The effect of thermocycling and different pH of artificial saliva on the impact and transverse strength of heat cure resin reinforced with silanated ZrO_2 nano-fillers.

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ABSTRACT

Background: The aim of this study was to evaluate the effect of thermo cycling and different pH of artificial saliva (neutral, acidic, basic) on impact and transverse strength of heat cure acrylic resin reinforced of with 5% silanated ZrO_2 nano fillers.

Materials and methods: 120 samples were prepared, 60 samples for impact strength test and another 60 samples for transverse strength test, for each test, samples were divided into two major groups (before and after thermo cycling), then each of these major groups were further subdivided into 3 subgroups according to the pH of prepared artificial saliva (neutral, acidic, basic). Charpy impact device was used for impact strength test and Flexural device was used for transverse strength test.

Result: There was a non-significant difference between the two major groups (before and after thermo cycling). Also results showed that there was a non-significant difference between the subgroups for each major group in reference to pH of artificial saliva. These results were found in both impact and transverse strength.

Conclusion Transverse strength and impact strength of heat cure acrylic resin reinforced with silanated ZrO_2 nanofillers was not affected by thermocycling and different PH of artificial saliva suggesting improvement in the mechanical properties.

Key word: Artificial saliva, Thermo cycling, Impact strength, Transverse strength, Nanocomposite. (J Bagh Coll Dentistry 2013; 25(Special Issue 1):12-17).

INTRODUCTION

Saliva in the mouth participates in the activity of speaking, swallowing, chewing, mucosal protection against penetration of various substances, regulation of pH, taste sensitivity and lubrication of tissue.(1)

Due to the impossibility of duplicating the properties of human saliva as a result of the inconsistent and unstable nature of natural saliva, a development of artificial saliva is essential for well justified and controlled experiments which were reported as early as since 1931.(2)

During use, Prostheses are submitted to various clinical situations like different temperature, alteration in PH, salivary flow, and the denture base material should withstand all these condition without change. Acrylic resin was the material which were used in complete dentures, so this material has been subjected to different alterations including several types of reinforcement to improve their properties.(3)

Because denture base materials had a high incidence of fractures and need to a constant repairs. Different methods are used to reinforce these materials. (4) So an improvement in properties of polymers can be achieved with the addition of nano-sized fillers.

The purpose of using nano fillers is to achieve higher abrasion resistance, improved esthetic, physical and mechanical properties of dental materials.

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Increase several properties of the material like mechanical strength can be obtained by blending polymer material with different organic fillers (5). The size, shape, concentration, type of nano-particals, and interaction with polymer matrix affect the properties of polymer nano composites. (6)

The ability of a denture base material to be strong to withstand functional and masticotary forces can be reflected by the flexural and impact strength of the material.(7) In denture wearing patients saliva has an important role in providing comfort to the mucosa under denture base, maintenance of oral health and denture retention.(8)

The normal range of salivary pH is between 6.2-7.4, so any change in the pH of saliva would be due to different types of food ingested. So it may be more acidic or more basic which may have an erosion effect on denture base materials. (9)

During service in oral cavity denture resins subjected to a variation in temperatures by ingestion of cold and hot foods which causes a thermal stresses in denture base materials, this thermal stress will have an effect on the water sorption of denture base materials because water sorption is a process that depended on temperature. (10)

Although reports on the effect of thermo cycling on impact and transverse strength are limited but it is important to be investigate. (10)

The aim of this in vitro study was to evaluate the effect of different pH of artificial saliva and

thermo cycling on impact and transverse strength of heat cure acrylic resin reinforced with 5% of silanated ZrO_2nano fillers.

MATERIALS AND METHOD

120 samples were prepared for this study, these samples were divided into 60 samples for impact strength test and 60 samples for transverse strength test. For each test the samples were divided into two major groups: before thermo cycling contains 30 samples and after thermo cycling which also contain 30 samples.

Each major group were subdivided into three subgroups contains 10 samples for each and stored in artificial saliva with different pH (neutral, acidic and basic).

The materials used in this study was: heat cured acrylic denture resin (superacryle plus,Spofa Dental; Europe), dental stone (type III, Zermack; Italy), distilled water (Iraq) , separating medium (Iso dent,Spofa Dental; Europe), Silanated Zirconium oxid (ZrO_2) nano fillers (Sigma, Aldrich; Germany).

