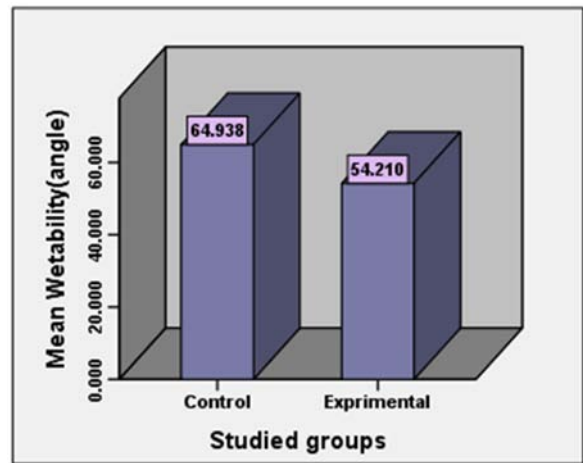


Figure 13: Bar chart for the mean value of Wettability test of the control PMMA and experimental groups

Wettability test

The results indicated that the experimental group contact angle (54.2100) lower than the control (64.93800) as presented in table (1) and figure (13).

Table 1: Descriptive statistic of the tested groups of all tested properties

Group Statistics					
Properties Tested	Category	N	Mean	Std. Deviation	Std. Error Mean
Tensile strength(N/mm ²)	Experimental	7	62.1143	2.60540	.98475
	Control	7	42.9571	1.34642	.50890
Sorption(mg/cm ²)	Experimental	7	.15349	.052809	.019960
	Control	7	.08597	.042885	.016209
Solubility(mg/cm ²)	Experimental	7	.0278133	.00570718	.00215711
	Control	7	.0519214	.00212496	.00080316
Roughness(μm)	Experimental	7	1.504671	.1376957	.0520441
	Control	7	1.335929	.0996028	.0376463
Wettability(angle)	Experimental	7	54.21000	4.858734	1.836429
	Control	7	64.93800	2.626914	.992880

Table 2: Independent sample t test for the properties used

Tested properties	t-test for Equality of Means						
	t	df	Sig. (2-tailed)	Mean Difference	Std. Error Difference	95% Confidence Interval of the Difference	
						Lower	Upper
Tensile strength(N/mm ²)	17.282	12	0	19.15714	1.10847	16.74199	21.57229
Sorption(mg/cm ²)	-2.629	12	0.022	-0.0675429	0.0256908	-0.1235183	-0.0115675
Solubility(mg/cm ²)	10.474	12	0	0.0241081	0.0023018	0.019093	0.0291233
Roughness(micron)	2.627	12	0.022	0.1687429	0.0642326	0.0287919	0.3086938
Wettability(angle)	-5.139	12	0	-10.728	2.08765	-15.276598	-6.179402

The limitations within vitro study, shows that properties of heat polymerizing resin and the modified one by salinized Nano zirconium oxide and plasma treated polypropylene fiber has been changed with the percentages used. Silanes are frequently used in various apps to give the chance for chemical bonding. In addition, the Silane has the ability to function as mediators between dispersed and organic phases^[5].

Usage of fibers as an accepted method within the process of reinforcement material of denture, mostly due to the progression of fibers as strengthening materials. Fibers reinforcement improve the characteristics of mechanical

strength such as impact strength, transverse strength, and ultimate tensile strength^[8].

In this study plasma treatment is used for polypropylene fibers since plasma convenient technique and accepted procedure^[26]. As FTIR test was applied for characterization of the modification that has been done to the nano particles and the used fiber. Peaks appeared with two prominent peaks at 2945 cm⁻¹ and 2841 cm⁻¹ that attributed to the characteristic (C=O) with stretching occurs at 1720 cm⁻¹, while the characteristic of (C=C) stretching occurs at 1637 cm⁻¹, plus the characteristic (Si-O-CHR3R) which its stretching to be found between (400 -470) cm-1 as in figure (4, 5 and 6)^[18].

