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Original Article

Dentistry Section

Evaluation of the Effect of Titanium Dioxide and Silicon Dioxide Nanoparticles on Impact Strength of Two Commercially Available Heat Cure Acrylic Resins

APARANA SHARMA[1], ANURAG HASTI[2], ASHISH CHOUDHARY[3], MEGHA CHOPRA[4], JAY VIKRAM[5], POOJA SRIVASTAVA[6]

ABSTRACT

Introduction: Poor mechanical properties are among the main limitations of denture base resin. There has been a continuous attempt to improve the mechanical properties of denture base resins. Nanotechnology has evolved healthcare industry to a large scale and its applications are a boon to modern medicine and dental science. Nanoparticles are nowadays, extensively used in prosthodontics as they are incorporated in Polymethyl Methacrylate denture bases to alter the properties such as impact strength.

Aim: To evaluate and compare the effect of Titanium Dioxide (TiO_2) and Silicon Dioxide (SiO_2) nanoparticles on impact strength of two commercially available heat cure acrylic resins.

Materials and Methods: The in-vitro study was conducted in Department of Prosthodontics, Crown and Bridge, School of Dental Sciences, Sharda University, Greater Noida, Uttar Pradesh, India between April 2019 to May 2021, that involved 120 samples. Materials compared were Dental Products of India (DPI) heat cure acrylic and Trevalon heat cure acrylic. Each group was further categorized into four groups to measure the impact strength i.e, without incorporation of nanoparticles and with incorporation of nanoparticles SiO_2 and TiO_2 and a combination of both. Samples obtained were tested for impact strength using Izod method. Statistical analysis was done using a one-way Analysis of Variance (ANOVA), Student's t-test and Post-hoc Bonferroni test.

Results: In the two types of materials studied, the mean impact strength of Trevalon was statistically significantly higher (p-value=0.045) than DPI. After the addition of nanoparticles, i.e; SiO_2 and TiO_2 the mean impact strength was higher in Trevalon (8.66 kJ/m^2 without the addition of nanoparticles, 5.79 kJ/m^2 addition of 1% TiO_2 nanoparticles, 5.77 kJ/m^2 addition of 1% SiO_2 nanoparticles and 5.75 kJ/m^2 when a 1% combination of both the above was added) than DPI (7.19 kJ/m^2 without the addition of nanoparticles, 5.86 kJ/m^2- addition of 1% TiO_2 nanoparticles, 5.77 kJ/m^2 addition of 1% SiO_2 nanoparticles and 5.66 kJ/m^2 when a 1% combination of both the above was incorporated).

Conclusion: Mean impact strength of Trevalon was higher than mean impact strength of DPI. Incorporation of TiO_2, SiO_2 nanoparticles or in combination decreases impact strength of both the commercially available heat cure denture base resin with statistically no significant difference.

Keywords: Mechanical properties, Nanotechnology, Polymethylmethacrylate

INTRODUCTION

Polymethyl Methacrylate (PMMA) is one of the most frequently used material in dentistry that is commonly used for prosthetic dental applications, that includes the fabrication of artificial teeth, denture bases, dentures, obturators, orthodontic retainers, temporary/provisional crowns, and for the repair of dental prostheses [1]. Many desirable properties such as stability in the oral environment, ease of manipulation, polish ability, and fabrication with the use of inexpensive equipment has led to its extensive use in fabrication of prosthesis, and orthodontic appliances. Since its introduction there has been continuous trials to improve the mechanical properties of acrylic resins [2].

Various properties of heat cure acrylic resin like tensile strength, compressive strength, and surface hardness are important but impact strength has a significant role to play clinically. Impact strength is the ability of the material to withstand a sudden applied load and is stated in terms of energy lost per unit of thickness [1]. Many studies have been carried out to improve the properties of denture base materials by adding suitable fillers into PMMA denture base that included, PMMA reinforcement with glass fibers, sapphire whiskers, aramid fibers, carbon fibers, metal wires, nylon, polyurethane fibers and zirconia that showed improved fracture resistance [3-5].

