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RESEARCH ARTICLE

INFLUENCE OF HEAT TREATMENT AND IN-VITRO AGING ON MECHANICAL PROPERTIES AND SURFACE ROUGHNESS OF COLD ISOSTATIC PRESSED ZIRCONIA - AN INVITRO STUDY

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Abstract

Purpose: Mechanical and surface characteristics of the zirconia dental ceramic's may be adversely impacted by temperature changes during the veneering firing cycles. A potential synergistic failure could be caused by ageing when exposed to the oral environment and heat treatment. The current research aimed to determine the influence of heat treatment and in-vitro ageing on mechanical properties and surface roughness of cold isostatic pressed zirconia.

Materials and Methods:15 bar shaped specimens with dimensions 28mm×4mm×2mm are milled from two types of partially sintered Y-TZP ceramic blanks (IPS e.max ZirCAD (IZ),ZENO Zr (WZ)). Out of 15 samples; five are used as control (C group), five are heat treated (H group) and five are heat treated and subsequently aged (HA group). Flexural strength and shear bond strength are measured using universal testing machine and surface roughness is measured using proliferometer.

Results: Heat treatment on the ivoclar and weiland samples led to an increase in surface roughness and a loss in mechanical properties (flexural strength and shear bond strength), although the differences are not statistically significant ($p>0.05$). Yet, the combined ageing and heat treatment created a synergistic effect on surface roughness and mechanical characteristics. ($p<0.001$). More variations were seen in the weiland samples.

Conclusion: Firing and ageing processes may have an impact on zirconia ceramics' clinical performance, which raises the risk of failure.

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Introduction:-

Growing aesthetic standards and concerns about the bio-compatibility of dental alloys have raised the popularity of ceramic restorations in recent years. Although alumina prostheses perform sufficiently both biological, and aesthetic aspects, it is apparent that they possess a fracture risk. Many technical failures have been attributed decreased mechanical strength, which has led connector region fractures and veneering material chipping[1]. Zirconia (ZrO_2) ceramics, particularly yttria (Y_2O_3)-stabilized tetragonal zirconia polycrystal (Y-TZP), were proposed as an alternative material for all-ceramic dental restorations as a result of these flaws in the mechanical properties of alumina ceramics.

Three different shapes can be seen in pure zirconia. Zirconia has a characteristic known as polymorphism, which refers to the spatial arrangement of the atoms being characterised by various crystallographic structures. Its three crystal phases, Monoclinic, Tetragonal, and Cubic, are distinct from one another in terms of specific geometrical and dimensional characteristics. Zirconia can undergo a tetragonal-to-monoclinic phase change when subjected to stress, heat, and surface treatments

Zirconia restorations that are particularly aesthetically pleasing have a zirconia core that is veneered with a feldspathic porcelain layer and fired at temperatures between 750 and 900 degrees Celsius before being cooled. The restoration process should go through a minimum of 2 to 5 firing cycles in order to achieve an aesthetic restoration.

The residual compressive stresses from milling and other processing steps, such as grinding and sandblasting, that are released during the heat treatment, as well as the tetragonal to monoclinic (t→m) transformation and the alteration of the grain size that takes place during the firing cycles of the veneering process, have all been linked to the reduced mechanical properties of zirconia ceramics after heat treatment.

Oral prostheses, in contrast to other prostheses in the body, are constantly exposed to moisture with varying pH and temperature[1,2].The material's mechanical properties may deteriorate considerably more in the oral environment, lowering its strength. As a result, a likely irreversible early failure could be caused by the synergistic effects of ageing and heat treatment. This is referred to as low temperature deterioration or ageing.Low-temperature degradation is responsible for increased surface roughening, grain push-out, increased wear, loss of fracture strength, and decreased flexural strength, hardness, which may lead to deterioration. Surface roughness produced through low-temperature degradation is responsible for bacterial plaque accumulation that affects the long-term success of restorations.

Therefore, the present study was done to evaluate the influence of both heat treatment and in-vitro aging on mechanical properties and surface roughness of cold isostatic pressed zirconia restorations.

