ZIRCONIA RECYCLING AND EVALUATING THE EFFECT OF WET AND DRY GRINDING, WITH OR WITHOUT HEAT TREATMENT, FLEXURAL STRENGTH OF THIS CERAMIC

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ABSTRACT

Aim: The purpose of this study was to determine the possibility of zirconium recovery and the effects of dry and wet grinding and heat treatment on its flexural strength.

Methods and materials: This experimental study was carried out on zirconium recycled disks from Amann Girbach, Germany's zirconium block. In terms of preparation by dry and wet grinding methods, the use of diamond bur or zirconia specific bur with and with out heat treatment were investigated in different groups. Phase changes of samples were determined by x-ray diffraction. The strength of the discs was measured with biaxial flexural strength test was measured in the STM-250 traction and pressure test device at the blade tip speed of 1mm / min. Data were analyzed using SPSS 25 software.

Results: The zirconia waste recovery has been successful, although the flexural strength of the recycled samples is about one-third the flexural strength of the sample from the main block (Mean flexural strength of commercial samples 650 Mpa versus average flexural strength of recycled samples 250 MPa). The heat treatment before dry Grinding with zirconia specific bur improves the flexural strength of the samples. Regardless of received or not received, heat treatment was higher than diamond bur. However, there was no significant difference in terms of flexural strength of prepared samples with specific zirconia bur in dry or wet grinding and in either case, with and without heat treatment. The zirconia specific bur function was better than the diamond bur to improve flexural strength of the samples. However, the heat treatment process had no significant effect on the flexural strength of the discs grinding with diamond bur as dry or wet.

Conclusion: Preparation through various methods in most cases increases the strength of zirconium disks and a better performance of zirconium specific bur was seen compared to diamond bur. The results of this research can be used in clinical conditions and in order to increase the clinical success of zirconia restorations.

Key words: Flexural strength, Wet and dry grinding, Diamond bur, Zirconia bur, heat treatment, Recycled zirconia

Introduction

One of the most important challenges in using zirconia in the clinic is high cost due to the large unused volume of zirconia block after Turning by CAD / CAM is one of the challenges.[1] Of course, newer devices have higher accuracy in cutting. But in the process of cutting the block, there are still large parts in the form of powders and unused patches that have imposed a high cost on the patient and also it has problems in its decomposition and recycling in the environment.[2] Also, there are no specific guidelines in some countries on recycling and disposal of zirconium remnants. There are different reports on the effects of abrasion and heat treatment on the values of flexural strength of zirconia, and some studies have shown that the grinding in the flexural strength is unaffected and in some studies, the negative effects of wear on flexural strength and increased strength after heat treatment are mentioned.[3,4] It has been argued that presence in damp environments and at relatively low temperatures can change the Tetragonal (T) constant zirconium into the monoclinic (M) phase at the surface of the samples.[5] This phenomenon is related to the incomplete stability of the tetragonal phase at room temperature, causing small and coarse cracks, a decrease in strength and the loss of mechanical properties of zirconia.[6-8] The heat produced during the abrasion process should be taken into account, since abrasion under conditions of use of the cooling agent causes the transformation of the tetragonal phase to the monoclinic phase and also increases the compressive strength of the zirconia surface.[9] Passos *et al*. (2015); In a study, the flexural strength and fracture behavior of ceramic with zirconium-based ceramics were evaluated following preparation by heat treatment before and after the synthesizer process. The results of their study showed that phase transition from T to M was evident after sandblasting and phase transfer from M to T after heat treatment. Sandblasting with 30-micron SiO_2 particles and 50-micron Al_2O_3 particles resulted in a limited phase change, but the application of 30-micrometers of SiO_2 particles had better results.[10] Another study by Vatali *et al*. (2014) examined the effects of heat treatment after veneer application and the aging process on mechanical properties and microstructure of zirconia. The results of this study showed that the clinical performance of zirconium dental ceramics may be affected during baking and aging processes and may increase their chances of failure.[11] Zucuni *et al*. (2017); in a study of the effects of different preparation methods (polishing, heat treatment, glaze, polishing + heat treatment, polishing + glase) in surface properties (morphology and violence), phase transfer and fatigue strength of zirconium ceramics determined after abrasion with diamond bur. In part of their results, they showed that heat treatment had limited effects on Levels of superficial violence, resulting in the complete reversal of phase m to phase t (without increasing the fatigue performance of samples).[12] There are various reports about the effects of grinding and heat treatment on the values of the zirconia flexural strength and, on the other hand, there is no specific guidelines on how to recover and remove the zirconia residues, therefore, the aim of this study was to investigate the possibility of recycling zirconia wastes and the effect of dry and wet grinding and heat treatment on its flexural strength.

