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Evaluation of Fluorides on Colour and Mechanical Properties of Monolith Zirconia & Layered Zirconia

[1]Dr. Sravani Garimella, Post graduate student, Gitam dental college and Hospital, Visakhapatnam, 530045.

[2]Dr. M. Harikrishna, Reader, Gitam dental college and Hospital, Visakhapatnam, 530045.

[3]Dr. Y.Ravishankar, Professor and Head of the department, Gitam dental college and Hospital, Visakhapatnam, 530045.

[4]Dr. Kalluri Srinivas, Professor, Gitam dental college and Hospital, Visakhapatnam, 530045.

Corresponding Author: Dr. Sravani Garimella, Post graduate student, Gitam dental college and Hospital, Visakhapatnam, 530045.

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Abstract

This is an in vitro study that was aimed to assess the effect of three different fluorides (2% NaF, 0.4% SnF_2 & 1.23% APF) on colour and mechanical properties includes roughness & hardness of monolith zirconia and ceramic layered zirconia. A total of 80 rectangular bar shaped samples were fabricated with two different dimensions. ACCORDING TO ISO 6872 PROTOCOL the samples were divided into monolith zirconia (4×3×15mm; n=40) and layered zirconia (4×2×15mm; n=40). The rectangular bar shaped samples were treated with three commercially available fluorides (2% NaF gel, 0.4% SnF_2 gel & 1.23% APF gel). These samples were used to measure colour, roughness, and hardness before and after fluoride application.

Results: Results showed that layered zirconia had high roughness, colour change & low hardness values compared to monolith zirconia samples. Within the group 1.23% APF gel treated samples showed high roughness, colour change & low hardness values compared to 0.4% Stannous fluoride gel & 2% sodium fluoride gel.

Conclusion: Within the limitations of this study, 2% sodium fluoride gel showed least effect on zirconia (monolith zirconia & layered zirconia) followed by 0.4% stannous fluoride gel. The 1.23% APF gel showed more effect on roughness, hardness & colour of zirconia (monolith zirconia & layered zirconia) which were tested in this study.

Keywords: Monolith Zirconia, Layered Zirconia, Sodium Fluoride Gel, Stannous Fluoride Gel, APF Gel, Roughness, Hardness, Colour.

Introduction

Zirconia (zirconium dioxide, ZrO_2), *also named as* "ceramic steel", has optimum properties for dental use [1]. Zirconia doesn't exist in refined state and the principal source of zirconia is conjunction of mineral name

ZIRCON ($ZrO_2 \times SiO_2$) and free oxide (ZrO_2) with mineral name BADDELYITE also known as zirconia [2].

Zirconia is a polymorphic material and occurs in 3 phase's i.e monoclinic, tetragonal and cubic phase. Pure zirconia exhibits monoclinic microstructure which is stable up to 1170°C. Tetragonal zirconia is obtained between 1170°C to 2370°C also called as metastable phase where as cubic zirconia is obtained at temperature above 2370°C. On heating zirconia undergoes monoclinic to tetragonal phase transformation with decrease in volume upto 5%. While cooling, 3% - 4% volume increase is observed and results in cracks due to stress [3,4].

Several oxides are added to zirconia to stabilize the tetragonal and cubic phase such as Magnesia (MgO), Ceria (CeO), Calcia (CaO), and Yttria (Y_2O_3). Addition of varying amounts of stabilizers allow formation of partially/ fully stabilized zirconia with expected properties such as high flexural strength, fracture toughness, high hardness, excellent chemical resistance and good conductivity[3, 5].

Fixed prosthodontics is an essential part of prosthodontic treatment & mostly porcelain is used in fixed restorations due to its natural appearance. Caries control is unavoidable to patients for long term success of porcelain restoration[6,7]. Fluoride treatment resists caries attack on natural as well as restored teeth [7,8].

