Section A-Research paper
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COMPARATIVE EVALUATION OF EFFECT OF ADDING SILVER NANOPARTICLES AND TITANIUM DIOXIDE NANOPARTICLES ON TEAR STRENGTH, PERCENTAGE ELONGATION, WATER ABSORPTION ON MAXILLOFACIAL SILICONE ELASTOMER: AN IN-VITRO STUDY

Dr. Della S Indran, Postgraduate Student, Department of Prosthodontics Crown and Bridge, Coorg Institutes of Dental Sciences, Virajpet

Dr. Basavaraj S Salagundi, Prof and Head, Department of Prosthodontics Crown and Bridge, Coorg Institutes of Dental Sciences, Virajpet

Dr. B L Bhavana, Reader, Department of Prosthodontics Crown and Bridge, Coorg Institutes of Dental Sciences, Virajpet

Dr. Unni Pympallil, Reader, Department of Prosthodontics Crown and Bridge, Coorg Institutes of Dental Sciences, Virajpet

Dr. Ashika B K, Assistant Professor, Department of Prosthodontics Crown and Bridge, Coorg Institutes of Dental Sciences, Virajpet

Dr. Mada Shreya Chandrashekar, Postgradutae Student, Department of Prosthodontics Crown and Bridge, Coorg Institutes of Dental Sciences, Virajpet

Corresponding author: Dr. Della S Indran, Postgraduate Student, Department of Prosthodontics Crown and Bridge, Coorg Institutes of Dental Sciences, Virajpet

Objectives: To evaluate and compare the tear strength, water absorption, percentage elongation of a maxillofacial silicone elastomer with and without incorporation of nanoparticles.

Methods: 30 specimens each of circular, dumbbell and trouser shapes prepared out of maxillofacial silicone MDX- 2- 4210. Out of 30, 10 specimens were control, 10 specimens prepared with silver nanoparticles added to silicone elastomer and other 10 with titanium dioxide nanoparticles added to silicone elastomer. Circular specimens checked for water absorption by measuring the dry and wet weights. Dumbbell specimens measured for tear strength and other trouser shaped specimens checked for percentage elongation using universal testing machine.

Conclusion: A marked decrease in water absorption was noticed for silver and titanium dioxide nanoparticle groups. There was an increase in mechanical properties for nanoparticle groups over control group.

Keywords: Maxillofacial silicone elastomer, Silver nanoparticles, Titanium dioxide nanoparticles, Tear strength, Water absorption, Percentage elongation

Introduction: Maxillofacial deformities as a result of disease, injury, surgery or as a congenital malformations can negatively affect the physical and psychological well-being of an individual. Maxillofacial prostheses are devices used to restore missing parts[1], with an intention of, duplicating it in a life like way thus improving patients' normal appearance, social acceptance and psychological well-being[2].

The search for an ideal maxillofacial material started since 1500 A.D with the use of gold, silver, paper, cloth, leather[3]. Recently used materials in maxillofacial prosthetics includes acrylic resins, acrylic copolymers, polyvinyl chloride, chlorinated polyethylene, polyurethane elastomers, polyphosphazines, metals and silicones. These materials should possess ideal properties like biocompatibility, good tensile strength, hardness, tear strength, colour stability etc[4].

Silicone material are a combination of organic and inorganic compounds manufactured from silica that possess a texture that is similar to human skin[5]. They have excellent thermal and thermo-oxidative resistance, resistant against attack by oxygen, ozone and sunlight and to electromagnetic and particle radiation (UV (ultra violet), alpha, beta and gamma rays)[6]. To improve the properties like poor tear strength, colour instability, material deterioration etc, nanoparticles of copper, silver, zinc, titanium etc were added[7].

A nanoparticle (Np) or ultrafine particle is defined as the particle of matter which possess a size in between 1 and 100 nanometres (nm) diameter[8]. They exhibit unique physical, chemical and biological properties compared to their macro counterparts. They have high surface area to volume ratio and provides a tremendous driving force for diffusion, can confine their electrons and can produce quantum effects. These particles are having higher colour stability, hence commonly used in cosmetics[9].