Artificial saliva preparation

Artificial saliva was prepared in different pH (acidic: 5.7-Nuetral: 7-Basic: 8.3) by using pH meter device (WTW, Germany). Preparation of saliva had been done by using an electrolytes composition similar to that of human saliva according to (Pusz et al, 2010) and as illustrated in table 1.

Table 1: The chemical constituents of the artificial saliva used in the study

Material	Conc.	Material	Conc.
NaCL	0.700 g/l	**Na_2HPO_4**	0.260 g/l
KSCN	0.330 g/l	**KCL**	1.200 g/l
KH_2PO_4	0.200 g/l	**$NaHCO_3$**	1.500 g/l

Buffer solution from KH_2PO_4 and Na_2HPO_4 was prepared by dissolving each one in 1 liter of de-ionized distilled water. [12]

Then basic saliva was prepared by taking 500 ml of Na_2HPO_4 and added KH_2PO_4 gradually to it until reached the exact pH, after that added the other salts to saliva and complete the volume to 1 liter by de-ionized distilled water. [12]

On the other hand, neutral and acid saliva were prepared by taking 500 ml of KH_2PO_4 and added Na_2HPO_4 gradually to it until reached the exact pH. Then adding the other salts just like in basic saliva. For neutral saliva greater amount of Na_2HPO_4 was added to reach the exact pH, To control the measurement, a freshly artificial saliva was prepared every day because the Ph of saliva may change within 48h. [12]

Impact strength test

A mould of stone in dental flask was prepared by using bar shaped metal pattern with dimensions of (80mm, 10mm and 4mm) length, width and thickness respectively figure (1). After that the mould was coated by separating medium and left to dry for 15 minutes, then packing with acrylic resin. Preparation of acrylic was done by the following:

The P/L ratio for acrylic was 2.5g:1g according to the manufacturer's instructions, the amount of acrylic powder was (11.400 g) weighted by using electronic balance (Sartorius BP 30155, Germany) of (0.0001g) accuracy, the amount of monomer was (6ml=4.80g) to which the 5% silanated (0.600g)of ZrO_2 nano fillers was added. The ZrO_2 filler was mixed with the monomer by ultra sonication for 3 minutes using a probe sonication apparatus (Saniprep-150, England, 120w,60khz), the silanated nanofiller were well dispersed into the monomer[13], then the acrylic was mixed and packed in the dough stage in the mould and cured. The curing process was done according to ADA specification no 12, 1999 by heating the dental flask in water bath at 74C° for 1.5 hours, then increased the temperature to boiling for 30 minutes, after that cool the flask for 30 minutes at room temperature, then for 15 minutes under tap water. After flask was cold, the acrylic samples were removed, finished and polished. Half of samples (30) were stored in artificial saliva (neutral, acidic and basic) for 16 hours and then in distilled water for 8 hours at 37C° in an incubator for 30 days. [12, 14]

The other half of samples (30) were subjected to thermo cycling by (1000 cycle) then stored in artificial saliva (neutral, acidic and basic) for 30 days like above. The thermo cycling was done by using machine (Haack, Germany) that subject the samples in bath of hot distilled water (55c°±2c°) for 30 seconds then in other bath of cold distilled water (5c°±2c°) for 30 seconds, this cycle was repeated until complete 1000 cycles.[12] The samples were tested with charpy type impact testing device (Impact tester, N. 43-1, INC. USA) of 2 joules capacity, impact strength was calculated by this formula:

Impact strength = $\frac{E}{bd} \times 10^3$ in KJ/m² by Anusavice [15] Where:

E= impact energy, b= width of sample, d= thickness of sample.

Transverse strength test

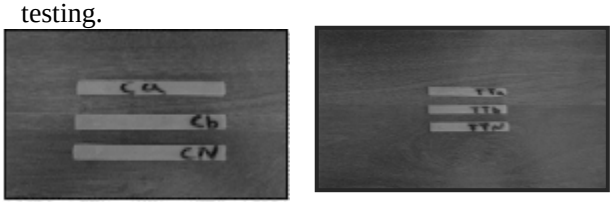
The procedure for preparation of the transverse strength samples were done just like the procedure for preparation of impact strength samples, the dimensions of transverse test

samples were (65mm, 10mm and 2.5mm) length, width and thickness, respectively figure (1). The samples were tested by Flexural measuring device (Jian, Qiao, Japan). The full scale load was (7.5 KN), the distance between 2 parallel supports was (50mm) and transverse strength was calculated by this formula:

Transverse strength= $\frac{3P.I}{2b.d^2}$ in N/mm^2 (Anusavice, 2008). [15] Where:

P= peak load, I= distance between 2 support, b= width of sample, d= thickness of sample.