Various fluoride delivering methods are available out of them tooth paste is a major source of fluoride for communities where fluoridated drinking water is not available. Fluorides added in tooth paste are Sodium Fluoride, Stannous Fluoride, Acidulated Phosphate Fluoride, Sodium Monofluorophosphate, and Amine Fluoride. Common concentrations fluoride used in tooth paste are 1000 – 1500 ppm (adult dosage), 250 - 500 ppm (children) [9].

Recommended levels of fluoride daily intake should be 3 - 4 mg/day in adults and 0.05 mg/day in children. More than recommended levels of fluoride intake may result in toxicity which include dental fluorosis, skeletal fluorosis, muscle fiber derangement, skin rashes, head ache, neurological manifestations & gastrointestinal problems[10]. But the daily fluoride intake may affect the properties of permanent restorative materials including porcelain, composite, glass ionomer cement and sealants. At low PH, fluoride gel can result in formation of hydrofluoric acid, which leads to etching of restorations that contain silica, such as porcelain.

Materials and Methodology

Prior to the procedure a rectangular wax bar was prepared and scanned with optical scanners which collects 3D data of specimens by a process called triangulation procedure, and data is utilized for milling zirconia block into desired dimensions. The dimensions of rectangular bars were programmed using CAD-CAM software, and it transfers data into numerical code and transferred to the CNC machine. Zirconia block was delivered in a presintered state into milling machine. After milling, bar-shaped specimens were placed in sintering furnace. Volumetric shrinkage of about 20% observed in sintered samples, so oversized sample were taken and sintered to compensate shrinkage.

ACCORDING TO ISO 6872 PROTOCOL, a total of 80 rectangular bar shaped samples were milled in the CAD-CAM milling machine with two different dimensions, i.e., 4×3×15mm (i.e., monolith zirconia) & 4×2×15mm (i.e., layered zirconia). These samples were divided into two groups, i.e., monolith zirconia (n= 40) and, layered zirconia (n=40).

The layered zirconia samples (n = 40) were subjected to porcelain layering. Prior to layering the 40 layered zirconia samples were sandblasted to enhance bonding of ceramic layering. Porcelain VITA dentine, enamel layer and glaze was applied on the sandblasted surface of sample with Renfert brush and firing was done in calibrated porcelain furnace. After firing, the samples were finished and polished. To obtain standard uniform layering of zirconia, after each layer of ceramic application the sample is measure with boley's gauge.

Leaving samples of layered zirconia (n=10) and monolith zirconia (n=10) as a control without applying fluoride gel remaining samples were treated with 3 different fluoride gel (2% NaF gel, 0.4% SnF_2 gel & 1.23% APF gel) for about 3 min / 4 times a day for 4 months to simulate 1 year of exposure. A putty mold was prepared to position the samples & fluorides were applied over one side of the samples in unidirectional vertical strokes. Samples were evaluated for colour, roughness, and hardness before and after fluoride application.

Roughness was evaluated by using Profilometer. The specimens to be tested were placed on fixed table and a diamond stylus (50µm tip radius) with length of 2mm at a speed of 0.1mm/s was placed on samples the tip will move for specific distance of 15mm and specific contact force of 3.9N.

Hardness was evaluated by Vickers hardness tester. Vickers hardness tester consist of rotating table which have microscopic lens, light source and, hardness indenter on upper member. First sample was marked with a colour pencil on target site and placed on a platform of testing machine. The lens was adjusted until the marking was visible in microscopic lens. The standard load of 9.807N was recommended for ceramic diamond pyramid head of tester for a holding time of 10 sec on the surface of sample. These values were adjusted in display of hardness testing machine. After unloading indenter moves up from the surface of sample and, the rotating table rotates by positioning lens onto the target area of sample. Now zoom in on the indentation, adjust the center lines to the diagonal and overlap the outer lines at the center of the indentation then hit zero. Later from the center move the outer lines back to the edges then rotate the eye piece & *lock it. Hit the black button on right side of the testing machine which gives L_1 value. Re*peat the above procedure by rotating the eye piece to 90° *again hit the black button for L_2 value. The average of L_1 & L_2 gives* hardness value of sample.