Silver nanoparticles (AgNps) have antibacterial and bactericidal effects and hence effective in controlling biofilm production. In addition they possess antifungal, anti-inflammatory, anti-cancer, anti-angiogenic effects that favours its use in medical field in therapeutic purposes[10]. They have good magnetic properties, high thermal and electrical conductivity, surface-enhanced Raman scattering, chemical stability, catalytic activity and nonlinear optical behaviour. It is said that silver nanoparticles of size 20 to 80 nm when added to silicone elastomer, it effectively improved the colour stability by blocking the UV rays and also increases hardness, tear strength, tensile strength, percentage elongation[11].

Titanium dioxide nanoparticles (TiO_2 Nps) are white in colour and insoluble in water with high refractive index. They have versatile applications, ranging from sunscreens, to advanced devices, such as photovoltaic cells, photosensors, semiconductors etc. They seems to have environmental and biomedical applications, such as removal of pollutants by photocatalytic degradation, water purification, biosensing, and drug delivery[12]. They have improved optical, mechanical properties and quantum effects when added to silicone elastomers. It is said that when titanium dioxide nanoparticles of range 30 to 50 nm added to elastomer improved colour stability, increased tear strength, percentage elongation[13].

In the current study, silver and titanium dioxide nanoparticles were added to maxillofacial silicone elastomer, to determine the effects on mechanical properties like tear strength, percentage elongation and water absorption. The null hypothesis is to state that, there is no difference in the tear strength, percentage elongation and water absorption among the control, silver nanoparticle, titanium dioxide nanoparticle groups.

Methodology

Specimen preparation: A total of 90 specimens were prepared for the study. 30 specimens for each group. Out of which 10 were circular shaped specimens of dimension 45 mm x 1mm[44] for checking water absorption. 10 were dumbbell shaped specimens prepared according to ASTM D 142[10] for checking percentage elongation and the other 10 trouser shaped specimen for checking tear strength according to ASTM D 624[10] (Fig. 1).

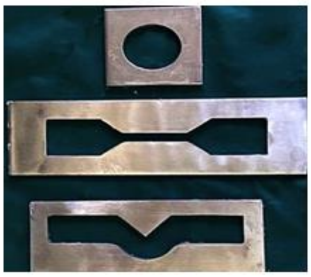


Figure no 1: metal moulds (circular, dumbbell, trouser shaped)

30 specimens were prepared from TiO_2Nps of size 30 – 50 nm mixed base (Fig. 2). Other 30 specimens were prepared from Ag Nps (30- 50 nm) mixed base (Fig. 3). Remaining 30 were control specimens prepared by adding silicone base and catalyst in ratio provided be manufacturer (Fig 4).

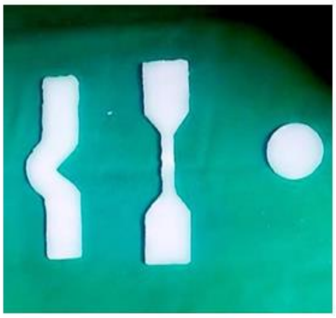


Figure no 2: Specimens prepared from base containing titanium dioxide nanoparticles

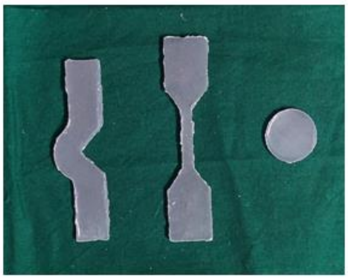


Figure no. 3: Specimens prepared from base with silver nanoparticles

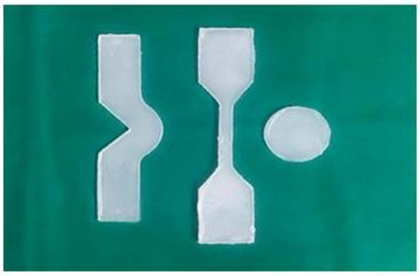


Figure no 4: Control specimens

Base and catalyst of Silastic 4 - 4210 silicone elastomer (RBS enterprises, Gurgaon) (Fig. 5) were mixed in ratio of 1: 10 in a container. When mixed in proper ratio the viscosity decreases compared to the initial viscosity of base. The metal moulds were placed above glass slab to obtain a flat surface as the moulds are open in both sides. After pouring the mix in moulds, it was covered with another glass plate to obtain a flat surface and to remove the excess materials.

Figure no 5: Silastic 4- 4210 Elastomer (base and catalyst)

The specimens were recovered after 3 days, when the specimens become rubbery form indicating the completion of polymerisation.

