



EVALUATION OF PHYSICAL PROPERTIES OF CERIUM AND YTTRIUM STABILIZED ZIRCONIA COPINGS - AN IN VITRO STUDY.

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Abstract:

Zirconia-based restorations have turned out to be better on account of its high mechanical properties, biocompatibility and aesthetic properties. The negligible marginal fit is the most prominent basis for deciding the clinical success rate. Fracture resistance ought to be surveyed in light of the utilization of the restoration in load bearing regions.

Aims and objectives:

This in vitro study is aimed to assess the marginal fit, fracture resistance, surface roughness, hardness and phase transformation of yttrium and cerium stabilized zirconia copings with three different occlusal thicknesses and subjecting them to two different aging heat treatment regimens (134° C and 180°C).

Materials and methodology:

A total of 180 zirconia copings (yttrium and cerium) were fabricated and divided into three groups control group, group 1(134° C) and group 2 (180°C). After subjecting the samples to two different aging conditions, marginal fit and fracture resistance were assessed by using stereomicroscope and universal testing machine.

Results:

The mean marginal fit and fracture resistance of zirconia copings showed a statistical significance ($p < 0.01$). Mean marginal gap for 1.8mm thickness samples which were aged at 180°C showed much significance ($p < 0.01$). The mean surface roughness, hardness and phase transformation of zirconia samples did not show much significance ($p > 0.09$)

Conclusion:

When comparing the results between cerium and yttrium stabilized zirconia; cerium has high fracture resistance and exhibited marginal gap, increased surface roughness, hardness and phase transformation when compared to yttrium which was statistically significant.

Keywords: Y-TZP, Ce-TZP, t-m transformation, LTD, marginal fit, fracture resistance.

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INTRODUCTION

The main objective of all the oral restoration processes is to endow with functional, aesthetic and appealing results.¹ It helps in maintaining harmony with the stomatognathic system and patient's quality of life is improved. Porcelain-fused-to-metal restorations have been considered as a gold standard as these restorations combined the strength & accuracy of cast metal and aesthetics of porcelain.^{1,2}

Driven by the debatable drawbacks associated with porcelain fused metal ceramic restorations all-ceramic restorations i.e zirconia-based ceramics was considered in dentistry.^{3,4} Pure zirconia exhibits monoclinic microstructure at room temperature and tetragonal above 1170 degrees centigrade and cubic above 2370 degrees centigrade.^{5,6} The tetragonal form was stabilized at room temperature (37°C) by adding up of various oxides including yttrium (Y_2O_3), Ceria (CeO_2), Calcium (CaO) and Magnesia (MgO).⁶

Zirconia tends to have elevated mechanical toughness, excellent properties of esthetic. When it is intended for the biomedical application it is often stabilized with 3 mol percentage yttria which increased the mechanical strength and lowered the fracture resistance due to their limited transformability.^{7,8} By doping ceria, i.e. Ce-TZP (12 mol %) it has been described that during low-temperature aging, t-m phase transformation was controlled fully and showed resistance to phase transformation, so cerium is more frequently used.⁹ Due to escalating utilization of zirconia as monolithic restoration material and fact that its mechanical properties were affected negatively by Low Temperature Degradation, this study has been carried out to assess the performance of yttrium- and cerium stabilized monolithic zirconia samples of three different occlusal thicknesses (0.8mm,1.2mm,1.8mm) which were subjected to two different ageing regimens to evaluate the

marginal fit, hardness, surface roughness, fracture resistance and phase transformation.