Colour is evaluated by using spectrophotometer. The colour change calculated with the use of common international de IE dairge L*A*B* (CIE-LAB) uniform scale determined using a VITA Easyshade Advance 4.0. L* representing colour luminosity varies from white to black. The higher the L* indicates the specimen is lighter . a* and b* coordinate represent the chromacity of colour with an axis vary from green to red, blue to yellow. A positive a* relates to the amount of redness, and a negative a* relates to the greenness of specimen. A positive b* refers to the amount of yellowness, while a negative b* relates to the blueness of the sample.

ΔE*= [(ΔL*) 2 + (Δa*) 2 + (Δb*) 2] ½

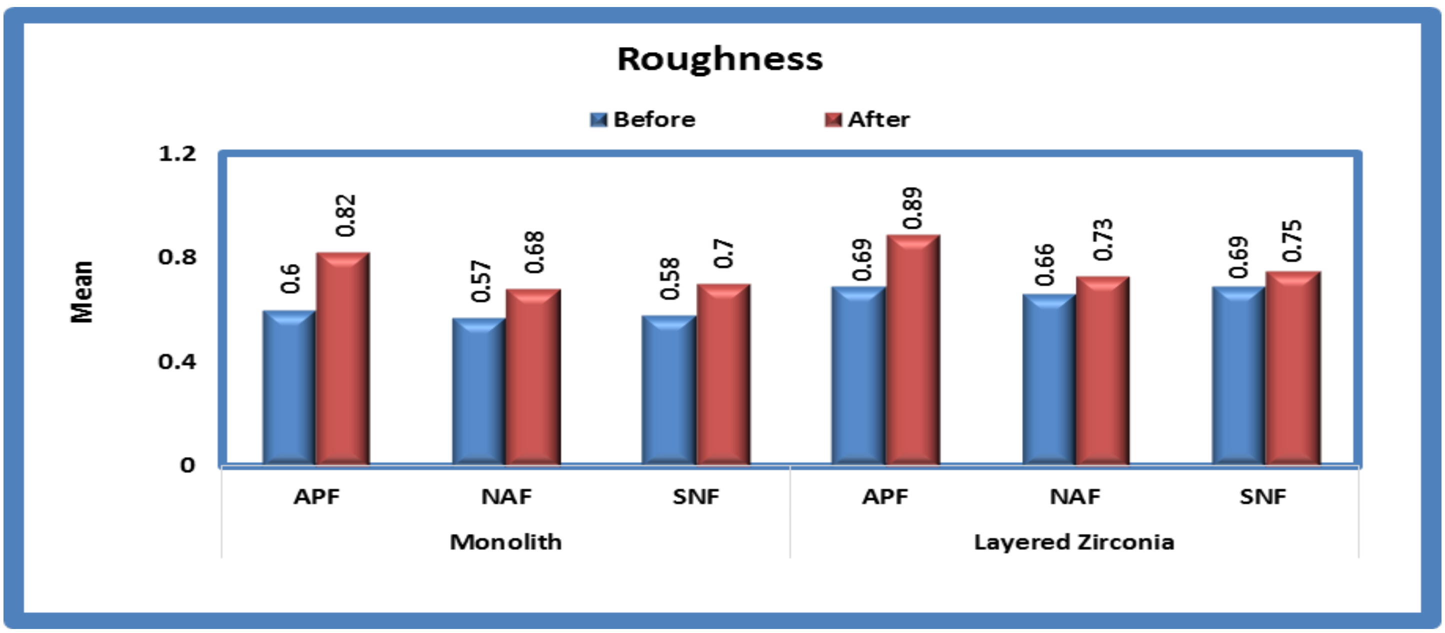
Using L*, a* & b* values all ΔE values were obtained from the above formula. The ΔE of all samples before fluoride treatment and ΔE values obtained for all samples after treatment were useful for evaluating colour change.