Base preparation: Titanium dioxide nanoparticles (TiO_2Nps) (2 wt %) in powder form were added to base of silicone elastomer (viscous jelly) using vacuum mixer to obtain a homogenous mixture. The base mixture turned to opaque white after adding the TiO_2Nps. Silver nanoparticles (Ag Nps) (2 wt. %) in powder form were added to base of silicone elastomer using ultrasonic vibrator.

Base and catalyst were mixed in the ratio provided by manufacturer. The mixed base turned greyish black in colour. In control group, nanoparticles were not added to the base.

Testing of parameters: The specimens were calculated for parameters like water absorption, percentage elongation, tear strength.

a) Water absorption: The initial weight of the control group, TiO_2Nps and Ag Nps groups were measured using a digital weighing machine. The specimens were then placed in a beaker with water for 2 days (Fig. 6) and wet weight of individual specimens and total specimens of each group was measured using digital weighing machine. After obtaining wet weight the specimens were kept in desiccator for complete drying over a period of 2 days (Fig. 7) and specimens weighed to obtain the dry weight of each specimen and total specimens of each group.

Water absorption was calculated by the following formula[45],

$$\text{Water absorption} = \frac{\text{wet weight} - \text{dry weight} \times 100}{\text{Dry weight}}$$

Figure no 6: Circular specimens in beaker

Figure no 7: Circular specimens in desiccator

b) Tear strength testing: Tear strength can be defined as the maximum force required to break the specimens, divided by the specimen thickness. The thickness of specimens were measured at the intersection of trouser leg using a vernier calliper. The specimens were then placed between the upper and lower jaws of universal testing machine and stretched at the rate of 500 ram/ rain (Fig. 8). The tear strength was then calculated by formulae,
Tear strength = Max force required to break the specimen / thickness of specimen[10].

Figure no 8: Trouser shaped specimens in universal testing machine for checking tear strength

c) Percentage elongation: The original length of dumbbell shaped specimens were measured with vernier calliper (L^o). The specimens were then placed between the jaws of universal testing machine and stretched at a rate of 500 mm/ min until it breaks (Fig. 9). The length at break was measured (L^b) using vernier caliper[31] and Percentage elongation was calculated by formulae,

Percentage elongation = L^b- L^o/ L^o x 100.

L^b= length of specimen at break.

L^o = original length.

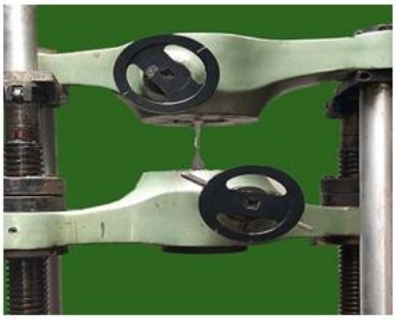


Figure no 9: Dumbbell shaped specimens in universal testing machine to check percentage elongation

Results

Water absorption: The water absorption of individual samples of the groups were calculated. Shapiro Wilk test was performed on the water absorption of 3 groups (table 1) followed by parametric test, namely one-way ANOVA followed by post hoc Tukey's test.

It was noted that the mean water absorption of silver nanoparticle group showed lowest value of (3.31600 ± 0.002867) %, followed by titanium dioxide nanoparticle group of value (4.70420 ± 0.023342) % and control group value of (8.34200 ± 0.005578)%. (Table 2) (Graph 1). The one-way ANOVA was performed to compare the water absorption in the Control groups, silver nanoparticle group, and Titanium Dioxide nanoparticles group (Table 3). The one-way ANOVA revealed that there was a statistical difference in the water absorption between at least two groups (F (2,27) = 345968.005, p value=.000). Tukey's post hoc test was done for multiple comparisons (table 4) and it was found that the mean value of water absorption was significantly different between

- Control group and Silver nanoparticle group (p=.000, 95% C.I = (5.01053, 5.04147),
- Control group and Titanium Dioxide Nano particle group (p=.000, 95% C.I = 3.62233, 3.65327)

- Silver nano particle group and Titanium Dioxide Nano particle group (p=.000, 95% C.I = -1.40367, -1.37273)

The null hypothesis is rejected since the p value (.000) is less than .005 and alternate hypothesis accepted, that is there was a statistical difference in values of water absorption in control group, titanium dioxide and silver nanoparticle group. Water absorption was highest for control group, followed by titanium dioxide nanoparticle group. The least value of water absorption was showed by silver nanoparticle group.