Oxygen containing plasma most frequently adopted to effect the properties of polymer surface, these results might be due to the presence of chemical oxidation reactions and/or chemical etching process, in which plasma induced adhesion by making a new chemical functional groups such as the hydroxyl group which made more surface energy^[9, 31].

The size of filler particles with its shape and distribution in the polymer matrix and in the strong adhesion at the interface performed a more focused role the particulate filled polymer composites on its mechanical properties^[27]. The size of particles need to be very fine for good processing^[28]. Other study shows that a composite material of PMMA integrated with 5 wt.% of (80/20; Al₂O₃/ZrO₂) filler has the best combination of tensile properties^[29]. So incorporation of low percent nano filler 2.0% decrease the stiffness and the homogenous dispersion of the material in the matrix, with the presence of this pp fiber which had high tensile property in its chain which affect the structure of PMMA, all these may lead to increase the tensile property. This mechanism may explain the improvement of the mechanical properties of the heat polymerizing acrylic resin in this study, as agreed^[9, 17].

The increase in water sorption may be due to presence of polypropylene fibers, which is hydrophilic resin, result in an increase in water uptake since diffusion property of water molecules through this material is more than that through PMMA matrix as the structure was changed and this agreed with Ferracane study (2006) who stated that dental polymer have been shown to be highly effective to hygroscopic and hydrolytic effects to varying extents dependent upon their structural forma and chemistry^[30], also may be attributed to the water absorption of PP fibers which was enhanced by oxygen plasma^[5]. Many studies revealed that inclusion of oxygen plasma treated poly propylene fibers to PMMA had raised the water absorption effectively^[9].

The increased in water sorption could be due of the fact that pp fiber hydrophilic polymer result in increase in water uptake as the property of diffusion for the water molecules within this material is more higher than that within PMMA matrix^[23].

While water solubility decreased which could be related to the presence of stiff and hard particles in the composite and fine distribution of the nanoparticles that might allow these nanoparticles to enter within linear macromolecules chains of the polymer and to fill the spaces between those chains.

The mentioned decreased in water solubility could be mostly because that ZrO₂ material had insoluble property in water, so that the incorporation of silanized ZrO₂ to matrix would to reduction in the overall solubility of acrylic resin, yet the results were within the limitation of ADA specification No. 12, 1999.

Table (2) illustrated that the composite surface roughness was significantly increased ($p < 0.05 > 0.01$) when 2.0% percentage of silanized ZrO₂ nanoparticles and 2.0 % poly propylene fiber has been incorporated, most studies have suggested that, the effect of nanoparticles is less than that of fiber in surface roughness in case it is well dispersed and in low percentages, may be due to that the ZrO₂ nanoparticles

has well dispersion as well has very small size^[23, 24]. Additionally, the noticeable raising in surface roughness mean value of specimens after integration of plasma treated pp fibers in comparison with control group, this increment could be referring to fact that oxygen plasma treatment increase the surface roughness of the polymer^[25]. This result might be because of that 2.0 % nano-ZrO₂ particles has well dispersion as well has very small size, and 2.0% pp fibers, this percentage lead to increase in the surface roughness test which is not involved with the inner surface but involved with the outer surface of composite thus when small percentage of nano-ZrO₂ particles were added to acrylic resin only few of these particles will be concerned with the specimen surface^[24]

Wettability revealed highly significant decrease in the contact angle between the control and experimental groups which may be related to the hydrophilicity property of the pp fibers described in^[22] which lead to distribute the drop of water on the surface. And the change in the structure of the composite material, and this is in agreement with the other study^[5].

4. Conclusion

Within the limitation of this in vitro study to prepare a composite with high performance when compare it with the conventional hot cured acrylic denture base material. Highly significant increase in tensile strength and wettability angle, while statistical significant increase in water sorption and surface roughness occurred, and highly significant decrease in solubility are related to new composite formed from 2.0 % silanized nano-ZrO₂ particles and 2.0 % plasma treated PP fibers.

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