The samples shown in Figure 1, 2 for impact and transverse strength test before and after testing.



(A) (B)

Figure 1: A- Samples for impact strength before testing. B- Samples for transverse strength before testing.

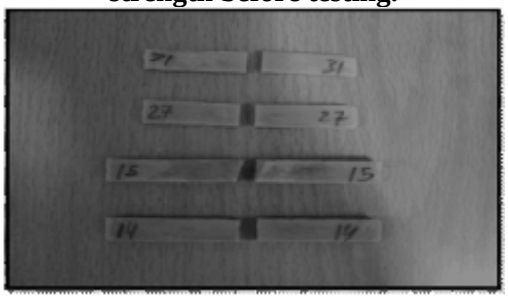


Figure 2: Samples for transverse and impact strength after testing.

RESULTS

Impact strength test: (before thermo cycling)

Table (2) and figure (3) shows the mean distributions, standard deviation, maximum value and minimum value for samples before thermo cycling in neutral, acidic and basic artificial saliva, the maximum mean value was found in basic saliva (8.92 KJ/m²) and the minimum mean value was found in acidic saliva (8.02 KJ/m²).

Table 2: Mean, SD, Max and Min for impact strength in KJ/m² for samples before thermo cycling.

	Sample groups		
	Neutral	**Acid**	**Base**
Mean	8.553	8.545	8.612
SD	0.229495	0.280802	0.24939
Max	8.9	8.9	8.92
Min	8.25	8.02	8.26

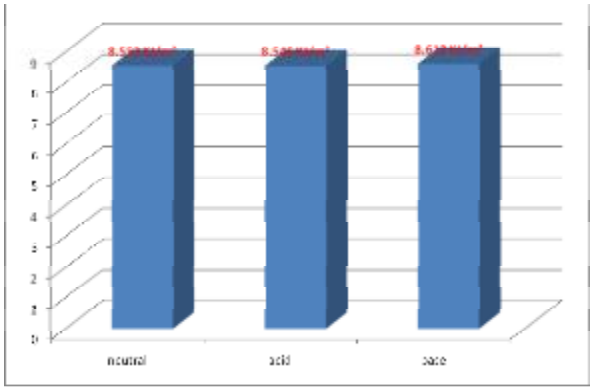


Figure 3: Bar chart for impact strength means in KJ/m² for samples before thermo cycling.

In table (3) one way ANOVA analysis of variance shows a non-significant difference between the three groups at different pH of artificial saliva, the P-value was (0.814).

Table 3: ANOVA (F-test) analysis between neutral, acidic and basic saliva for samples before thermo cycling.

	F-test	**P-value**	**Sig**
ANOVA	0.207	0.814	NS

Impact strength test: (after thermo cycling).

Table (4) and figure (4) show the mean distribution, standard deviation, maximum value and minimum value for samples after thermo cycling in neutral, acidic and basic saliva. the maximum mean value was found in basic saliva (8.97 KJ/m²) also the minimum mean value was found in basic saliva (8.12 KJ/m²).

Table 4: Means, SD, Max and Min for impact strength in KJ/m² for samples after thermo cycling.

	Sample groups		
	Neutral	**Acid**	**Base**
Mean	8.550	8.599	8.583
SD	0.206667	0.170258	0.284138
Max	8.82	8.85	8.97
Min	8.25	8.38	8.12

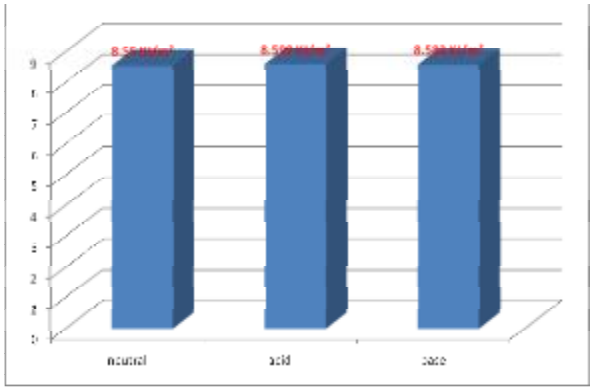


Figure 4: Bar chart for impact strength means in KJ/m² for samples after thermo cycling.