Materials And Methodology:-

Using CAD/CAM technology, fifteen bar-shaped specimens (28 mm x 4 mm x 2 mm) were machined from two zirconia blocks (Ivoclar IPS e.max ZIRCAD and Wieland ZENO Zr (Fig: 1,2)) and sintered to full density. Five of the fifteen are untreated (C), five are heat-treated (HT), and five are simultaneously heat-treated and aged in steam at 134°C for five hours (HA). According to the manufacturer's recommendations, the heat treatment employed corresponds to four fire cycles of the porcelain veneering.

Heat treatment of the samples

Ten samples from each zirconia group (5 in H group & 5 in HA group) are layered using VITA LUMEX (Fig.) veneering material. The veneering procedure is done using the manual layering technique(Fig.7) . The ceramic layering is done on one side of the blank.

Finally the samples are finished to achieve a uniform thickness of 2mm of veneered porcelain. The total thickness of each sample including zirconia substructure with veneered porcelain is 30 mm (28 mm zirconia core plus 2mm veneered porcelain) (Fig.10).

Aging

Autoclaving-induced low temperature deterioration is a well-established technique for accelerating the ageing of Y-TZP materials, despite the fact that thermocycling has been recommended in the literature as a traditional ageing test. So, in accordance with ISO 13356, the samples are autoclaved at 134 ºC and 2 bar pressure for 5 hours to mimic oral circumstances for 15 years.

Surface roughness analyzation:
The surface roughness of all the specimens of all groups are measured with a contact proliferometer (Model - SJ210, Mitutoyo) (Fig.11,12) .

Flexural strength test
Following the assessment of surface roughness, a 3-point flexural strength test was performed using a universal testing machine in accordance with International Organization for Standardization (ISO) 6872:2015 (Dentistry-Ceramic materials) at a loading rate of 1mm/min . The load was applied to the specimen's centre (Fig.13). The maximum load at fracture for each specimen was measured in N, and flexural strength values (MPa) were computed using the formula below: FS =3Fd/ 2wh2 , B where F is the maximum load at fracture (N), d is the distance between the supports.

Shearbond test
The samples were held in place using a metal jig to measure the shear bond strength. The samples were supported so that the ceramic veneering/zirconia substructure contact was facing the chisel load applicator (Fig.13). A chisel load applicator was used to apply a parallel shearing force to the substructure/veneer ceramic interface (Fig.14).The following formula was used to calculate the shear bond strength (MPa):

Shear bond strength (MPa) = Fracture load (kg) / Area of disc (mm2).

Fig.1 Fig.2 Fig. 3

Fig. 1:- (Emax ZIRCAD) ; **Fig. 2:-** (Weiland Zeno zr) ; **Fig. 3:-** (Vita lumex)

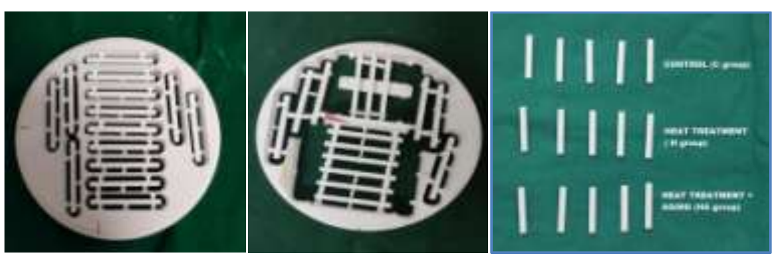


Fig. 4 Fig. 5 Fig. 6

Fig. 4& 5:- (milling), Fig. 6(15 samples of size 28mm×4mm×2mm)

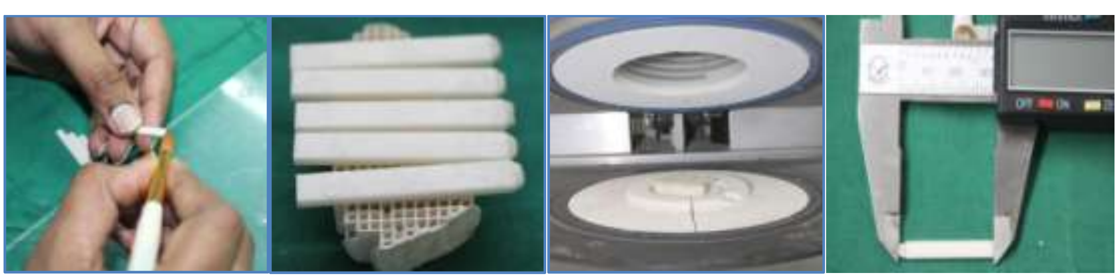


Fig. 7 Fig. 8 Fig. 9 Fig. 10

Fig. 7,8,9:- (manual ceramic layering and firing);
Fig. 10:- (final sample 30mm×4mm×2mm)

Fig.11 Fig. 12 Fig. 13 Fig.14

Fig. 11,12,13,14 (11&12: surface roughness evaluated with proliferometer;
Fig. 13:- Flexural strength & Fig. 14 shear bond srength Using universal testing machine).

