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A Comparative Study to Evaluate the Mechanical Properties of Zirconium Oxide Added Polymethyl Methacrylate by Two Different Methods at Two different Concentrations – In Vitro Study.

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ABSTRACT

Background: Poly methylmethacrylate (PMMA) is one of the most commonly used denture base material. Various attempts have been used to strengthen acrylic resin by incorporating filler particles. **Method:** In the present study zirconium oxide is used as a filler material and a silane coupling agent is added for bonding with PMMA by two different methods that is sol gel method and functionalizing zirconium oxide with silane coupling agent. **Result:** The present study demonstrated a significant increase in impact strength, surface hardness as the percentage of ZrO2 fillers increased. **Conclusion:** From the study it was concluded that incorporation of Zirconium oxide to PMMA will increase in flexural strength, impact strength and hardness of PMMA and the mechanical property obtained by functionalizing nanoparticles method is better than sol-gel method.

Keywords: PMMA, Silane coupling agent, Zirconium oxide

INTRODUCTION

Polymethyl methacrylate is the commonest denture base material that was introduced by Walter, Wright and Vernon brothers in 1937.[1,2] Apart from various advantages, poor impact strength, flexural strength still exist.[3,4]Poor mechanical properties like low impact strength, and flexural strength are the shortcomings of this material. Various methods have been attempted to increase material strength and decrease the risk of denture fracture like reinforcing it by adding fillers such as silver, copper, aluminum particles,[5,6] calcium carbonate,[7] glass fibers,[8,9,10] carbon fibers,[11,12] changing the chemistry of denture base polymer by co-polymerization and cross – linking of resin materials.

Zirconium oxide is a polymorphic material which is available in three forms: monoclinic, tetragonal and cubic.

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It has high flexural strength and fracture toughness as a result of transformation toughening.

In the present study zirconium oxide is tried as filler and silane coupling agent is added for proper bonding with PMMA.

The aim of preset study is to evaluate the mechanical property of zirconium oxide added with polymethyl methacrylate. A) To compare and evaluate the impact strength, flexural strength and surface hardness of various concentrations of zirconium oxide nanoparticles added PMMA by functionalizing method. B) To compare and evaluate the impact strength, flexural strength and surface hardness of various concentration of zirconium oxide nanoparticles added PMMA by sol gel method. C) To compare and evaluate the impact strength between the functionalizing zirconium oxide nanoparticles added PMMA and the sol gel method zirconium oxide added PMMA. D) To compare and evaluate the flexural strength between the functionalizing zirconium oxide nanoparticles added PMMA and the sol gel method zirconium oxide added PMMA. E) To compare and evaluate the surface hardness between the functionalizing zirconium oxide nanoparticles added PMMA and the sol gel method zirconium oxide added PMMA.

MATERIALS AND METHODS

This is in vitro study performed to evaluate and compare the inclusion of zirconium oxide to PMMA with two different additive techniques in view of enhancement of some physical properties. Materials used in the study are tabulated [Table 1].

Table 1: Materials and their manufactures

S.No	Name of the material	Manufacturer
1	Heat polymerizing poly methyl methacrylate resin	Hiflex,prevestdentproltd,india
2	Zirconium oxide	Strem-chemicals pvtltd.india
3	Trimethoxysilylpropylmethacrylate	Sigma-aldrichpvtco,Germany
4	Tetrahydrofuran	SRL-chemicals.India
5	Ethyl alcohol	Jiandsnhaaxi international trade, china
6	Zirconium oxide - monoclinic	Reneto venture pvt ltd India

Armamentarium used for the study were:

1) Custom fabricate stainless steel dies
2) Glass plate
3) Rubber bowl
4) Plaster mixing spatula
5) Camel hair brush
6) Conventional metallic flasks with clamp
7) Cement mixing stainless steel spatula
8) Porcelain jar
9) Acrylic trimmers
10) Ultrasonic bath
11) Electronic balance

Sol gel method:

It is a technique for the synthesis of organic polymer and inorganic compounds. With this technique, hybrid materials can also be synthesized through the in-situ formation of inorganic species with a polymer matrix.

Successful approach for in-situ polymerization of metal alkoide precursors in the presence of polymer matrices via sol-gel method involves hydrolysis of metal alkoxides to produce metal hydroxides, followed by the condensation of hydroxyl group to form inorganic clusters.

When inorganic clusters are mixed with polymer solution, organic/inorganic hybrid nano composites can be obtained.