Table 1: Value of significance calculated by Shapiro Wilk test for water absorption

Parameter		Shapiro-Wilk		
		Statistic	df	Sig.
Water absorption	Control group	.919	10	.347
	Silver nano particles	.891	10	.172
	Titanium Dioxide nano particles	.958	10	.765

Table 2: Descriptive statistics; mean, minimum and maximum values of water absorption (%) for each group.

Parameter	Group	N	Minimum	Maximum	Mean	Std. Deviation
Water absorption	Control group	10	8.336	8.352	8.34200	.005578
	Silver nano particles	10	3.313	3.321	3.31600	.002867
	Titanium Dioxide nano particles	10	4.665	4.735	4.70420	.023342

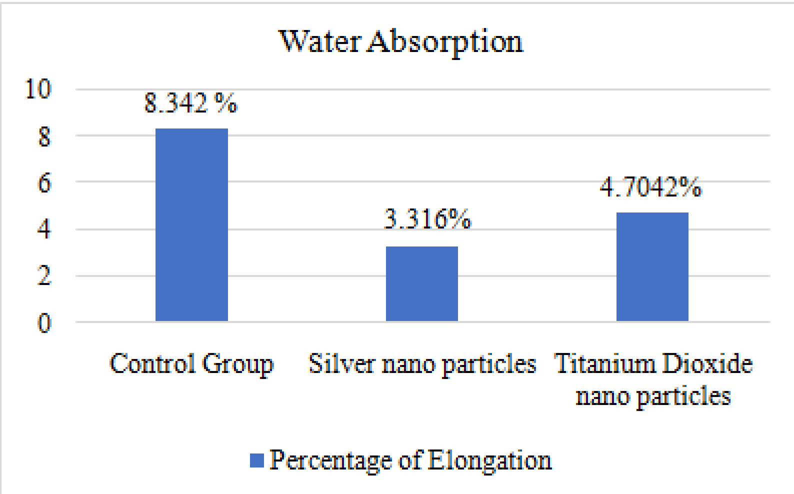
Table 3: Analytical statistics of water absorption between and within groups

Parameter		Sum of Squares	df	Mean Square	F	Sig.
Percentage Elongation	Between Groups	134.738	2	67.369	345968.005	.000
	Within Groups	.005	27	.000		
	Total	134.743	29			

Table 4: Tukey's Post Hoc test for comparing water absorption between groups

Parameter	(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Water absorption	Control group	Ag Nps	5.026000	.006241	.000	5.01053	5.04147
		TiO_2Nps	3.637800	.006241	.000	3.62233	3.65327
	Ag Nps	Control group	-5.026000	.006241	.000	-5.04147	-5.01053
		TiO_2Nps	-1.388200	.006241	.000	-1.40367	-1.37273
	TiO_2Nps	Control group	-3.637800	.006241	.000	-3.65327	-3.62233
		Ag Nps	1.388200	.006241	.000	1.37273	1.40367
The mean difference is significant at the 0.05 level.							

Graph 1: Mean values of water absorption (%) of control, titanium dioxide and silver nanoparticle groups



Tear strength: The tear strength of each specimen of the three groups were calculated. Shapiro Wilk test was performed on the tear strength of the 3 groups (table 5) followed by non-parametric test i.e., Kruskal Wallis test.

It was noted that the mean tear strength of TiO_2Nps showed highest value of (17.28 ± 4.16 N/mm), followed by silver nanoparticle group of value (14.7 ± 3.39) N/mm and control group value of (14.53 ± 4.41)N/mm Table 6 (Graph 2). Statistical analysis was done between the groups using Kruskal Wallis H test, there was no statistically significant difference in the tear strength between the three groups (Table 7). Since the p value is greater than 0.05, the null hypothesis is accepted, as there is no statistical difference in tear strength between control, silver and titanium dioxide nanoparticle groups.