Results:-

The data was subjected to Statistical Analysis using IBM SPSS version 21.

Descriptive Statistics were applied to obtain the Mean and Standard Deviation (SD) of the continuous variables present in the data.

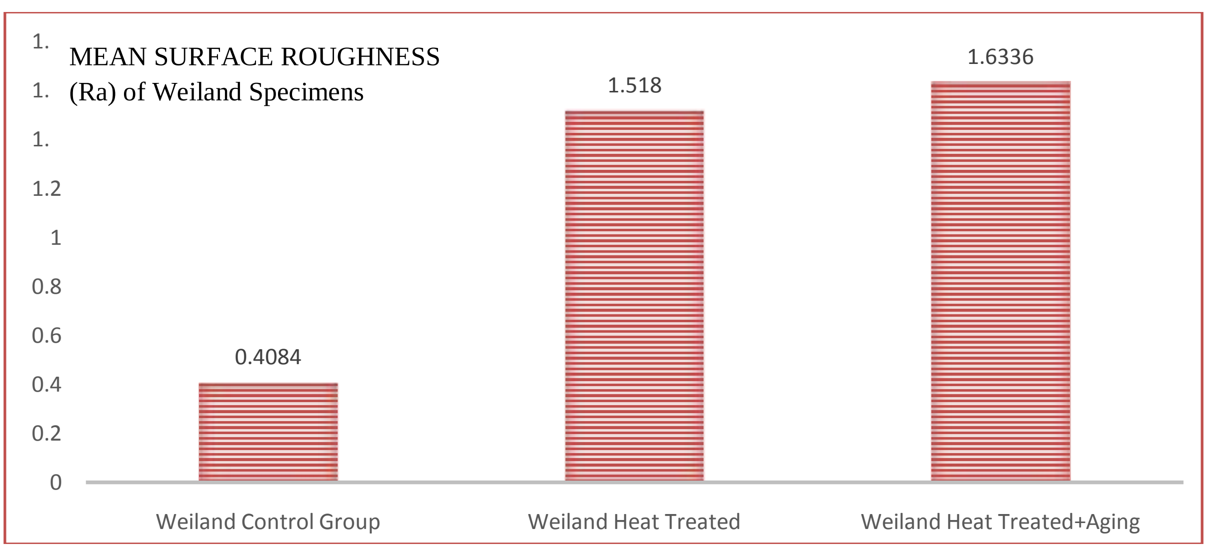
To compare within Group ANOVA with Post Hoc Tukey's was applied.

All tests were applied keeping Confidence interval at 95% and ($P<0.05$) was considered to be statistically significant.

Mean Distribution of Surface Roughness (Ra) values obtained from Weiland specimens and Ivoclar specimens are listed in table 1,2 and graphs.

(Ra) Weiland					
	N	Minimum	Maximum	Mean	Std. Deviation
Weiland Control Group	5	.33	.54	.4084	.08564
Weiland Heat Treated Group	5	1.49	1.58	1.5180	.03699
Weiland Heat Treated+Aging Group	5	1.53	1.71	1.6336	.06740
Total	15	.33	1.71	1.1867	.57502