Nanotechnology has evolved healthcare industry to a large scale and its application are a boon to modern medicine and dental science. Futuristically, it is expected that it will pervade and further revolutionise the art and science of dentistry and will expand all the aspects of oral diseases, diagnosis, prevention and treatment. Nanomaterials are now successfully being used in caries inhibitors, antimicrobial resins, hard tissue remineralising agents, targeted drug delivery, scaffolds, biomembranes, restorative cements, adhesion promoters and boosters, bioactive glass, tissue wires and nano composites [6].

The scientific advancements have led to the era of nanotechnology and nano-phased materials, and thus; a great attention is directed towards the use of nano-sized fillers to reinforce the denture base resins leading to the production of a polymer nanocomposite with improved mechanical and physical properties as compared to those filled with microscale particles [7].

Inorganic carriers like Titanium dioxide (TiO_2) nanoparticles have been used as additives to biomaterials due to its certain characteristics such as white colour, low toxicity, antimicrobial properties, high stability and efficiency as well as availability and low cost [8]. Among compounds as inorganic carriers, such as apatite, zeolite, and phosphate, Silicon dioxide (SiO_2) is more promising due to its porous structure and adsorption properties. Nano SiO_2 particle possess extremely high surface activity and adsorb various ions and molecules [8].

In literature, various advantages as well as disadvantages of TiO_2 and SiO_2 nanoparticles on mechanical properties, especially flexural

strength of PMMA have been recorded; few studies have evaluated the combination effect of these particles [9,10]. The addition of nano-filler TiO_2 improved the thermal, mechanical and viscoelastic properties of the PMMA. A study done by Alzayyat ST et al., wherein, the incorporation of SiO_2 in denture base resin, led to a significant increase in the flexural strength [9]. Also, in a study done by Sodagar A et al., demonstrated that the incorporation of TiO_2 and SiO_2 nanoparticles into acrylic resins can adversely affect the flexural strength of the final products, and this effect is directly correlated with the concentration of nanoparticles [10]. As many studies done before evaluated the flexural strength therefore, the present study aimed to evaluate the and compare the effect of titanium dioxide and silicon dioxide nanoparticles on impact strength of two commercially available heat cure acrylic resins (DPI and Trevalon).

MATERIALS AND METHODS

This in-vitro study was conducted in the Department of Prosthodontics and Crown and Bridge, School of Dental Sciences, Sharda University, Greater Noida, Uttar Pradesh, India, between April 2019 to May 2021. The laboratory study was approved by the Institutional Ethical Review Committee (Ref No. SU/ SMS&R/ 76-A/ 2017/75). A total of 120 samples were made and divided into two groups of 60 samples each. Further each group was subdivided into four subgroups.

Preparation of Test Samples

A stainless steel master die [Table/Fig-1] of dimensions 60 mm in length, 7 mm in width and 4 mm in thickness was duplicated in putty elastomeric impression material (Zhermack Zetaplus Putty impression material) and molten modeling wax (Pyrax Polymers) was poured to prepare wax blocks. Two pour technique, was used for the flasking of wax blocks. After 15-20 minutes when the gypsum was completely set, it was placed in the dewaxing unit at 100°C for 5-7 minutes. Flask was carefully opened and clean boiling water was poured over it to completely eliminate the wax. A brush and soap solution was used to clean any traces of wax. It was allowed to cool for 10 minutes and then two layers of cold mold seal (DPI- The Bombay Burmah Trading Corporation Ltd. Cold Mold seal -batch no.- 5194) was applied all over the set gypsum. Gypsum moulds were thus, obtained [11].

I. **Preparation of samples for group A:** Appropriate amount of DPI heat cure acrylic resin (DPI-The Bombay Burmah Trading Corporation Ltd. P-5191, L-4193 was used to prepare dough in the ratio of 3:1 by volume (21 grams of powder and 10 mL of liquid) [Table/Fig-2]. The gypsum moulds were filled. The flask was closed and trial closure was carried out using hydraulic press. The flask was then clamped and then pressure was maintained for 30 minutes to allow proper penetration of monomer into polymer. An overnight bench curing was done for the same.