Results

Table 1

		Roughness				P-value
		Before		After		
		Mean	SD	Mean	SD	
Monolith	APF	0.60	0.05	0.82	0.05	<0.01*
	NaF	0.57	0.04	0.68	0.04	<0.01*
	SnF_2	0.58	0.05	0.70	0.03	<0.01*
Layered Zirconia	APF	0.69	0.06	0.89	0.03	<0.01*
	NaF	0.66	0.04	0.73	0.04	<0.01*
	SnF_2	0.69	0.06	0.75	0.03	<0.01*

Graph 1



From the above table 1 & graph 1

Monolith zirconia shows higher roughness value for 1.23% APF gel (0.82), 0.4% SnF_2 gel (0.70) & 2% NaF gel (0.68) after fluoride gel treatment when compared with same monolith zirconia samples before fluoride exposure

Layered zirconia shows higher roughness value for 1.23% APF gel (0.89), 0.4% SnF_2 gel (0.75) & 2% NaF gel (0.73) after fluoride gel treatment when compared with same layered zirconia samples before fluoride exposure

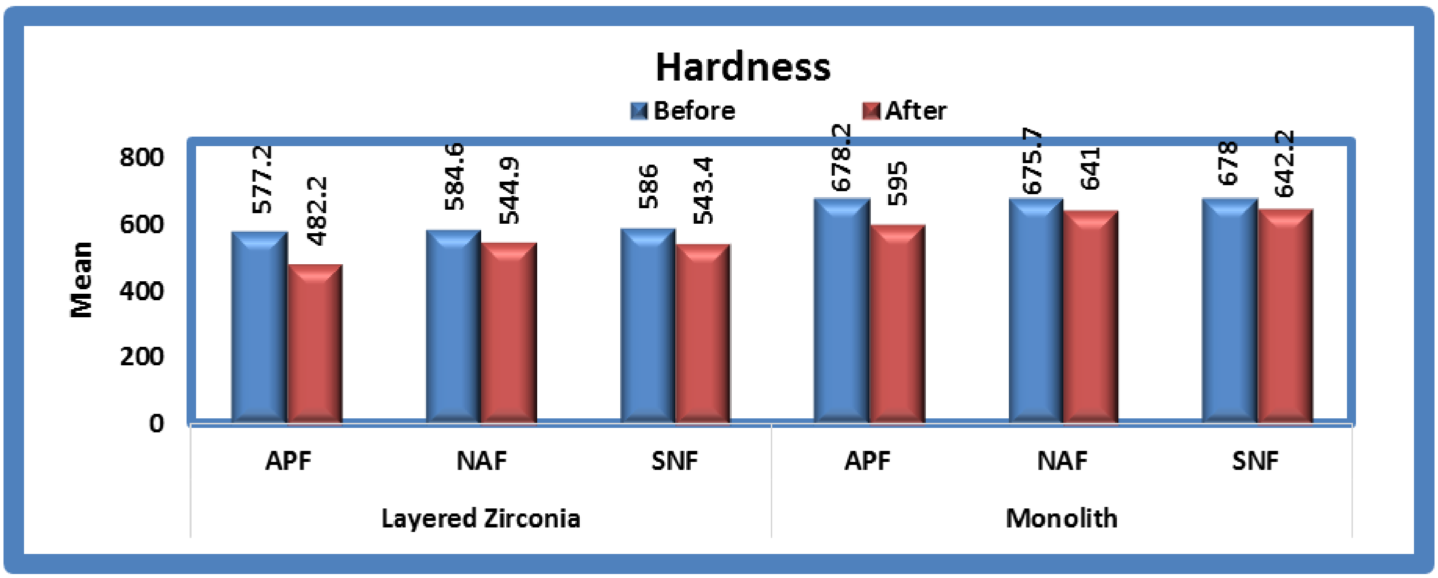
When compared between monolith zirconia, and layered zirconia, layered zirconia treated with fluorides (1.23% APF gel, 0.4% Stannous fluoride gel, and 2% Sodium fluoride gel) showed high roughness than monolith zirconia samples.

When compared within group 1.23% APF gel treated zirconia samples (monolith zirconia & layered zirconia) showed higher roughness values.