Table 5: Value of significance calculated by Shapiro-Wilk test for tear strength

Parameter		Shapiro-Wilk		
		Statistic	df	Sig.
Tear strength	Control group	.857	10	.069
	Silver nano particles	.939	10	.542
	Titanium Dioxide nano particles	.793	10	.012

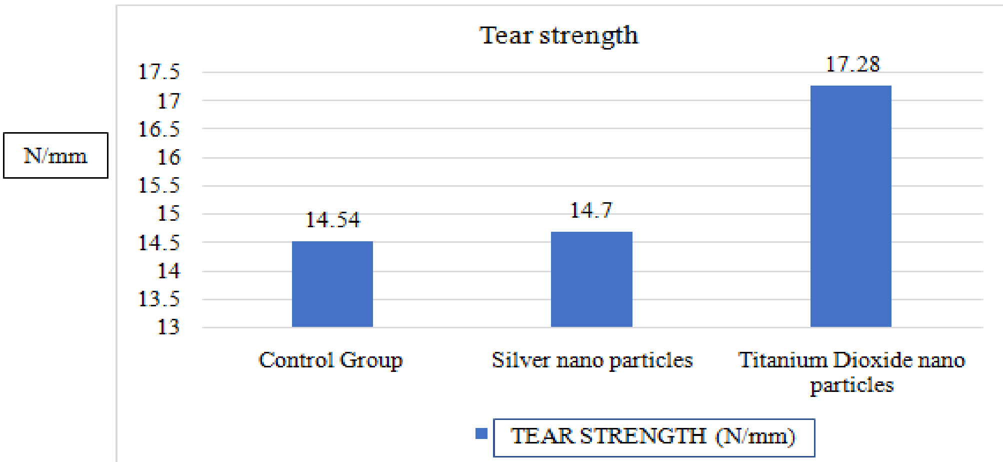
Table 6: Descriptive statistics; mean, minimum and maximum values of tear strength for each group

Parameter	Group	N	Minimum	Maximum	Mean	Std. Deviation
Tearing strength	Control group	10	9.50	20.31	14.5360	4.41516
	Silver nano particles	10	9.24	19.01	14.6950	3.39196
	Titanium Dioxide nano particles	10	11.30	21.80	17.2770	4.16977

Table 7: Kruskal Wallis H statistical analysis of tear strength for groups

Ranks							
Parameter		Group	N	Mean Rank	Kruskal Wallis H	df	Asymp. Sig
Tear strength	Values	Control group	10	13.55	3.765	2	.152
		Silver Nano Particles	10	13.05			
		Titanium Dioxide Nano Particles	10	19.90			
		Total	30				

Graph 2: Mean values of tear strength (N/mm) in control, Ag Nps, TiO_2 Nps groups



Percentage elongation: The percentage elongation of individual specimens of each group were calculated.

Shapiro Wilk test was performed on the percentage elongation of the 3 groups (table 8), followed by parametric test i.e., ANOVA and Post Hoc Tukey's test.

Titanium dioxide nanoparticle group showed highest mean value of (42.03 ± 2.16) % followed by silver nanoparticle group of mean value (38.9 ± 3.67) %. Least value of (33.14 ± 1.98) %, shown by control group (Table 9) (Graph 3).

The one-way ANOVA revealed that there was a statistical difference in the percentage elongation between at least two groups (F=27.63, p value=.000) (Table 10).

Tukey's HSD test for multiple comparisons found that the mean value of percentage of elongation was significantly different between Control group and silver nano particle group (p=.000, 95% C. I = (-8.7470, -2.7350), Control group and Titanium Dioxide Nano particle group (p=.000, 95% C. I = -11.8930, -5.8810), and silver nano particle group and Titanium Dioxide Nano particle group (p=.039, 95% C. I = --6.1520, -.1400) (Table 11)

Table 8: Value of significance for percentage elongation of control, silver and titanium dioxide nanoparticle groups

Parameter		Shapiro-Wilk		
		Statistic	df	Sig.
Percentage Elongation	Control group	.932	10	.473
	Silver nano particles	.844	10	.050
	Titanium Dioxide nano particles	.936	10	.510

Table 9: Descriptive Statistics; mean, minimum and maximum values of percentage elongation for control, silver and titanium dioxide nanoparticles

Parameter	Group	N	Minimum	Maximum	Mean	Std. Deviation
Percentage of Elongation	Control group	10	30.52	36.01	33.1390	1.98315
	Silver nano particles	10	35.08	45.01	38.8800	3.66621
	Titanium Dioxide nano particles	10	39.21	46.00	42.0260	2.16203

Table 10: Analytical statistics of percentage elongation between and within groups