Zrbutoxide was used as a precursor to yield ZrO_2 with respect to yield 0.5,1,1,5 % wt. of ZrO_2, required amount of precursor and 3-trimethoxylsilyl propylmethacrylate TMSPM coupling agent were suspended in 210 ml of 95% ethyl alcohol. The weight percentage of ZrO_2 is with respect to the polymer for 0.5 wt. % $Zr0_2$, 0.15 ml precursor was added to 10 ml of THF and same was ultrasonicated for 1 hour, in order to complete the hydrolysis of the precursor. Further the required amount of monomer was subsequently added and the sonication was continued for 1 hr.The resultant mixture containing monomer with $Zr0_2$ was kept constantly to evaporate the solvent and the solvent and appropriate amount of polymer was added to the mixture which is free from solvent.

Functionalizing zirconium nano particle method:

Surface modification of fillers ($Zr0_2$):

Silone coupling agent was used to introduce double bond onto the surface of $Zr0_2$ nanoparticles.9 gms of $Zr0_2$ and .45 gms of TMSPM were suspended in 210 ml of 98% ethyl alcohol and the ph. of the solution is brought to 4 by adding the required amount of acetic acid, then the mixture was subjected to sonication [Figure 1] for 3 hrs and the same was refluxed for 24 hrs. After that, the nanoZr02 particles were centrifuged, and the precipitate was extracted with ethyl alcohol for 16 hrs to remove the unreacted silone.

Then the modified nano-$Zr0_2$ were air dried at 80 degree C under vacuum oven for 24 hrs. The infrared spectra were performed to determine whether or not functional groups of the TMSPM have been attached to the Nano filler by analyzing the characteristic vibrations of functional group.

Sample preparation

Conventional flasking technique for complete denture was followed and short curing cycle followed. Addition of modified Zirconium oxide Nano filler was done by weight in three groups, include 0.5%, 1%, 1.5% to monomer [Table 2].

Table 2: Modified Zirconium oxide nano filler

$Zr0_2$ %	Amount of $Zr0_2$	Amount of polymer	Amount of monomer
0.5%	0.5gm	9.5gm	5gm
1%	1gm	9gm	5gm
1.5%	1.5gm	8.5gm	5gm

Grouping and number of samples:

Sample for this in vitro study was prepared according to ISO specification (1567). A total number of 150 samples were prepared with dimension of 80-10-3 mm for flexural strength and impact strength testing out of which 20 samples were prepared for control (Group I) and 60 samples for sol-gel method (Group II) and from 60 samples , 20 samples were allotted to their subgroup, denoted as A,B,C.

Figure 1: Surface modification of fillers ($Zr0_2$)

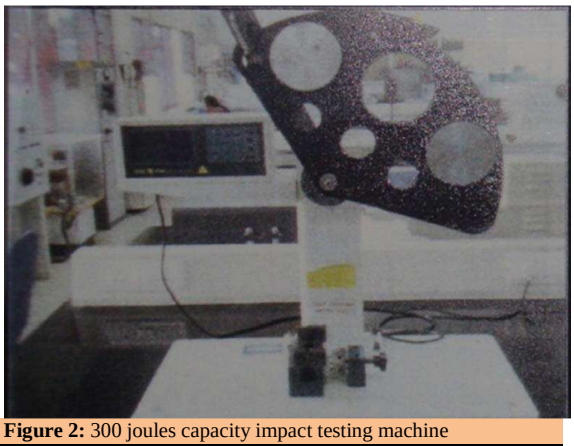

Figure 2: 300 joules capacity impact testing machine

Remaining 60 for functionalized nano particle method (Group III) and from 60 samples, 20 samples were allotted to their sub groups denoted as A1, B1, C1 and 70 samples of 10mm×10mm×3mm were prepared for surface hardness test.

The samples were prepared from tested / fractured specimens of Group I, Group II, Group III.

Ten samples allotted to Group I, thirty for Group II and their sub groups A, B,C, like ten for each sub group. Similarly thirty for group for Group III and subgroups.

Preparation of specimens:

Steel die was milled from a metal blank measuring 80×10×3mm to simulate the bar. This was used to create stone mold by investing in type II dental stone. This mold was filled with modeling wax then invested in conventional dental flask.

After dewaxing procedure, heat cured acrylic resin samples were prepared in a usual manner following the normal processing procedure.

All the prepared specimens were then subjected for Impact strength, flexural strength and surface hardness test.

Figure 3: Universal testing machine

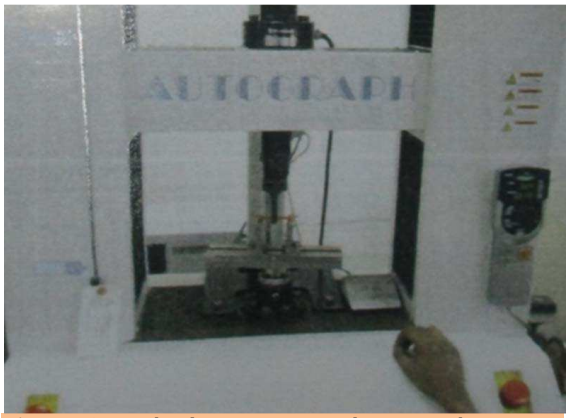

Figure 4: 3-point bending test in universal testing machine

Impact strength of acrylic resin was calculated using 300 joules capacity impact testing machine [Figure2] (ASTM D-256) and was tested by the charpy system.