- **Subgroup D1:** Polymer was mixed with monomer without incorporation of nanoparticles.
- **Subgroup D2:** 1% TiO_2 (0.2 grams) nanoparticles (TiO_2 Nanoparticles: Souvenier chemicals, Ultra reagents, IISN code- 2823) were incorporated in polymer and then mixed with monomer.
- **Subgroup D3:** 1% SiO_2 (0.2 grams) nanoparticles (Ultra reagents, IISN code- 2811) was incorporated in polymer and then mixed with monomer.
- **Subgroup D4:** 1% combination of TiO_2 (0.1 grams) and SiO_2 (0.1 grams) nanoparticles was incorporated in polymer and then mixed with monomer.

II. **Preparation of samples for group B:** Appropriate amount of Trevalon heat cure acrylic resin (Dentsply India Pvt. Ltd. T-180414) was used to prepare dough in the ratio of 3:1 by volume (21 grams of powder and 10 mL of liquid) [Table/Fig-3]. The moulds were filled. The flask was closed and trial closure was carried out using hydraulic press. The flask was then clamped and then pressure was maintained for 30 minutes to allow proper penetration of monomer into polymer. An overnight bench curing was done for the same.

- **Subgroup T1:** Polymer was mixed with monomer without incorporation of any nanoparticles.
- **Subgroup T2:** 1% TiO_2 (0.2 grams) nanoparticles was incorporated in polymer and then mixed with monomer.
- **Subgroup T3:** 1% SiO_2 (0.2 grams) nanoparticles was incorporated in polymer and then mixed with monomer.
- **Subgroup T4:** 1% combination of TiO_2 (0.1 grams) and SiO_2 (0.1 grams) nanoparticles was incorporated in polymer and then mixed with monomer [Table/Fig-4].

Curing of the samples: The flask was immersed in an acryliser at room temperature. The temperature was raised to 73°C, held for 1 ½ hours, then to 100°C and this temperature was maintained for half an hour. After the curing cycle, the flask was removed from the acryliser water-bath and bench cooled for 30 minutes, immersed in cool tap water for 15 minutes preceding the deflasking [11].

Finishing and polishing of samples: The acrylic specimens were then retrieved, finished and polished. The dimension and quality of specimens were verified for any porosity, visible impurities and dimensional deformity. Finally there were two groups of 60 samples in each group [Table/Fig-5,6].

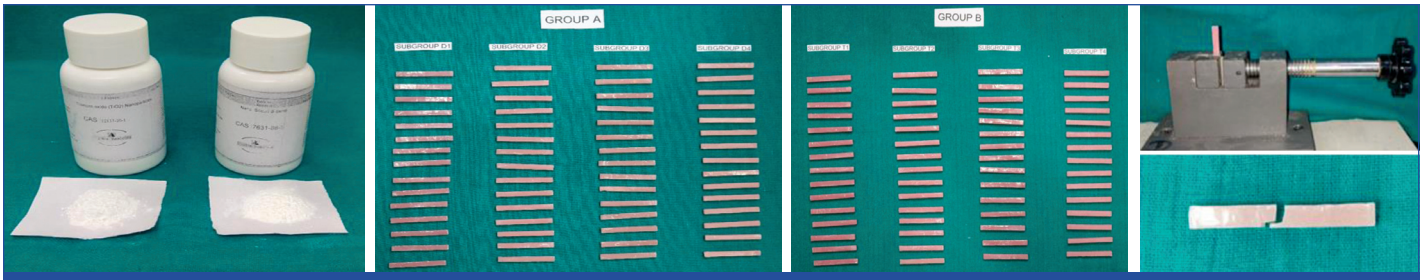
Evaluation of Samples for Impact Strength

Evaluation of test samples was done using Izod Impact Testing Machine (Saumya technocrats Model: IZB-B Sr. No.-ST-10-287). The un-notched sample was clamped vertically and hammer with 2 Joules was used to break the samples [Table/Fig-7]. Breaking energy was then recorded in joules and then Impact strength was calculated in kJ/m^2 [4].