Parameter		Sum of Squares	df	Mean Square	F	Sig.
Percentage of Elongation	Between Groups	406.117	2	203.059	27.629	.000
	Within Groups	198.436	27	7.349		
	Total	604.553	29			

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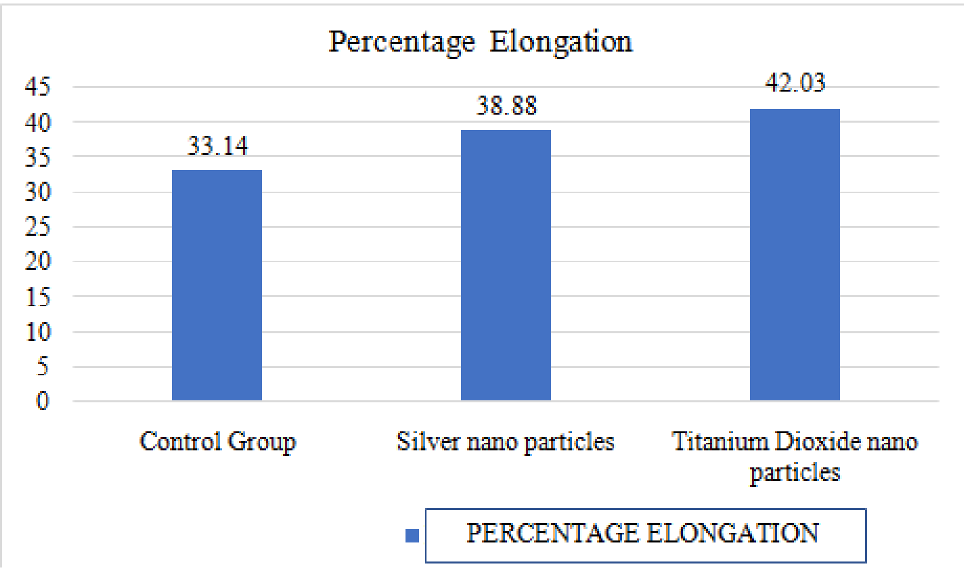
Table 11: Tukey's Post Hoc test for comparing percentage elongation between groups

Parameter	(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Percentage Elongation	Control group	Ag Nps	-5.74100*	1.21239	.000	-8.7470	-2.7350
		TiO_2Nps	-8.88700*	1.21239	.000	-11.8930	-5.8810
	Ag Nps	Control group	5.74100*	1.21239	.000	2.7350	8.7470
		TiO_2Nps	-3.14600*	1.21239	.039	-6.1520	-.1400
	TiO_2Nps	Control group	8.88700*	1.21239	.000	5.8810	11.8930
		Ag Nps	3.14600*	1.21239	.039	.1400	6.1520
The mean difference is significant at the 0.05 level.							

Graph 3: Mean values of percentage elongation (%) in control, Ag Nps, TiO_2Nps groups



Since the p value (p = .000) is less than 0.05, the null hypothesis of percentage elongation is rejected and alternate hypothesis accepted, that is there is statistically difference in the percentage elongation in control, titanium dioxide and silver nanoparticle groups, with highest for titanium dioxide nanoparticle group, followed by silver nanoparticle and control groups.

Discussion: Even though silicone elastomers, chemically known as polydimethylsiloxane (PDMS) are commonly used for maxillofacial prosthesis. They have some undesirable properties like low tear and tensile strength, insufficient elasticity and degradation of physical and colour properties. The mechanical properties of silicone elastomers are dependent on factors like molecular weight of polymer chains, incorporation of filler and cross-linking density. The addition of fillers helps to attain a certain degree of reinforcement that leads to improvement on the mechanical properties. The reinforcement depends on the polymer properties, filler characteristics like particle size, surface area, structure and filler loading amount and its processing[10]. Nanoparticles are characterized by its small size, large specific area, active function, and strong interfacial interaction with the organic polymer.

