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Evaluation of the Effect of silanized Zirconium Oxide nano-fillers and Plasma Treated polypropylene fibers Addition on some Properties of Heat Cured Acrylic Resin Denture Base Material

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Abstract

Background: The polymethylmethacrylate is the most reliable material for the construction of dentures but it has the disadvantages of lacking strength and radio-opacity. The purpose of this study was to evaluate the effect of addition of modified nano-zirconium oxide (zro_2) and plasma treated polypropylene fibers on some mechanical properties of heat cure acrylic resin materials.

Materials and methods:- Zirconium oxide nanoparticles were coated with a layer of propyltrioxysilane before dispersed and sonicated in monomer(MMA)in percentage of 2% by weight . The plasma treated polypropylene fibers were in percentage of 2% by weight added to polymer powder. Sixty specimens were constructed and divided into 3 groups according to the using tests; each group was subdivided into two subgroups. The tests used were impact strength, transverse strength and surface hardness. Data were analyzed T-test.

Results:- After the addition of plasma treated polypropylene fibers and modified nano-Zro_2 fillers there was a highly significant increase in impact strength, transverse strength and surface hardness.

Conclusion: It can be concluded that the addition of modified nano-Zro_2 fillers and plasma treated polypropylene fibers effective method to enhance the fracture resistance of acrylic denture base material.

Key words: impact strength, transverse strength, hardness, polypropylene fibers, nano-filler.

Introduction

The currently available denture base material is the polymethyl methacrylate . Despite the satisfying esthetic demands, biocompatibility and adequate strength it is not ideal in fulfilling the mechanical requirements of prosthesis [(1)]. The main problem with PMMA is unsatisfactory strength so many attempts have been used to improve it's strength such as incorporation of metal wire, addition of nanofillers and reinforcement with fibers such as Glass fiber, Carbon fiber, aramide fibers, nylon fibers, polyethylene fibers and polypropylene fibers [(2)]. PP fibers are of olefin type have the advantage of being chemically stable and of low cost but they have the surface energy is low [(3)], therefore surface treatment are necessary to increase surface wetting and adhesion properties [(4)]. There are

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several methods used to increase the surface energy of pp fibers: mechanical, chemical or plasma treatment. Chemical method is the most utilized one, however the ecological requirements need an alternative environmentally benign methods ,recently cold plasma treatment represents an alter active method which can replace both mechanical and chemical methods. The advantage of this method is that plasma treatment changes the uppermost atomic layers of a material without modifying bulk properties and rapid treatment time [(5)] .The main purpose of plasma treatment is to improve the fiber-matrix adhesion by introducing polar groups that can form strong covalent bonds between fiber and matrix and roughening the surface of fibers to improve the mechanical bond fiber and matrix [(6)].

The purpose of using nano technologies in dental materials is to have higher mechanical properties ,less shrinkage of dental composite , higher abrasion resistance ,improved optical and esthetic properties of the composites and ceramics[(7)]. One of the nano materials incorporated with polymer is the zirconia(Zro_2)because it is biocompatible material and being white will not effect the esthetic ,the fabricated composite will be with high refractive index, higher hardness and scratch resistance[(8)].

It is important that the filler particles be bonded to resin matrix and this bond can be provided by a properly applied coupling agent which can improve physical and mechanical properties and prevent leaching by inhibiting water from penetrating along the filler resin interface[(9)].

The mono- functional saline, Y-trimethoxysilypropylmethacrylate have been used widely in dental resin based composite materials.[(10,11)]

The present study was designed to study and test the effect of addition plasma treated fibers to polymer powder and silaneted nano-zirconia to methyl methacrylate(monomer) and polymerized by conventional mean on some mechanical properties such as :impact strength, transverse strength and indentation hardness of heat cure acrylic resin.

Materials and Methods

Surface modification of nanofillers (Zro_2)

Reactive groups introduced onto the surface of nano fillers was achieved by the reaction of (trimethoxysilypropylmethacrylate) silane coupling agent with Zirconium oxide nano fillers.[(12)]

Plasma surface treatment of pp fibers

The oxygen plasma treatment of pp fiber was done by using device called Dc- glow discharge system .6 minutes selected as the treatment time for pp fiber in present study.

Pattern preparation

Sixty acrylic specimens were constructed by conventional flasking technique using heat cure acrylic resin (super- acryl®plus) .The specimens were classified into 3 groups according to the test be used and each group subdivided into 2 subgroups.

Two different plastic patterns were used to prepare the mould for acrylic specimens. They constructed by cutting plastic plate into the required shape and dimension according to the test be used by laser cutting machine.