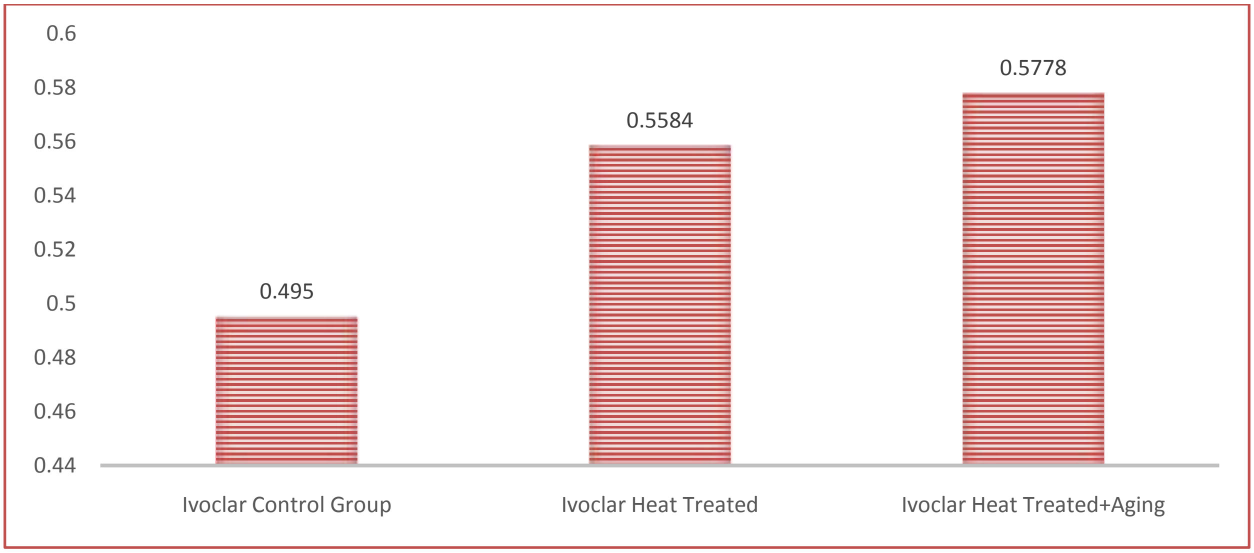
Table 1:- Mean Distribution of Surface Roughness (Ra) in Weiland specimens.



Graph 1:- Mean surface roughness of weiland specimens.

Table 2:- Mean Distribution of Surface Roughness (Ra) in Ivoclar specimens.

MEAN SURFACE ROUGHNESS (Ra) OF IVOCLAR SPECIMENS	N	Minimum	Maximum	Mean	Std. Deviation
Ivoclar Control Group	5	.39	.61	.4950	.10409
Ivoclar Heat Treated	5	.52	.65	.5584	.05664
Ivoclar Heat Treated+Aging	5	.50	.62	.5778	.05356
Total	15	.39	.65	.5437	.07856



Graph 2:- Mean surface roughness of Ivoclar specimens.

When comparison of the Surface Roughness was done in the Weiland Group it was observed that the comparison was statistically significant for Surface Roughness (Ra) ($p<0.05$) .

When comparison of the Surface Roughness was done in the Ivoclar Group it was found that the comparison was statistically not significant for Surface Roughness (Ra) ($p>0.05$) .