The tear strength of silicone elastomer is very important factor because the margins of facial prosthesis are usually thin as it has to merge with the surrounding skin which are usually glued with the help of medical adhesives and when the patient tries to remove the prosthesis with varying degree of forces, chances are there for tearing[46]. So the material should have sufficient tear strength to resist it. In the current study, compared to control group (14.54 N/mm), tear strength values increased for TiO_2 Nps (17.28 N/mm) and Ag Nps (14.70 N/mm) groups (Table no.5). Zayed et al, reported an improvement in tear strength when the nano SiO_2 was added at a concentration of 3 %[31] to silicone elastomer. Duhuha A et al, reported an increase in properties like tear strength, percentage elongation, tensile strength, hardness when 0.25 and 0.5 w/w % of TiO_2Nps were added to cosmesil M511 elastomers[47]. Somnahalli reported that there was a marked decrease in hardness at a concentration of 20 ppm of silver nanoparticles in silicone elastomer, but there were no significant changes in tear strength of the test group from control group. It is important to choose the proper concentration of nanoparticles, to avoid the particles to agglomerate and can act as areas of stress concentrations when the external forces are applied, thereby decreasing the mechanical strength of the material[10]. An increased tear strength for nanoparticle groups can be explained by the ability of nanoparticles to redistribute the energy at the end of growing cracks, when tearing force starts to take place suggesting the nanoparticles has participated in cross linking during polymerization reaction of elastomer[40].

An ideal facial prostheses should possess a certain degree of flexibility, which not only avoid the prostheses damage but also gives it a more natural like appearance. In case if the prosthesis don't exhibit much elongation during chewing, laughing and talking etc, it cause the remodelling of facial structures such as eyes, nose, mouth and creates unesthetic appearance. Thus, an ideal facial prostheses should own a certain degree of flexibility. In the current study, there was an increase in percentage elongation for TiO_2Nps group (42.03%) and Ag Nps group (38.8%) in comparison to control group(Table no 8). Han et al. reported that the incorporation of nano particles of Ti, Zn, or Ce at a concentration of 2 to 2.5% by weight into A 2186 silicone elastomer; improved the mechanical properties [40]. Radey et al, reported an increase in tensile, tear strength and percentage elongation in MED- 4210 silicone elastomer on addition of 2.5 % of TiO_2 nanoparticles after and before extra oral aging[48]. However, in a study by Pinar Cevik, showed addition of TiO_2Nps to silicone elastomer at a concentration of 10 % decreased the percentage elongation[24].Increased percentage elongation can be due to strong chemical bond between filler and polymers that allows the polymeric chain to uncoil and slide over nanoparticles when the force is applied.

Another desirable property of silicone elastomer is to have decreased water absorption. The prosthesis tends to absorb saliva, sweat and water, which can decrease the life of prosthesis, which leads to color deterioration and adds to weight of prosthesis. López-Zaldívar et al, correlated the concept of adding filler into the rubber, which was prone to cause greater porosity in silicone and more water was drawn inside. Hence, lesser the porosity, lesser the water absorption uptake[49]. Monika et al, reported a decrease in water absorption for MultisilEpithetics silicone compared to Biomed silicone, when intrinsic pigments were added. Thus the type of silicone also affects the rate of water absorption. The by products produced in condensation silicones during polymerization later leaves creating porosities which creates space for water molecules[50] . In the current study a decreased water absorption rate was seen for nanoparticle groups, while control group had highest range of 8.34200 % (Table 3). A decrease in water absorption was observed for groups added with nanoparticles, might be because of decrease in space for the water molecules to sweep in, which is taken up the fillers.

There were some limitations in the current study, a single particle size of nanoparticles was used, further studies should be done using different available types of RTV silicones and different sizes, concentrations and types of nanoparticles. Even though vacuum mixer was used for mixing nanoparticle with silicone base, air bubbles was noticed which can affect tear strength and percentage elongation and water absorption. When nanoparticles of silver and titanium dioxide were added to base, there was a change in color, so when fabricating prosthesis there should be more emphasis in intrinsic as well as extrinsic staining. Further studies should be done to find the silver and titanium dioxide nanoparticle concentrations that can improve both the mechanical properties without much changes in color.

Conclusion: Within the limitations of this study, the following conclusions were drawn,

- A marked decrease in water absorption was noticed for silver and titanium dioxide nanoparticle groups.
- There was an increase in mechanical properties for nanoparticle groups over control group.
- An increase in tear strength was seen for groups where nanoparticles were added compared to control group.
- Even though, there was an increase in tear strength for nanoparticle groups the results were not statistically significant.
- An increase in percentage elongation was seen for nanoparticle group, compared to control group.
- Titanium dioxide nanoparticle exhibited highest percentage elongation value followed by group with silver nanoparticles.

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