Table 2:

		Hardness				P-value
		Before		After		
		Mean	SD	Mean	SD	
Layered Zirconia	APF	577.20	15.96	482.20	13.10	<0.01*
	NaF	584.60	9.36	544.90	9.43	<0.01*
	SnF_2	586.00	11.04	543.40	10.69	<0.01*
Monolith	APF	678.20	8.27	595.00	3.50	<0.01*
	NaF	675.70	11.15	641.00	8.49	<0.01*
	SnF_2	678.00	9.68	642.20	7.18	<0.01*

Graph 2



From the above table 2 & graph 2

Monolith zirconia shows low hardness value for 1.23% APF gel (595.00), 0.4% SnF_2 gel (642.20)& 2% NaF gel (641.00) after fluoride gel treatment when compared with same monolith zirconia samples before fluoride exposure.

Layered zirconia shows low hardness value for 1.23% APF gel (482.20), 0.4% SnF_2 gel (543.4) & 2% NaF gel (544.90) after fluoride gel treatment when compared with same layered zirconia samples before fluoride exposure

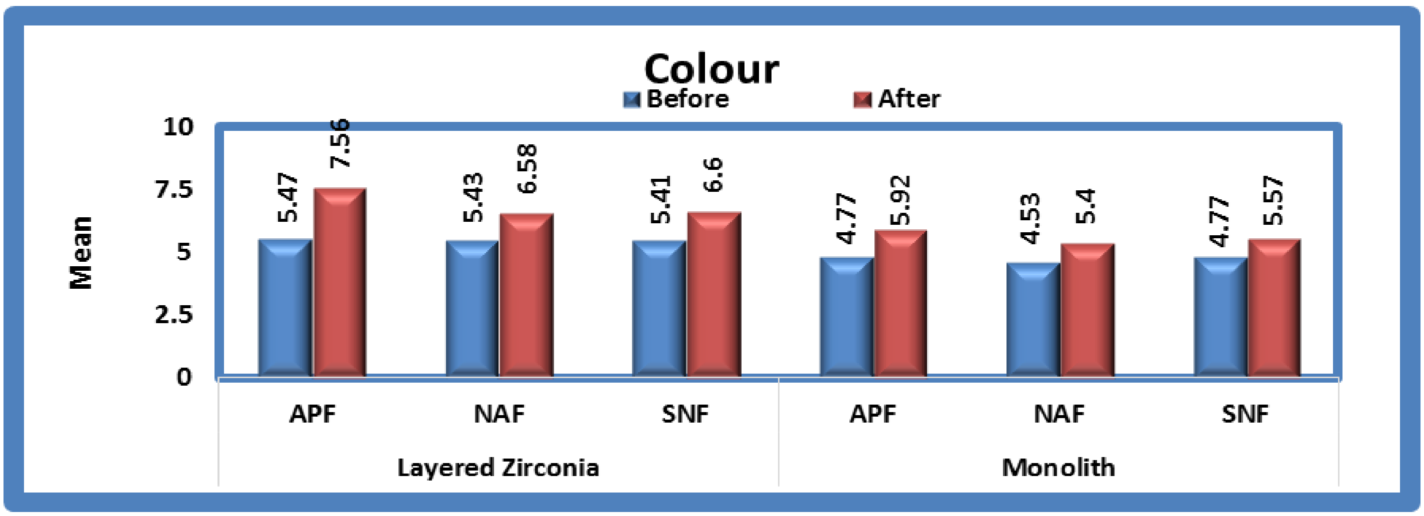
When compared between monolith zirconia and layered zirconia, layered zirconia treated with fluorides (1.23% APF gel, 0.4% Stannous fluoride gel, 2% Sodium fluoride gel) showed low hardness than monolith zirconia samples.

When compared within group 1.23% APF gel treated zirconia samples (monolith zirconia & layered zirconia) showed low hardness values.