STATISTICAL ANALYSIS

The data obtained were tabulated and subjected to statistical analysis using the IBM Statistical package for the Social Sciences

[Table/Fig-1]: Stainless steel master die. **[Table/Fig-2]:** DPI Heat cure acrylic resin (polymer and monomer). **[Table/Fig-3]:** Trevalon heat cure acrylic resin (polymer and monomer). (Images from left to right)



[Table/Fig-4]: Titanium dioxide and silicon dioxide nanoparticles. **[Table/Fig-5]:** Total 60 acrylic test samples for group A. **[Table/Fig-6]:** Total 60 acrylic test samples for group B. **[Table/Fig-7]:** Test sample clamped vertically for testing on the Izod impact testing machine and the broken test sample. (Images from left to right)

software for windows version 23.0 (Armonk, NY: IBM Corporation. Released 2015). The significance value was p-value <0.05 (α=0.05). Parametric tests namely Unpaired t-test, one-way Analysis of Variance (ANOVA) and Post-hoc Bonferroni test were used to statistically analyse the data.

RESULTS

The mean impact strength of group B (6.49 kJ/m^2) heat cure acrylic resin was significantly more than group A (6.12 kJ/m^2) (p-value-0.045) [Table/Fig-8]. The mean impact strength. was also compared between subgroups D1, D2, D3 and D4 and T1, T2, T3 and T4 using the one-way ANOVA test. There was a significant difference in the mean impact strength of all the subgroups [Table/Fig-9].

Groups	Impact strength			
	Mean±SD (kJ/m^2)	Mean difference (kJ/m^2)	t-test value	p-value
Group A (DPI Heat cure acrylic resin)	6.12±0.88	-0.37	-2.795	0.045*
Group B (Trevalon heat cure acrylic resin)	6.49±1.34			

[Table/Fig-8]: Mean and standard deviation values of impact strength (in kJ/m^2) in group A and group B by Unpaired t-test.

Subgroups	Impact strength		
	Mean±SD (kj/m^2)	F-value	p-value
Group A			
Subgroup D1	7.19±0.93	19.206	<0.001*
Subgroup D2	5.86±0.55		
Subgroup D3	5.77±0.53		
Subgroup D4	5.66±0.42		
Group B			
Subgroup T1	8.66±0.35	155.082	<0.001*
Subgroup T2	5.79±0.48		
Subgroup T3	5.77±0.54		
Subgroup T4	5.75±0.41		

[Table/Fig-9]: Statistical comparison (by ANOVA test, one way) of mean impact strength (in kJ/m^2), observed from the test samples of group A and group B for all subgroups.
p-value <0.05 was considered as statisticaly significant

The intragroup comparison of mean impact strength was also done for both the groups by Post-hoc Bonferroni test. In group A the mean impact strength was significantly more for subgroup D1 (p-value <0.001). The mean difference between subgroup D1 and D2, D1 and D3 and D1 and D4 was 1.33, 1.42 and 1.53, respectively and all were significant whereas; the mean difference of subgroup D2 and D3, D2 and D4 was 0.09 and 0.21 and the mean difference of subgroup D3 and D4 was 0.11 (all were insignificant) [Table/Fig-10]. In group B, similarly; the mean impact strength was significantly more in subgroup T1 (p-value <0.001). The mean difference between subgroup T1 and T2, T1 and T3 and T1 and T4 was 2.87, 2.90 and 2.92, respectively and all were significant whereas; the mean difference of subgroup T2 and T3, T2 and T4 was 0.03 and 0.05 and the mean difference of subgroup T3 and T4 was 0.02 (all were non significant) [Table/Fig-11]. The mean impact strength was significantly more in subgroup T1.