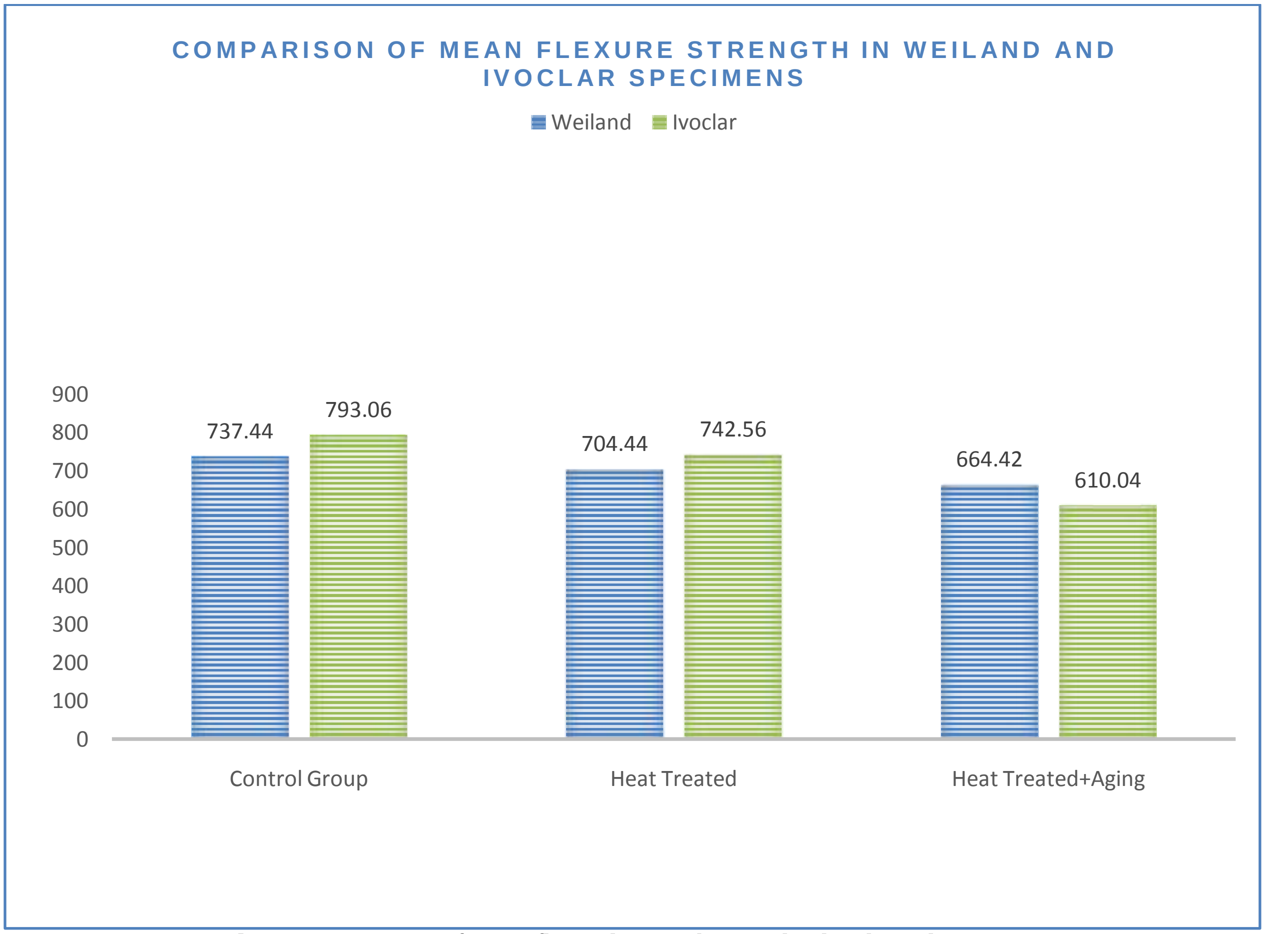
The mean Flexure Strength in Weiland Control Group, Heat Treated & Heat Treated+Aging was 737.440 ± 70.50, 704.44 ± 27.92 & 664 ± 25.47 respectively.

Descriptives					
Flexural Strength (MPa) Weiland					
	N	Minimum	Maximum	Mean	Std. Deviation
Weiland Control Group	5	641.40	805.60	737.4400	70.50208
Weiland Heat Treated	5	676.90	734.50	704.4400	27.92325
Weiland Heat Treated+Aging	5	637.50	693.30	664.4200	25.47022
Total	15	637.50	805.60	702.1000	52.75740

Table 3:- Mean Distribution of Flexure Strength in Weiland Specimens.

The mean Flexure Strength in Ivoclar Control Group, Heat Treated & Heat Treated+Aging was 793.06 ± 59.64, 742.56 ± 107.76 & 610.04 ± 47.35 respectively.

Descriptives					
Flexural Strength (MPa) Ivoclar					
	N	Minimum	Maximum	Mean	Std. Deviation
Ivoclar Control Group	5	749.20	858.40	793.0600	59.64812
Ivoclar Heat Treated	5	574.00	837.00	742.5600	107.76914
Ivoclar Heat Treated+Aging	5	565.30	679.00	610.0400	47.35333
Total	15	565.30	858.40	715.2200	106.57143