Table 3

		Colour				P-value
		Before		After		
		Mean	SD	Mean	SD	
Layered Zirconia	APF	5.47	0.17	7.56	0.20	<0.01*
	NAF	5.43	0.36	6.58	0.23	<0.01*
	SNF	5.41	0.40	6.60	0.35	<0.01*
Monolith	APF	4.77	0.31	5.92	0.30	<0.01*
	NAF	4.53	0.26	5.40	0.23	<0.01*
	SNF	4.77	0.22	5.57	0.28	<0.01*

Graph 3



From the above table 3 & graph 3

Monolith zirconia shows higher colour change for 1.23% APF gel (5.92), 0.4% SnF_2 gel (5.57) & 2% NaF gel (5.40) after fluoride gel treatment when compared with same monolith zirconia samples before fluoride exposure

Layered zirconia shows higher colour change for 1.23% APF gel (7.56), 0.4% SnF_2 gel (6.60) & 2% NaF gel (6.58) after fluoride gel treatment when compared with same layered zirconia samples before fluoride exposure.

When compared within group 1.23% APF gel treated zirconia samples (monolith zirconia & layered zirconia) showed higher colour change.

Discussion

Ceramic materials have a tooth-like color and can be shaded to match the natural adjacent tooth. Zirconia is polycrystalline ceramics material able to withstand large stresses & provide both necessary esthetics (tooth color) and material properties required of a modern tooth restoration[11].

The surface roughness was high in APF gel treated samples due to selective ion exchange of sodium phosphate from APF when in contact with zirconia leaves fluoride ions in solution which helps in formation of hydrofluoric acid. Previously formed HF during ion exchange exacerbate the APF gel results in dissolution of silica & precipitate formation on restorative surface results and further increase the surface roughness [6,11,19,20]. The lack of silica phase in zirconia has been attributed to its improved resistance to acid etching. Hence the surface of layered zirconia was highly etched than monolith zirconia[8,12,13,14,15,16,17,18].

The reason for decrease in hardness after fluoride treatment was due to acidic environment the hydrofluoric acid in APF gel will trigger superficial surface degradation results in dissolution, degradation & precipitate formation on restorative surface thereby generating loss of mass further results in slow micro crack propagation under tensile stress. This crack propagation effect the hardness of restorative material and results in fracture [18].

The increase in L* parameter (lightness) of colour was observed. The colour change may be due to intrinsic & extrinsic factors. Due to oxidation of amine accelerators certain chemical change takes place which results in intrinsic pigmentation (lightness) of ceramics. The high physical properties in zirconia will influence colour stability when compared to feldspathic porcelain. Inspite the crystalline structure of zirconia decrease the colour change when compared to porcelain which contain more glass phase. The colour change is more in layered zirconia when compared to monolith zirconia[10, 19, 20, 21].

So, when 1.23% APF gel treated samples were compared with 2% Sodium fluoride gel & 0.4% Stannous fluoride gel treated samples there was a significant change in roughness, hardness and, colour values were observed. No marked roughness difference was observed between 2% Sodium fluoride gel & 0.4% Stannous fluoride gel treated samples.

Conclusion

It was concluded that

1. Between monolith and layered zirconia, increased surface roughness, increased colour change & decreased hardness values were obtained for layered zirconia after 1.23 % APF gel, 0.4% Stannous fluoride gel & 2% Sodium fluoride gel treatment.
2. Within layered zirconia group, 1.23% APF gel showed increased surface roughness, increased colour change & decreased hardness values followed by 0.4% Stannous Fluoride gel & 2% Sodium fluoride gel treatment. Between 0.4% Stannous Fluoride gel & 2% Sodium fluoride gel samples there was no significant change in roughness, hardness & colour.
3. Within monolith zirconia group, 1.23% APF gel showed increased surface roughness, increased colour change & decreased hardness values followed by 0.4% Stannous Fluoride gel & 2% Sodium fluoride gel treatment. Between 0.4% Stannous Fluoride gel & 2% Sodium fluoride gel samples there was no significant change in roughness, hardness & colour.
4. On interpreting the zirconia samples (both monolith & layered zirconia) before and after fluoride treatment, the specimens after 1.23% APF gel, 0.4% Stannous Fluoride gel & 2% Sodium fluoride gel treatment showed increased surface roughness, increased colour change & decreased hardness values when compared with same values before fluoride treatments.

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