For flexural strength the specimens were submitted to 3-point bending test in a universal testing machine [Figure3,4], flexural value was obtained by formula

$F = 3\ WL/2bd^2$

Where F = Flexural strength

W =ultimate load before the failure

L = distance between the support points

B = specimen width

D = specimen thickness

Surface hardness:

Vickers hardness test was used for calculating the surface hardness of the specimens. It uses a square

based pyramid diamond indenter with an angle of 136 degree between the opposite faces at the vertex, which is pressed into the surface of the test piece using a prescribed force. The time for the initial application of the force is 2 to 8 sec and the test force is maintained for 10 to 15 sec. after the force has been removed, the diagonal lengths of the indentation are measured and the arithmetic mean, d, is calculated the Vickers hardness number, HV is the number then determined by the ratio F/A.

RESULTS

This in vitro study was conducted to evaluate the effect of reinforcement of acrylic resin with zirconium oxide on some mechanical properties (Flexural strength, impact strength, and surface hardness).

Total 150 samples were prepared according to ISO standardization which were divided into three groups Group I, Group II and Group III.

Within the limitation of the study following result was estimated.

1. Zirconium oxide nanoparticles added by functionalizing method (Group III) has showed significant increase in impact strength, surface hardness, but the mean value of the flexural strength of this method is not significant.
2. Zirconium oxide added PMMA by sol-gel method show significant increase in impact strength, surface hardness but the mean value of the flexural strength of this method is not statistically significant.
3. The impact strength showed significant increase for zirconium oxide nanoparticles added PMMA by functionalizing method (Group III) followed by the sol – gel method.
4. Mean value of the flexural strength is higher for Zirconium oxide added PMMA by functionalizing method (Group III) followed by the sol – gel method, but the mean value is not statistically significant.
5. Hardness of zirconium oxide nanoparticles by functionalizing method is greater than the sol gel method.

DISCUSSION

Acrylic resin is the most frequently used substance for the manufacture of denture due to its aesthetics, ease of manipulation and low price. Till now, polymers have been strengthened by adding materials such as metal oxides, metal strengtheners, carbon graphite fibres, aramid fibre, ultra-high molecular weight polyethylene fiber (UHMWPE) and glass fibers.[13] The size, shape and dispersal of filler particles in the polymer matrix and strong bond at the interface play foremost part on the mechanical properties of particulate filled polymer composites. The success of PMMA resin denture base material depended upon the mechanical properties and its resistance to biological degradation. This study was performed to evaluate the flexural strength, impact strength, surface hardness of zirconium oxide, added to PMMA by two different method at various concentration.

Fractures in an acrylic denture base are a common clinical problem. Flexural strength of denture base resin was measured in this study because it reflects the primary mode of clinical failure.[14]Authors establish that the fracture durability seems to be a appropriate measurement to validate the effects of resin modifications.[15]They conducted a study on reinforcement of complete denture with high performance poly-ethylene fibers and concluded that mechanical performance of reinforced denture based superior to conventional denture base resin showed increase in flexural strength, impact strength.[15]

John et al evaluated the effect of nylon fiber reinforcement on the strength of provisional crown and fixed partial denture. This study showed increase in strength.[16]Kannie et al studied on PMMA based nano composite filled with woven glass fibers nan particles using silane coupling agent. The result showed increase in hardness.[17]

Hardness of the polymerized resin has been found to be sensitive to the residual monomer content in the resin material. Measurement of hardness have been effectively used as an indirect method of appraising polymerization depth of resin-based composite materials and the degree of conversion of conventional heat polymerizing and self-curing acrylic resins. In addition, hardness has been used to predict the wear resistance of dental materials.[18]The Results of the present study demonstrated a significant increase in impact strength, surface hardness as the percentage of ZrO2 fillers increased. This up gradation is due to increase interfacial shear strength between resin & fillers. ZrO2 possesses strong ionic interatomic bonding, giving rise to its desirable material characteristics, that is, hardness and strength.

CONCLUSION

From the study it was concluded that incorporation of Zirconium oxide to PMMA will increase in flexural strength, impact strength and hardness of PMMA and the mechanical property obtained by functionalizing nanoparticles method is better than sol-gel method. Further studies are needed to investigate its effect on other mechanical and physical properties with different concentrations.

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