Materials and Methods

In this Experimental and laboratory study, 135 disks of recycled zirconium were prepared and after sintering using various grinding methods of zirconia specific bur and diamond bur in dry and wet conditions, along with and without heat treatment. Finally, their flexural strength was measured and recorded in MPa. 15 disc-shaped samples were prepared by the CAD-CAM machine from the main zirconia block with similar dimensions of recycled samples to examine the flexural strength difference. The selection of samples in the research was non-randomly easy and, taking into account the type-1 error, was 0.05 ($\alpha = 0.05$), the second type error was 0.2 ($\beta = 0.2$). But what samples were prepared using a special method (special zirconia and diamond bur, with or with out heat treatment) was randomly determined. The number of samples following the evaluation of studies in the research records (1) was considered as 135, containing 9 subgroups of 15 samples.

- Group 1 Control: Recycled zirconia disc without grinding and heat treatment
- Group 2: Recycled zirconia disc with dry grinding specific zirconia bur without heat treatment
- Group 3: Recycled zirconium disk with dry grinding diamond bur 80-micron without heat treatment
- Group 4: Recycled zirconium disk with dry grinding specific zirconia bur with heat treatment
- Group 5: Recycled zirconium disk with dry grinding diamond bur 80-micron with heat treatment
- Group 6: Recycled zirconia disc with wet grinding specific zirconia bur without heat treatment
- Group 7: Recycled zirconium disk with wet grinding diamond bur 80-micron without heat treatment
- Group 8: Recycled zirconia disc with wet grinding specific zirconia bur with heat treatment
- Group 9: Recycled zirconium disk with wet grinding diamond bur 80-micron with heat treatment

Commercial Zirconium Group: Disc without grinding and heat treatment

To create 135 samples of recycled zirconia, unused remnants of abrasion zirconia blocks were collected and turned into zirconium powders by special zirconium mortar. Then, using the threshing method, the zirconia particles were separated by measurements in the range of micrometer and smaller. The specified amount of weight of the powder was deposited in a prepared metal generative with a diameter of 18 mm and pressed in one direction under the force of bar50 presses then the discs with a diameter of 18 mm and a thickness of 1.6 mm were obtained. The samples were sintered in a furnace with a temperature of 1360 ° C for 2 hours and their final size was 14 mm in diameter and 1.4 mm in thickness. A total of 120 specimens were prepared using this method, and after the abrasion on mm0.1 on each side, their final thickness remained at 1.2 mm.15 control samples were prepared so that their thickness after the synthesizer was 1.2. Also, 15 samples of the same size with commercially prepared and non- synthesized blocks of zirconia were prepared and by a cutting machine (Nemo, Iran) at 90 microns per second, they were cut to a thickness of 1.6 mm and sintered at 1360° C for 2 hours. [Figure 1]

Figure 1: Sinter less cut out zirconia sample from the main block

A total of 30 samples were recycled using a zirconia grinding specific turbine bur in a dry form and 30 samples were eroded using a diamond bur in a dry form and their final thickness remained at 1.2 mm. 30 specimens eroded using a special grinding zirconia bur in a wet form and 30 specimens eroded using a 80-micron diamond bur in a wet form. Width and thickness of each specimen were measured by digital caliper and with a precision of ± 0.01. Performing this process was necessary, as it was likely that the original sample size would be changed during the grinding phase and delete the inappropriate samples. Afterwards, half of the specimens were heat treated at 1200 ° C for 2 hours. In addition, 15 samples of recycled zirconium specimens as a control group, they were not found to grinding, nor they were under the heat treatment process.

In Figure 2, studied subgroups in this study are shown.

Figure 2: Studied Subgroups.