For transverse strength test, specimens with (65mm*10mm*2.5⁻+0.1mm) as length, width and height respectively were used"[(13)].For impact strength test, specimens with (80mm in length*10mm in width*4mm in thickness) were used (Iso 179,2000)

for unnotched specimens[13] and for surface hardness test specimens with a dimensions 0f (65mm*10mm*2.5mm+_0.1mm) were prepared[13].

These plastic patterns were used in production of mould for construction of the specimens by conventional flasking technique.[13]

Proportion and mixing of acrylic resin

Addition of nano fillers and fibers:-

Modified Zro_2 powder was added by 2%weight to monomer. An electronic balance with accuracy of (0.0001g) was used (Sartorius Bp 30155, Germany).

After the addition of modified Zro_2 to monomer, the fillers were well dispersed in the monomer by ultra-sonication, using a probe sonication apparatus (120 w,60 khz)for 3 minutes[14],the required wait of the powder of the polymer and plasma treated pp fibers 2% by weight were mixed together randomly by using mortar and pestle until achieving homogenous mixture , the monomer with dispersed nanofiller was mixed with acrylic powder at once. The mixing of acrylic risen was carried out and manipulated according to the manufacturers instructions.

Mechanical tests utilized to examine the properties:

1. Impact strength: 20 specimens were constructed for the measurement of impact strength and all these specimens were immersed in distilled water in incubator at 37c for 48 hours before being tested [13]. This test was conducted according to ISO 179-1, 2000 with charpy impact testing device.A free swinging pendulum of 2 Joules testing capacity was used to strick the specimens, which was supported horizontally at its both ends. The scale reading gives the impact energy in Joules. The charpy impact strength of unnotched specimen was calculated in KJ/m^2 by the following equation :

   Impact strength= $E/b.d *10^3$ (Anusavice 2008)

   Where:

   E:- is the impact absorbed energy in Joules.

   B:- is the width in millimeters of the test specimen.

   D:- is the thickness in millimeters of the test specimen.

2. Transverse strength: : 20 specimens were constructed for the measurement of transverse strength and all these specimens were immersed in distilled water in incubator at 37c for 48 hours before being tested [13] . The transverse strength was measured by three point bending using a universal Instron testing machine. The specimen was positioned horizontally on the bending fixture, which consist of 2 parallel supports (50 mm) apart. The load was applied with cross head speed of 1mm/min by rod placed centrally between the supports making deflection until fracture occurred. The transverse strength was calculated using the following formula:-

   Transverse strength= $3pI/2bd^2$ (Anusavice 2008)

   P: is the peak load

   I: is the span length

   B: is the span width

   D: is the span thickness

3. Surface hardness test: : 20 specimens were constructed for the measurement of surface hardness and all these specimens were immersed in distilled water in incubator at 37c for 48 hours before being tested [13] . Shore (d) durometer hardness tester was used to determine the surface hardness, which is suitable for acrylic resin material. The instrument consist of spring- loaded indenter 0.8mm in

diameter and it is attached to digital scale. The method is to press firmly and quickly on the indenter and record the reading. Three readings were taken on each specimen(one in the center and other at each end)then the mean was calculated to each specimen.

Results

Spss version 20 was used to do the statistical analysis. Results obtained from the measured data were classified into control group (acrylic resin only) and experimental group (acrylic resin+2%silanted Zro_2+2%plasma treated polypropylene fibers).

Impact strength:- The result of this test showed that the experimental group exhibited higher impact strength mean value (21.25)than the control group(9.35) as shown in table (1). t-test showed a highly significant difference among the studied groups $p<0.01$.

Surface hardness:- The result of this test showed that the experimental group exhibited higher surface hardness mean value (92.20)than the control group(86.71) as shown in table (1). t-test showed a highly significant difference among the studied groups $p<0.01$.

Transverse strength:- The result of this test showed that the experimental group exhibited higher transverse strength mean value (219.12)than the control group(132.36) as shown in table (1). t-test showed a highly significant difference among the studied groups $p<0.01$.

Discussion

The concept of using nano composites as matrix material with fiber reinforcement in a new three-phase composite material has been showed to be very successful [15] and was used in this study. Plasma treatment for poly propylene fibers was used rather than other treatments since this method is a convenient procedure and environmentally friendly technique [16].

Zirconia (Zro_2) was choose because it is biocompatible material and being white is less likely to alter esthetic. The nano sized zirconia has been used to fabricate nano composite with high refractive index, hardness, and improved scratch resistance [8].