In table (5) one way ANOVA analysis of variance shows non-significant differences between the three groups of different pH of saliva, the P-value was (0.885).

Table 5: ANOVA (F-test) analysis between neutral, acidic and basic saliva for samples after thermo cycling

ANOVA	F-test	P-value	Sig
	0.123	0.885	NS

In table (6) and figure (5) t-test between samples before and after thermo cycling shows a non-significant difference for the three groups of different pH of saliva. P-value for neutral groups was (0.972), for an acidic group was (0.635) and for basic groups was (0.814).

Table 6: t-test between samples before and after thermo cycling for impact strength test

Groups	t-test	P-value	Sig
Neutral	0.036	0.972	NS
Acid	0.491	0.635	NS
Base	0.242	0.814	NS

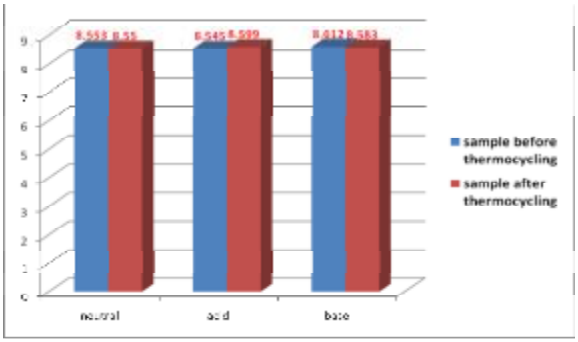


Figure 5: Bar chart for comparison between means in KJ/m² for impact strength samples before and after thermo cycling.

Transverse strength test (before thermo cycling)

Table (7) and figure (6) shows the mean distribution, standard deviation, maximum value and minimum value for samples before thermo cycling in neutral, acidic and basic artificial saliva, the maximum mean value was found in basic saliva (588.1 N/mm^2) and the minimum mean value was found in acidic saliva (511.8 N/mm^2).

Table 7: Mean, SD, Max and Min for transverse strength in N/mm^2 for samples before thermo cycling

	Sample groups		
	Neutral	**Acid**	**Base**
Mean	556.03	556.18	554.56
SD	19.90612	26.205	20.3732
Max	582.1	583.1	588.1
Min	521.8	511.8	527.2

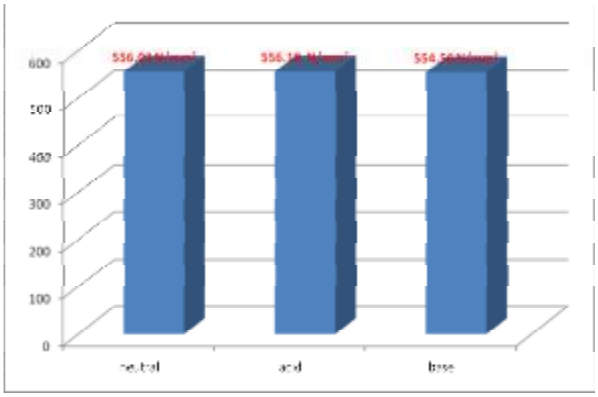


Figure 6: Bar chart for transverse strength means in N/mm^2 for samples before thermo cycling.

In table (8) one way ANOVA analysis of variance shows a non-significant difference between the three groups at different pH of saliva, the P-value was (0.984).

Table 8: ANOVA (F-test) analysis between neutral, acidic and basic saliva for samples before thermo cycling

ANOVA	F-test	P-value	Sig
	0.016	0.984	NS

Transverse strength test (after thermo cycling)

Table (9) and figure (7) shows the mean distribution, standard deviation, maximum value and minimum value for samples after thermo cycling in neutral, acidic and basic artificial saliva, the maximum mean value was found in basic saliva (588.2 N/mm^2) and the minimum mean value was found in neutral saliva (525.2 N/mm^2).