MATERIALS AND METHODOLOGY

After obtaining the institutional ethical clearance, a total of 180 samples were fabricated by CAD/CAM technology with three different occlusal thickness 0.8mm, 1.2mm and 1.8mm each and were subjected to two different aging conditions (134° C and 180° C, 0.2 Mpa pressure, for 10 hours). Zirconia unsintered blank Yttrium Stabilized Zirconium Disc (Sagemax, Nexxer, USA) and Cerium Stabilised Zirconia Disc (Bloomden, Bloomden Bioceramics Co, Ltd, CHINA) were used for this study. Copings were fabricated to evaluate the marginal fit and fracture resistance by using a customized stainless steel die replicating maxillary 1st premolar tooth with 12° convergence angle, 8mm occlusogingival height and flat surface of occlusion. A groove of length 3mm and depth 0.5mm was prepared to aid in the repositioning of the crown and acts as an anti-rotation aspect. Titanium dioxide powder was sprayed on the master die for precise scanning, designing and milling.

40 bar-shaped samples of dimensions 24×3×1.2mm were fabricated according to ISO 4049 to measure surface roughness and hardness. 20 Disc-shaped samples of dimensions 10×3mm were fabricated according to ISO 6872 to evaluate phase transformation before and after two aging treatments. 90 Copings for each of yttrium and cerium stabilised zirconia were fabricated (T 1 - 134°C: 30 samples per group) and (T 2 - 180°C: 30 samples per group) with a control group of 30 samples. The test samples of three thickness were divided into two groups (n=10) in which each group of zirconia samples (yttrium and cerium stabilized) were subjected to aging treatment, i.e., 134°C, 10 hours, 0.2Mpa and 180°C, 10 hours, 0.2Mpa respectively. After the completion of aging treatment, samples were evaluated for marginal gap using stereomicroscope (Fig 1)

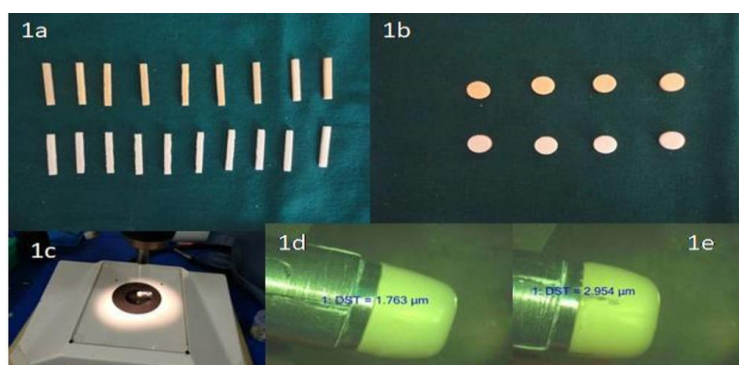


Figure 1a: Bar shaped zirconia samples ,1b. Disc shaped zirconia samples,1c. zirconia coping placed on stereomicroscope,1d. Result obtained for yttrium sample, 1e. Result obtained for cerium sample

Testing for fracture resistance was done by placing the sample on the loading platform with compressive pressure applied at a cross head rate of 0.5mm/min using a ball ended plunger in Instron (universal testing machine). Bar-shaped sample of dimensions was evaluated before aging and after aging by profilometer to study surface roughness and to evaluate the hardness, Vickers hardness tester is used. A load of 19.6N was used. Samples were placed on the platform and a standard load is set with a dwell time of 10 seconds.

Disc shaped samples were subjected to x ray diffraction before and after aging heat treatment and the amount of monoclinic phase alterations were determined. To assess the micro structural characteristics of samples, scans were done at 2 θ , from 20 to 70 degrees with incremental step up of 0.05 degree.

Results and observations:

On comparison of mean values of marginal fit of cerium and yttrium samples of various thickness (0.8mm,1.2mm and 1.8mm) after subjecting them to aging treatments (control, 134 $^{\circ}$ c, 180 $^{\circ}$ c) (Table 1) shows that the marginal discrepancy was more for cerium samples compared to yttrium samples and gradually increased from 0.8mm to 1.8mm. For 0.8mm, 1.2mm and 1.8mm thickness samples, control group showed less marginal gap followed by 134 $^{\circ}$ c and 180 $^{\circ}$ c.