Subgroups comparison		Mean difference	p-value
Subgroup D1	Subgroup D2	1.33	<0.001*
	Subgroup D3	1.42	<0.001*
	Subgroup D4	1.53	<0.001*
Subgroup D2	Subgroup D3	0.09	1
	Subgroup D4	0.21	1
Subgroup D3	Subgroup D4	0.11	1

[Table/Fig-10]: Intergroup comparison of mean impact strength (kJ/m^2) using the Post-hoc Bonferroni test.
p-value <0.05 was considered as statisticaly significant

Subgroups comparison		Mean difference	p-value
Subgroup T1	Subgroup T2	2.87	<0.001*
	Subgroup T3	2.90	<0.001*
	Subgroup T4	2.92	<0.001*
Subgroup T2	Subgroup T3	0.03	1
	Subgroup T4	0.05	1
Subgroup T3	Subgroup T4	0.02	1

[Table/Fig-11]: Intergroup comparison of mean impact strength (kJ/m^2) using the Post-hoc Bonferroni test.
p-value <0.05 was considered as statisticaly significant

The intergroup statistical comparisons (unpaired t-test) between subgroup D1 and T1 depicted the mean impact strength to be significantly more in T1 (8.66 kJ/m^2) compared to D1 (7.19 kJ/m^2) (p-value <0.001). The comparisons between subgroups D2 and T2, D3 and T3 and D4 and T4 were insignificant [Table/Fig-12].

Subgroups	Mean±SD (kJ/m^2)	Mean difference	t-test value	p-value
Subgroup D1	7.19±0.93	-1.47	-5.729	<0.001*
Subgroup T1	8.66±0.35			
Subgroup D2	5.86±0.55	0.07	0.371	0.713
Subgroup T2	5.79±0.48			
Subgroup D3	5.77±0.53	0.00	0.017	0.986
Subgroup T3	5.77±0.54			
Subgroup D4	5.66±0.42	-0.09	-0.582	0.565
Subgroup T4	5.75±0.41			

[Table/Fig-12]: Comparison of the mean impact strength between subgroups using the unpaired t-test.
p-value <0.05 was considered as statistically significant

DISCUSSION

The above study was planned to evaluate and compare the effects of titanium dioxide and silicon dioxide nanoparticles individually and in combination on the impact strength of PMMA. The ideal denture base material should possess adequate physical and mechanical properties which are key attributes for the basic requirements of denture base materials like biocompatibility, good aesthetics, high bond strength with available denture teeth, radiopacity and ease of repair [12]. The impact and flexural strength of PMMA is not satisfactory as continuous efforts are being done to improve the same.

Fracture in an acrylic denture base is a common clinical problem. Therefore, numerous trials were done to improve the mechanical

properties of PMMA, but they can be summarised in three ways: replacing PMMA with an alternative material; chemically modifying it; and reinforcing the PMMA with other materials like fibers or metals, and recently nanoparticles [13].

Acrylic specimens in size 60 mm in length, 7 mm in width and 4 mm in thickness were prepared from two different heat cure acrylic denture base resin (DPI and Trevalon). The test samples of each group were divided into four subgroups and were incorporated with 1% nano TiO_2 and 1% nano SiO_2 and their mixture (1:1 W/W). The test samples thus obtained were tested for Impact Strength using Izod method. The results were obtained and data was statistically analysed.

Narendra R et al., in an in-vitro study evaluated the impact strength of conventionally heat cured and high impact heat cured polymethyl methacrylate denture base resins. They concluded that Trevalon had higher impact strength than DPI. The mean impact strength of Trevalon was found to be higher than DPI [14]. Praveen B et al., in a Scanning Electron Microscopy (SEM) study compared the impact strength and fracture morphology of different heat cure denture acrylic resins and it was concluded that the mean impact strength of Trevalon was higher than DPI. The SEM was also done to study the matrix structure. It was concluded that the impact strength of the acrylic resins was affected by the reinforcement of fibers [15].

The reason for high impact strength in Trevalon is co-polymerization of Methyl Methacrylate (MMA) and butadiene monomer molecules. The resultant polymer is further surface coated with MMA, thus enabling further cross linkage on polymerization. Due to this complex structure there is increased cross linking amongst the polymer chains resulting in rapid polymerization and increase in mechanical properties specially impact strength [16].