Salinization of the nano-filler particles gives a better dispersion, improve its compatibility with organic polymer and prevent aggregation [17].

Impact strength: The results of impact strength test shows that the addition of salinized Zro_2 and plasma treated fibers increased the value of the impact strength compared to control group . this increase can be due to the surface modification of the nano filler with the TMPSM coupling agent which gives better dispersion of particles, avoid agglomeration and provides better interfacial adhesion of the fillers to polymer matrix due to the formation of cross-links or supra molecular bonding which shield the nano fillers which in turn prevent crack propagation by transferring the stress from matrix to fillers [18] . on the other hand when the particle size decrease the total particle/matrix interfacial surface area available for energy dissipation increase also the stress for particle /matrix debonding increase and this could be a reason for the elevated values of the impact strength test in the experimental group[19] . ultra sonication of nano filler may also lead to the proper distribution of nano fillers in the polymer matrix which helped in a better embedding in a polymer matrix and this can affect the impact strength positively.

Also the presence of plasma treated fibers in experimental group can cause this increase in the impact strength because they prevent the crack propagation and change cracks direction leading to smaller cracks between fibers. These results are in agreement with the results obtained by (Mowade et al, 2012) [20].

On the other hand the plasma introduce functional groups on the surface of fibers which makes the surface polar this can improve the surface energy of fibers and its compatibility with other materials [6] which increase the impact strength . These findings are in agreement with results obtained by (Mukhlif OR. 2014)[21] as they showed significant improvement in impact strength when added plasma treated poly propylene fibers with alumina nano – fillers.

Transverse strength : The results revealed that the addition of 2% wt modified Zro_2 and 2% plasma treated poly propylene fibers produce high significant difference in transverse strength mean values compared with the control group . this may be related to the fact that the good distribution of the nano –fillers make them able to enter between linear macromoleculars chains of the polymer and fill spaces between chains ,so increasing strength and rigidity of the resin by restricting the segmental motions of the macromolecular chains and this cause improve for the fracture resistance and lead to increase of the transverse strength .

On the other hand this increase could be due to transfer stress from more flexible polymer matrix to the higher modulus filler particles. In addition this increase in transverse strength can be explained on the basis of transformation toughening as sufficient stress develops and micro cracks start to propagate, a transformation of modified Zro_2 from the meta stable tetragonal crystal phase to the stable monoclinic phase occurs which depletes the energy of crack propagation , also in this process expansion of modified Zro_2 crystals occurs and places the crack under a state of compressive stress and arrests the crack propagation [11].

The result of this study agrees with the result obtained by (Alhareb and Ahmed, 2011)[22] as they found that the transverse strength of heat cure acrylic resin increased after the addition of 5% wt of Al_2o_3/Zro_2 micro sized powders in different proportions , also the results go in agreement with (Unalan et al,2010)[23] and the results of (Kamble et al,2012)[24] as they found that reinforcement of acrylic with 2%wt of glass and polyethylene fibers increased the flexural strength of the specimens compared to unreinforced PMMA and bisacryl composite resins.

Surface hardness: The results showed a highly significant increase in surface hardness mean value of the experimental group compared with control group, this increase could be due to the presence of polypropylene fibers at or near the surface of the composite which extremely stiff and hard , also could be attributed to plasma treatment which increase the hardness of fibers.

Ahmed and Wel[25] showed that there was an improvement in the interfacial bonding between the matrix and glass fibers after the addition of saline coupling agent only without giving any impression to the value of hardness.

In addition the presence of modified nano-fillers could cause the increase in hardness of the nano composite which would be dominated by the density of the network.

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Table 1: Mean distribution and t- test for surface hardness, impact strength and transverse strength in control and experimental groups

Variables	**Groups**	**Descriptive Statistics**						**Groups difference**		
		N	**Mean**	**S.D.**	**S.E.**	**Min.**	**Max.**	**t-test**	**d.f.**	**p-value**
Impact Strength	Control	10	9.35	0.65	0.21	8.40	10.66	-45.445	18	0.000 (HS)
	Experimental	10	21.25	0.51	0.16	20.29	22.10			
Hardness	Control	10	86.71	2.79	0.88	83.00	90.30	-5.949	18	0.000 (HS)
	Experimental	10	92.20	0.86	0.27	90.70	93.10			
Transverse strength	Control	10	132.36	7.11	2.25	122.40	141.60	-28.517	18	0.000 (HS)
	Experimental	10	219.12	6.48	2.05	205.20	228.00			