On comparison of mean values of fracture resistance of cerium and yttrium samples of various thickness (0.8mm,1.2mm and 1.8mm)

after subjecting them to aging treatments (control, 134 $^{\circ}$ c, 180 $^{\circ}$ c) (Table 2) shows that higher values were reported for cerium samples followed by yttrium. For 1.8mm thickness samples at 180 $^{\circ}$ C cerium samples showed higher values which means that cerium was able to withstand more amount of load when compared to yttrium. At 134 $^{\circ}$ C there is not much significance. For control group, 134 $^{\circ}$ C and 180 $^{\circ}$ C the significance value is (p value <0.01). It shows that higher values were reported for cerium samples followed by yttrium.

On comparison of surface roughness of cerium and yttrium samples after subjecting them to aging treatments (control, 134 $^{\circ}$ c, 180 $^{\circ}$ c) (Table 3) shows that cerium samples have higher surface roughness values when compared to yttrium at 134 $^{\circ}$ C and 180 $^{\circ}$ C.

On comparison of hardness values between cerium and yttrium samples after subjecting them to aging heat treatment (control, 134 $^{\circ}$ c and 180 $^{\circ}$ c) (Table 4) shows that there is not much statistically significance of cerium and yttrium samples at 134 $^{\circ}$ C and 180 $^{\circ}$ C but cerium reported higher hardness values when compared to yttrium which suggests that cerium is more resistant to hardness. The phase transformation values of cerium samples and yttrium samples before subjecting them to aging heat treatment was 30.62 and 30.44 at 134 $^{\circ}$ c (Graph 1 & 3) and 30.52 & 30.48 at 180 $^{\circ}$ c (Graph 2 & 4) which means that monoclin phase transformation has begun at that peak value and was higher for cerium samples when compared with yttrium.

Table 1: Comparison of mean values of marginal fit of cerium and yttrium samples of various thickness(0.8mm,1.2mm and 1.8mm) after subjecting them to aging treatments(control, 134 $^{\circ}$ c, 180 $^{\circ}$ c) by using independent t-test.

Thickness	Groups		Mean	SD	P-value
0.8 mm	Control	Cerium	9.32	0.81	0.52
		Yttrium	9.05	0.98	
	134 $^{\circ}$ C	Cerium	9.79	0.98	0.45
		Yttrium	9.47	0.91	
	180 $^{\circ}$ C	Cerium	10.57	1.14	0.3
		Yttrium	10.06	1.00	
1.2 mm	Control	Cerium	10.9	1.5	0.06
		Yttrium	9.59	1.37	
	134 $^{\circ}$ C	Cerium	11.41	1.43	0.04*
		Yttrium	10.02	1.38	
	180 $^{\circ}$ C	Cerium	12.02	0.54	<0.01*
		Yttrium	10.58	1.6	
1.8 mm	Control	Cerium	11.04	0.84	<0.01*
		Yttrium	10.00	0.86	
	134 $^{\circ}$ C	Cerium	11.46	0.72	<0.01*
		Yttrium	10.23	0.76	
	180 $^{\circ}$ C	Cerium	12.35	1.32	<0.01*
		Yttrium	10.67	0.77	

* - P value statistically significant by using independent t-test

Table 2 : Comparison of mean values of fracture resistance of cerium and yttrium samples of various thickness(0.8mm,1.2mm and 1.8mm) after subjecting them to aging treatments (control, 134°C, 180°C) by using independent t-test.

Thickness	Groups		Mean	SD	P-value
0.8 mm	Control	Cerium	2188.3	102.21	<0.01*
		Yttrium	1723.7	238.52	
	134°C	Cerium	1513.3	174.49	0.09
		Yttrium	1384.3	149.45	
	180°C	Cerium	1257.1	96.71	<0.01*
		Yttrium	1121.2	120.62	
1.2 mm	Control	Cerium	2575.9	109.47	<0.01*
		Yttrium	2351.2	95.99	
	134°C	Cerium	2443.2	124.97	<0.01*
		Yttrium	2167.6	178.36	
	180°C	Cerium	2335.3	135.77	<0.01*
		Yttrium	1845.9	308.14	
1.8 mm	Control	Cerium	3671.3	430.59	<0.01*
		Yttrium	2683.3	333.74	
	134°C	Cerium	2805.8	300.79	<0.01*
		Yttrium	2477.5	198.3	
	180°C	Cerium	2386.7	311.33	0.13
		Yttrium	2191.7	230.21	

* - P value statistically significant. Comparison of fracture resistance of cerium and yttrium by using independent t-test

Table 3: Comparison of surface roughness of cerium and yttrium samples after subjecting them to aging treatments(control, 134°C, 180°C) by using independent t-test.