Some studies have also shown the adverse effect of addition of TiO_2 nanoparticles [10,17,18]. One of such examples is a study conducted by Ahmed MA et al., where it was concluded that the addition of TiO_2 nanoparticles adversely affected the impact strength of conventional acrylic resin [17]. The reason of decrease in the impact strength on addition of TiO_2 nanoparticles in PMMA could be the lack of chemical bond between TiO_2 nano particles and PMMA. Also, the TiO_2 nanoparticles agglomerate into larger particles, thus acting as stress concentrating center in the matrix. This agglomeration of TiO_2 nanoparticles probably gives rise to some microporosities and microcracks as structural defects resulting in loss of mechanical properties. Thus, concluding that the content of nanoparticle additives is of critical importance [17]. Similarly, Han Y et al., had a similar conclusion and related the results to agglomeration of particles within the matrix, which makes them stress concentration areas [18].

Salman AD et al., studied the effect of comparative study of the effect of incorporating SiO_2 nanoparticles on properties of polymethyl methacrylate denture base resin. Nanoparticles with different concentrations were incorporated and samples were tested for impact strength and surface hardness along with SEM Impact strength. The results showed adverse effects. The SEM demonstrated porous structure that comprised of mainly large sized pores. Numerous cracks were also spotted which indicated that the interaction process was not completed. The SiO_2 nanoparticles form clusters by adhesion thus acting as impurity and decreasing the strength [8].

In this study the result was found to be co-related with the study mentioned previously done by Albeladi HK et al., [16]. The reason of decrease in the impact strength on addition of SiO_2 nanoparticles in PMMA could be because of the improper dispersion of SiO_2 nanoparticles in PMMA matrix which act as impurities and unfavorably affects the reaction of monomers, leading to increased levels of unreacted monomer, which act as a plasticizer.

Kango S et al., also reviewed the surface modification of inorganic nanoparticles for development of organic-inorganic nano composites. Various methods were described to improve metal bonding between inorganic nanoparticles like TiO_2 and SiO_2 with PMMA. Silanisation of the nanoparticles is an effective pretreatment for improved bonding thus resulting in improved mechanical properties. The Silane coupling agent act as a mediator for formation of metallic bond between the metal nanoparticles and the polymer chains, which otherwise are not reactive and act only as impurities. This is detrimental on the mechanical properties of the resin especially the impact strength [19].

Limitation(s)

The present study has its limitation in use of single concentration of the nanoparticles. Also, there could have been a lack of visualisation of dispersion of nanoparticles in the resin matrix especially at the fracture site.

CONCLUSION(S)

Within the limitations of this in vitro study, it can be concluded that the mean impact strength of Trevalon was found to be the higher than the mean impact strength of DPI. Also, it was observed that the incorporation of TiO_2 and SiO_2 nanoparticles and their combined incorporation decreased the impact strength of both the commercially available heat cure denture base resin (though, statistically no significant difference was found). Finally to conclude, as the number of studies evaluating the effect of nanoparticles on the mechanical properties of heat cure acrylic resin are limited; the futuristic approach can aim on the pretreatment of the nanoparticles, SEM can be utilised for better understanding of dispersion of nanoparticles in resin matrix.

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PARTICULARS OF CONTRIBUTORS:
1. Assistant Professor, Department of Prosthodontics, Crown and Bridge, Shree Bankey Bihari Dental College, Ghaziabad, Uttar Pradesh, India.
2. Professor, Department of Prosthodontics, Crown and Bridge, School of Dental Sciences, Sharda University, Greater Noida, Uttar Pradesh, India.
3. Professor and Head, Department of Prosthodontics, Crown and Bridge, School of Dental Sciences, Sharda University, Greater Noida, Uttar Pradesh, India.
4. Assistant Professor, Department of Prosthodontics, Crown and Bridge, School of Dental Sciences, Sharda University, Greater Noida, Uttar Pradesh, India.
5. Private Practitioner, Private Clinic, New Delhi, India.
6. Private Practitioner and Consultant, Private Clinic, New Delhi, India.

NAME, ADDRESS, E-MAIL ID OF THE CORRESPONDING AUTHOR:
Megha Chopra,
H. No-1040 C, Sector 46, Faridabad, Haryana, India.
E-mail: drmeghachopra@gmail.com

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