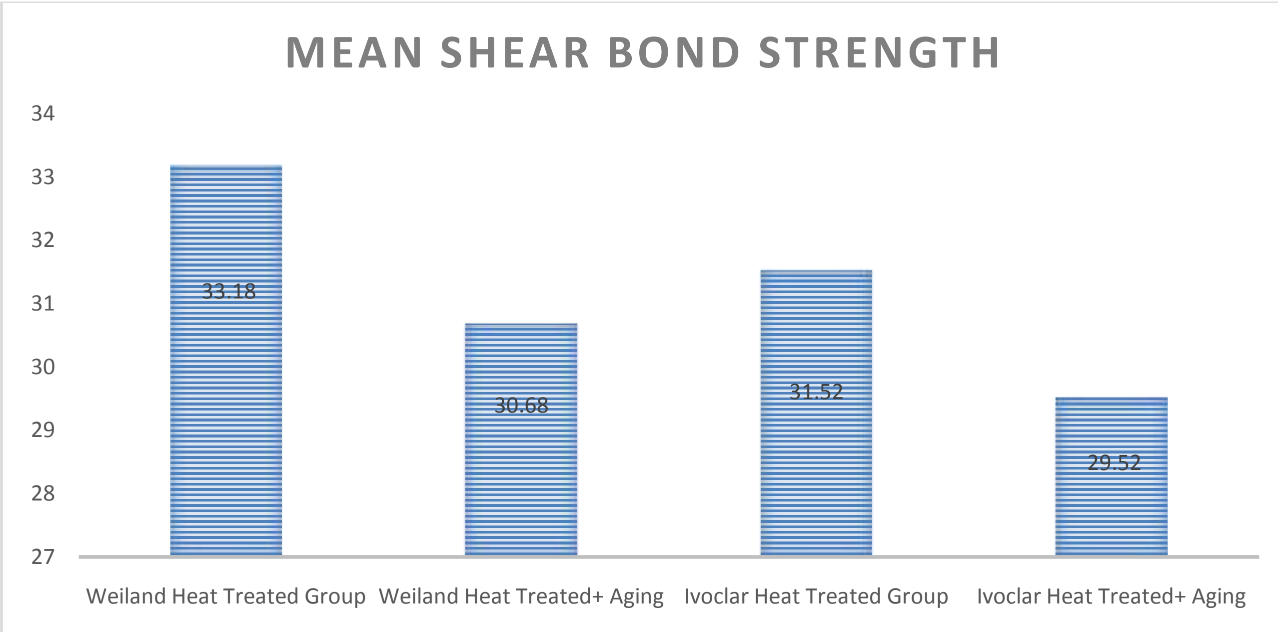


Graph 3:- Comparision of mean flexural strength in weiland and Ivoclar specimens.

The mean Shear Bond Strength of Weiland Heat Treated Group, Weiland Heat Treated+ Aging, Ivoclar Heat Treated Group & Ivoclar Heat Treated+ Aging was 33.18 ± 0.38, 30.68 ± 0.70, 31.52 ± 2.01 & 29.52 ± 1.21 respectively.

Table 4:- Mean Distribution of Shear Bond Strength between Groups.

Mean Distribution of Shear Bond Strength between Groups					
	N	Minimum	Maximum	Mean	Std. Deviation
Weiland Heat Treated Group	5	32.70	33.60	33.1800	.38341
Weiland Heat Treated+ Aging	5	30.10	31.90	30.6800	.70852
Ivoclar Heat Treated Group	5	28.10	32.90	31.5200	2.01544
Ivoclar Heat Treated+ Aging	5	27.50	30.60	29.5200	1.21120
Total	20	27.50	33.60	31.2250	1.78115



Graph 4:- Mean distribution of shear bond strength between two groups.

Discussion:-

The present in-vitro study evaluated the influence of heat treatment and in vitro aging on mechanical properties and surface roughness of cold isostatic pressed zirconia(IPS emax ZirCAD & Weiland ZENO Zr).

Zirconia ceramics have gradually replaced other ceramics in dental prosthesis restorations due to their higher fracture toughness and flexural strength.To achieve esthetics particularly in anterior teeth region ceramic veneering is done in case of monolithic zirconia because these zirconia are opaque whitish in color and unesthetic.Multiple firings done to achieve desired esthetics may effect the flexural strength and shear bond strength and surface characteristics. This effect is due to release of residual compressive stresses that are produced during milling and various processing steps like grinding and sandblasting. Marit Øilo et al.,[5] and Subas et al.,[18] concluded that there is reduction in flexural strength after multiple firings.

The material's mechanical properties may further deteriorate after being exposed to the oral environment, lowering its strength and surface properties.This is due to surface tetragonal to monoclinic phase transformation (t→m) and grain push out. Sung-Hun Kim, and Yu-Sung Choi[9], showed that there is increase in surface roughness after hydrothermal aging. Shams waaz Amgd Ali & Shareen Koth Salem[10] , stated that aging has a significant effect on shear bond strength.