Groups		Mean	SD	P-value
Control	Cerium	0.65	0.10	0.51
	Yttrium	0.57	0.05	
134°C	Cerium	1.10	0.37	0.02*
	Yttrium	0.77	0.12	
180°C	Cerium	1.28	0.29	<0.01*
	Yttrium	0.87	0.12	

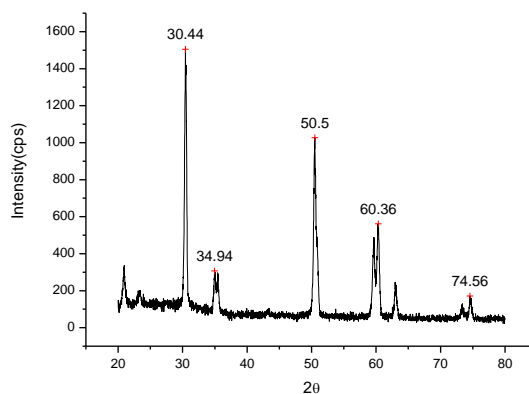
* - P value statistically significant by using independent t-test

Table 4: Comparison of hardness values of cerium and yttrium samples after subjecting them to aging heat treatment (control, 134°C, 180°C) by using independent t test.

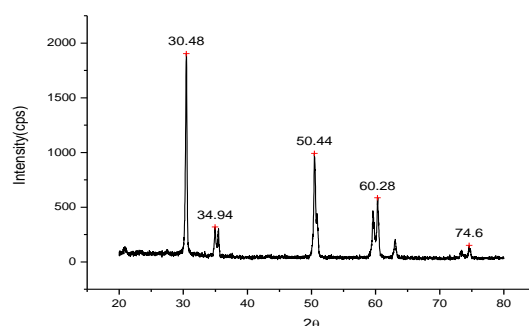
Groups		Mean	SD	P-value
Control	Cerium	100.31	0.22	0.06
	Yttrium	99.59	0.93	
134°C	Cerium	100.05	0.37	0.14
	Yttrium	99.41	1.36	
180°C	Cerium	99.3	1.58	0.16
	Yttrium	98.12	1.86	

* - P value statistically significant. Comparison of hardness values of cerium and yttrium samples by using independent t-test

Graph 1 & 2: Schematic representation of phase transformation values of yttrium samples after subjecting them to aging heat treatment 134°C and 180°C

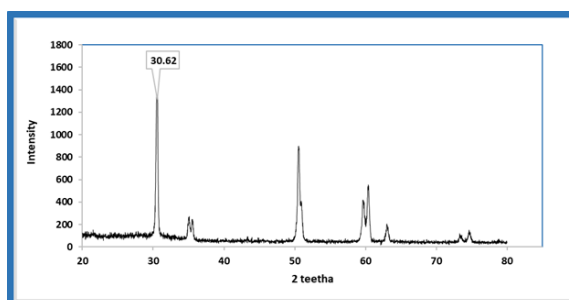


Graph 1: Phase transformation of yttrium samples after subjecting them to aging heat treatment 134°C. Peak value obtained at 30.44

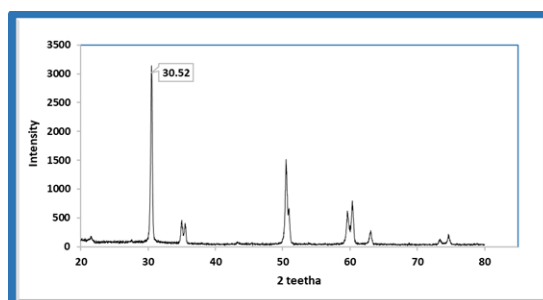


Graph 2: Phase transformation of yttrium samples after subjecting them to aging heat treatment 180°C. . Peak value obtained at 30.48

Graph 3 &4: Schematic representation of phase transformation values of cerium samples before subjecting them to aging heat treatment 134°C and 180°C.