Table 9: Means, SD, Max and Min for transverse strength in N/mm^2 for samples after thermo cycling

	Sample groups		
	Neutral	**Acid**	**Base**
Mean	555.95	559.42	557.85
SD	19.59077	18.66648	19.12551
Max	583.3	585.7	588.2
Min	525.2	528.6	526.3

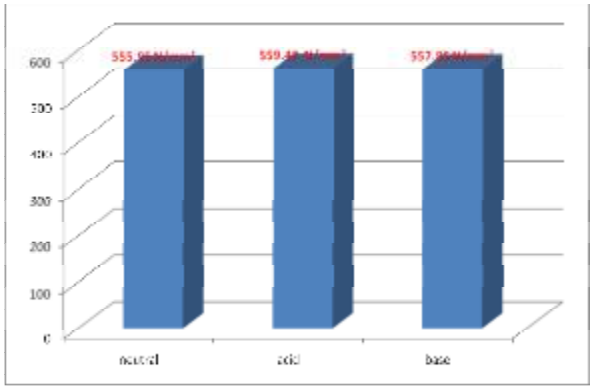


Figure 7: Bar chart for transverse strength means in N/mm^2 for samples after thermo cycling.

In table (10) one way ANOVA analysis of variance shows a non-significant difference between the three groups of different pH of saliva, the P-value was (0.921).

Table 10: ANOVA (F-test) analysis between neutral, acidic and basic saliva for samples after thermo cycling

	F-test	**P-value**	**Sig**
ANOVA	0.082	0.921	NS

In table (11) and figure (8), t-test between samples before and after thermo cycling shows a non-significant differences for the three groups of different pH of saliva, the p-value for the neutral groups was (0.992), for acidic groups was (0.739) and for basic groups was (0.705).

Table 11: t-test between samples before and after thermo cycling for transverse strength test

Groups	**t-test**	**p-value**	**Sig**
Neutral	0.011	0.992	NS
Acid	0.344	0.739	NS
Base	0.390	0.705	NS

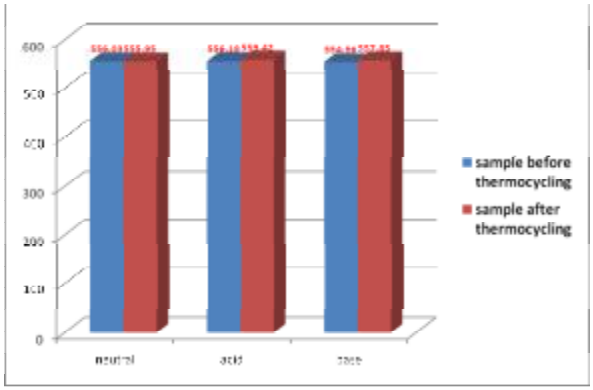


Figure 8: Bar chart for comparison between means in N/mm^2 for transverse strength samples before and after thermo cycling.

DISCUSSION

In this study addition of 5% silanated ZrO_2 nano-fillers to the heat cure acrylic resin were done in order to prepare reinforced acrylic material, It was found that there is no effect of thermo cycling process and different pH of artificial saliva on both impact and transverse strength. This can be explained by that Silane coupling agent has an affinity to the polymer matrix because silane contain vinyl group and alkoxy groups that can react with organic matrix and substrate. So this will form covalent bonds between both matrices. So the use of silane coupling agent was to coat the fillers and this will led to produced covalent bonded polymer chain because coated fillers has an affinity to polymer matrix.[16]

This bond can prevent water from penetrating filler resin interface by inhibition of fluid leaching and improved the adhesion between polymer matrix and enforcing filler, so this will improve the mechanical properties of the acrylic resin materials. [15,17]

Also due to the use of very fine size nano-fillers with concentration between (5 %) enable them to enter and fill the space between the chains of polymer, so it restrict the motion of chains and lead to increase rigidity and this will increase the transverse strength. [18]

The addition of 5% of silanated ZrO_2nano-fillers to the acrylic resin showed a higher impact and transverse strength by (Safi I.N, 2011). which made the acrylic material more strong by the addition of Zirconium.[19]

Water sorption could decrease the strength of the polymer because water molecules acts as a plasticizer and weak acids that causes mobility of polymer chains. [20, 21]

So the use of silanated fillers made the mixture more homogenous and decreases the mobility of polymer chains and this make PMMA stronger

and leads to increase the transverse and impact strength. [3, 22, 23]

On the other hand, the highly filled material by silanated molecules forms a layer to protect the surface of filler particles from the erosional effect of acids and bases. So the storage in the acidic and basic environments had no significant effects on the transverse and impact properties of the reinforced resin bases. [21]

As conclusion, the addition of 5% of silanated ZrO_2 nano- fillers makes the acrylic resistant to the effect of change in the PH of artificial saliva and the thermocycling process as shown by the non-significant effect on both transverse and impact strength.

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