Most research to date has examined the impact of either heat treatment or age alone on the mechanical properties and surface features of zirconia. Studies were ambiguous in determining how Y-TZP ceramics, were affected by heat treatment and concurrent ageing on their mechanical capabilities and surface characteristics.

In the present study, the types of zirconia ceramics used are Ivoclar IPS emax ZirCAD and Weiland ZENO Zr, which are cold isostatic pressed zirconia. Cold isostatic pressing of Y-TZP dental ceramics can result in products with identical compositional and microstructural properties, but there can also be variations depending on the manufacturing process. Based on prior understanding about dental zirconia ceramics, no safe forecasts can be made because manufacturers are always modifying and fine-tuning the processing variables to make their goods better. On the basis of this idea, this study looked at two widely used zirconia ceramics for which there is little knowledge about the mechanical and structural characteristics that can affect their clinical behaviour.

In the two test specimens (Ivoclar & Weiland) there is no significant difference in surface roughness between control (C) and heat treated (H) groups.

There is a significant difference in surface roughness between the control group (C) and the heat treatment with a simultaneous aging group (HA) of two test specimens (Ivoclar & Weiland) which is in consensus with the study done by Matalon et al.,[15] who concluded that there is an increase in surface roughness after aging.

The increase in surface roughness is due to interaction of water molecules with the yttrium (Y2O3) results in an increase in tetragonal to monoclinic(t→m) phase change, which increases grain size and pushes out on the surface[18.]

Between two test specimens (Ivoclar & Weiland), the surface roughness is higher in Weiland specimens than Ivoclar specimens. This may due to less Weibull modulus of Weiland specimens, indicating more flaws and defects in the material that describes the more tetragonal to monoclinic phase transformation[7].

The results in the present research were in accordance to the study on the surface roughness of monolithic zirconia conducted by Sung-hun Kim and Yu-sung Choi[9], in which the authors concluded that an increase in the monoclinic (m) phase on the surface is what causes an increase in surface roughness.

There is a significant difference in flexural strength between the control group (C) and the heat treatment with a simultaneous aging group (HA) of two test specimens (Ivoclar & Weiland) which is congruent to the study done by Khulud et al.,[19] who concluded that aging has significant negative effect on flexural strength. This might be becauseof release of compressive stresses produced during milling and various processing steps[7,8]

According to the current study, both test specimens (Ivoclar & Weiland) experienced a decrease in flexural strength after aging, which is contrary to the findings of Papanagiotou et al.[3] study, who found that aging had no appreciable detrimental impact on flexural strength.

In the current study there is a significant difference in shear bond strength values of both test specimens (Ivoclar & Weiland) after aging which is in congruent with the study done by Shams waaz Amgd Ali & Shareen Koth Salem[10], who stated that aging has a significant effect on shear bond strength between zirconia core and veneer.

According to the results of this study, simultaneous heat treatment and aging have a substantial impact on surface roughness, shear bond strength, and flexural strength. This is the outcome of the surface's tetragonal to monoclinic(t→m) phase transformation, which is brought on by the release of the structure's remaining compressive stresses following heat treatment and the subsequent low-temperature degradation brought on by aging. This means that heat treatment and concurrentageing have an additive influence on zirconia's mechanical characteristics and surface roughness (IPS emax ZIRCAD & Weiland ZENO Zr).

Limitations and further scope of the study:
Tests specimen's geometry is different from the crowns used in real clinical scenarios

The flexural strength and shear bond strength were investigated under static loading instead of dynamic loading, which better simulates intra-oral conditions.

Analyzing the distribution of monoclinic crystalline phase on the surface of the samples using scanning electron microscope or X-ray diffraction analysis might provide more logical reason for variations in mechanical and surface characteristics.

Conclusion:-

The following conclusions were drawn with in the constraints of this in-vitro study.

There is a decreased flexural strength and shear bond strength and increase in surface roughness of both the test specimens (i.e Ivoclar IPS emax ZirCAD & Weiland ZENO Zr) when the heat-treated zirconia samples are aged.

When both the test specimens are compared, Ivoclar specimens demonstrated higher flexural strength, shear bond strength and lower surface roughness than Weiland specimens.

From the above drawn conclusions, it is signified that during the fabrication of multi-layer zirconia restorations, the firing cycles should be minimized to enhance the longevity of the restorations.

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