Graph 3: Phase transformation of cerium samples before subjecting them to aging heat treatment 134°C. Peak value obtained at 30.62



Graph 4: Phase transformation of cerium samples before subjecting them to aging heat treatment 180°C. Peak value obtained at 30.52

Discussion

Ceramic restorations have turned out to be better known because of rising patient requests for aesthetics. With porcelain fused to metal restorations, some obstacles have been reported such as ceramic chipping or debonding, incompatibility between metal and ceramic^{1,2}. So with the end goal to beat those defects, zirconia-based ceramics as a restorative material has been considered in dentistry.^{3,4} These materials showed good mechanical quality, prevalent crack obstruction and superior fracture resistance. Zirconia core materials are usually added with translucent veneering porcelains for better esthetics.^{4,5} But for layered zirconia restorations failures have been reported. Fractures were observed to originate from the weak points, the veneer or the core/veneer interface, resulting in chipping or delamination of the veneer and cracks extending through the core materials.^{6,7} So, monolithic restorations have been introduced.

Zirconia polycrystals are commonly stabilized with 3 mol percentage of yttria (3Y-TZP) because it is more commonly used for medical grade purpose. Stabilizers such as cerium, magnesium and calcium were added to diminish the t– m change rate.^{7,8} Among them, cerium was generally utilized because it was steady in zirconia underneath 1000°C and there were no associated structural vacancies. Also, cerium did not exhibit any transformation after hydrothermal aging at 150°C.^{9,10}

Three different occlusal thickness were taken because clinicians have suggested that minimum occlusal thickness for zirconia restoration is 0.5mm.¹⁰ A minimum thickness of 1.5mm is required for monolithic full contour zirconia crown to avoid radial cracks.¹¹ Near the developmental grooves and fissures a minimum occlusal thickness of 1.5mm, for functional cusp about 1.5mm and non-functional cusps around 1mm should be maintained to avoid fractures.^{12,13} So 0.8mm thickness was taken to simulate core thickness which can be veneered with ceramics. 1.2mm and 1.8mm thickness were taken to simulate for functional and non-functional cusps and fabrication of monolithic restorations.

The obtained results concluded that as the thickness increases from 0.8mm to 1.8mm, the fracture resistance increases for both cerium and yttrium samples. The highest values ever obtained for 1.8mm, followed by 1.2mm and 0.8mm. When compared among cerium and yttrium, cerium has high fracture resistance because of the amount of the stabilizers.¹³ The strength and fracture toughness decrease with the increasing temperature

because the stability of t- phase increases and there is a chemical driving force for t-m transformation.^{14,15}

The phase transformation toughening phenomenon results in high strength and fracture toughness of zirconia.¹⁵ Tetragonal-to-monoclinic phase transformation can likewise occur at room temperature when tetragonal phase zirconia is in contact with water, which is called low-temperature degradation (LTD) or aging.^{16,17} Low-temperature degradation occurs within the temperature range of 65-500 °C, maximum at 250 °C¹⁸

Abdulrahman et al in their study investigated the thickness of core design of coping can affect the fracture resistance of zirconia veneered crowns. Zirconia crowns were prepared with 0.6mm, 1.2mm and 1.7mm. The results showed that the 1.7mm thickness group has greater fracture resistance than the other groups.⁹

Chevalier et al. stated that aging of zirconia for 1 hour at 134 °C at 2 bar pressure results in significant t – m transformation that has the same effect as 3-4 years in vivo.¹⁰

Kosuke Harada et al in their study focused on the condition of 180°C at 1.0MPa for 1 hour as this is used for the tolerance testing of industrial ceramics as part of the conditions for the penetration testing of sanitary ware in ISO 10545-11 Ceramic Tile and Japanese industrial standard (JIS A5207 Sanitary Wares) which is equal to 15-20 years in vivo condition¹⁸. These temperatures were chosen based on a pilot study in which longer hydrothermal treatment produced little change in the results. So in this study aging for 10 hours at 134°C and 180°C at 2 bar pressure was used.

The results concluded that, among the three thicknesses 0.8mm, 1.2mm and 1.8mm less amount of marginal discrepancy was reported for 0.8mm followed by 1.2 and 1.8mm thickness. The marginal fit for yttrium samples for the control group is less when compared to 134°C and 180°C groups. Among cerium and yttrium samples, cerium has reported higher marginal discrepancy compared with yttrium because the amount of stabilizer affected the phase content. Studies have reported that the phase transformation occurs at higher temperatures and changes occur at the microcrystalline level which results in instability of crystalline structure for cerium samples which might be one of the reasons for marginal discrepancy. The obtained values were less than the prescribed range of 40-90µm for both cerium (0.8mm- 10.57, 1.2mm – 12.02, 1.8mm- 12.35µm) and yttrium (0.8mm - 10.06, 1.2mm- 10.58, 1.8mm- 10.67 µm) CAD-CAM restorations. The

variation was very negligible and not statistically significant.

The results showed that cerium samples have higher surface roughness when compared to yttrium at 134°C and 180°C because when the stabilizer was added there was surface modification i.e. by severe micro-cracking, grain pullout and surface roughening.^{19,20}

Thermal aging led to a decrease in hardness values.²⁰ Hardness has to be evaluated because when zirconia restorations were fabricated in posterior region they should withstand the wear and occlusal loads^{20,21} The hardness values of cerium and yttrium samples after subjecting them to aging heat treatment (control, 134°C, and 180°C), shows that there is not much significance of cerium and yttrium samples at 134°C and 180°C. Among them, cerium showed higher values i.e. cerium was more resistance to load when compared to yttrium because by doping cerium the grain boundaries were modified and the t-m transformation was inhibited thereby increasing the hardness value.^{22,23}

According to Chevalier et al. 2004, the amount of m-phase is strongly dependent on the temperature and time, a higher temperature and longer time tend to produce a higher transformation rate¹⁰.

The phase transformation of zirconia samples was measured before and after subjecting them to two different artificial aging conditions. The results obtained showed that the phase transformation was more for cerium at 180°C (30.52) compared to yttrium at 180°C (30.48). The peak value i.e the point at which phase transformation begins is higher for cerium samples at 134°C (30.62) when compared to yttrium at 134°C (30.44) as the t-m transformation was stable in cerium because of the stabilizers and there is an increase in m phase.^{24,25}

Limitations of this study include that along with aging treatment cyclic loading have to be done to simulate the intra oral condition and also inclusion of physiological parameters like saliva, ph changes and the contact time have not been included in this study which may differ from patient to patient.

CONCLUSION

Within the limitations of this study, it was concluded that, Among various thicknesses tested, 1.8mm thickness samples showed more marginal gap and high fracture resistance followed by 1.2mm and 0.8mm thickness values. Cerium stabilized zirconia has reported more marginal gap compared to yttrium stabilized zirconia at 134°C and 180°C. The marginal fit was within the acceptable range. 1.8mm thickness samples can be used in high load bearing areas. Cerium samples showed more surface roughness compared to

yttrium samples. So clinically if cerium restorations were to be indicated frequent polishing protocol for every 6 months has to be followed. Cerium samples showed more hardness values compared to yttrium samples they are more resistant to wear and can be used in high load bearing areas. Cerium samples showed more phase transformation compared to yttrium samples when aged at 180°C because of the increased m phase which is dependent on temperature. .

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