To evaluate the phase changes, from each of the studied groups, 1 sample was randomly selected for X-ray diffraction (XRD) test. The residual flexural strength values of the samples were measured using a biaxial flexural strength test method in a STM-250 stretching and pressure test device manufactured by Centaur Company of Iran with a blade tip speed of 1mm / min. For this purpose, the discs were placed on 3 ball of 3 mm in diameter at 3 angles of 120 mm apart on a circle with a radius of 10 mm. Disks, were fixed from a position of force by 3M tape. A cylindrical piston with a smooth surface, with a diameter of 1.4 mm, with a blade tip speed of 1 mm / min, was used to apply force. Inserting forces continued until the specimens were defeated. Using the tape, the broken pieces were held together and caused the piston contact with the sample to be improved. [Figure 3]

Figure 3: Piston contact with the sample.

Biaxial flexural strength values were calculated according to the standard ISO 6872.2008 using specific formulas:

$S = -0.238P(X-Y) / d^2$ [ISO 6872]
S: Maximum flexural force in place
P: breaking force in Newton
d: Disc thickness in mm

X=(1+V)In(r2/r3)2+[(1-v)/2](r2/r3)2
Y=(1+v) [1=In (r1/r3)2]+(1-v)(r1/r3)2
V: The Poisson ratio is approximately 0.25
R1: The radius of the retaining circle in mm
R2: radius of force in mm
R3: sample radius based on mm

SPSS (Statistical Package for Social Sciences) version 25.0 was used to analyze the data. For this purpose, the mean and standard deviation of commercial and recycled zirconium disks were calculated and reported using different preparation methods (wet and dry grinding with diamond bur and specially for zirconia in terms of with and with out heat treatment).

Results

Based on the findings of this study, limited strength values were recorded in recycled zirconia samples compared to commercial zirconium samples, so that the flexural strengths obtained in recycled zirconium samples were about one-third of commercial zirconium (average flexural strength of commercial samples 650 Mpa versus average flexural strength of recycled samples 250 MPa). According to XRD analysis, the dominant phase of recycled samples is monoclinic.

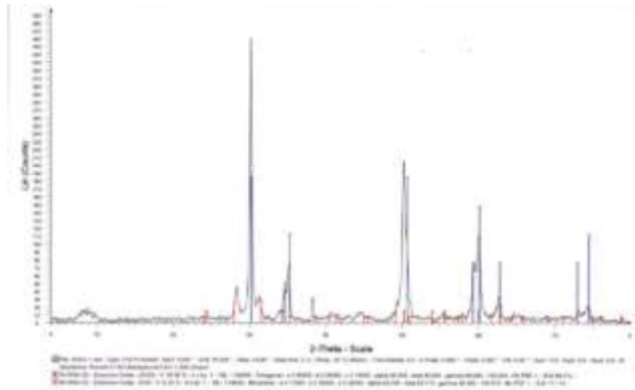

Figure 4: XRD analysis, the dominant phase of recycle samples is monoclinic.

In the diamond bur dry grinding method and zirconia specific bur, the average flexural strength of the zirconium was evaluated with and without heat treatment. It was observed that in recycled zirconia in the dry grinding method both zirconia specific bur and dimond bur, heat treatment did not cause any changes in the values of flexural strength (0.05 <p). [Table 1]

Group	Average Flexural Strength Without Thermal Correction	Average Flexural Strength With Thermal Correction	p Value
Recycled zirconia with special Dry milling	233.99 ± 41.75	254.24 ± 33.94	0.06
Recycled zirconia with Dry Milling	186.49 ± 23.71	205.61 ± 37.50	0 91

Table 1: The mean and standard deviation of the zirconia flexural strength in the dry grinding in both zirconia specific bur and diamond bur, with and without heat treatment.

Table 2 shows the mean values of zirconia flexural strength in the wet grinding in both zirconia specific bur and diamond bur, with and without heat treatment. The results showed that in recycled zirconia using wet abrasion in both zirconia specific bur and diamond bur, heat treatment did not make any changes in the flexural strength of the samples (0.05 <p).

Group	Average Flexural Strength Without Thermal Correction	Average Flexural Strength With Thermal Correction	p Value
Recycled zirconia with special Wet Milling	228.54 ± 30.64	256.20 ± 45.45	0.06
Recycled zirconia with Wet Milling	200.64 ± 30.74	201.78 ± 27.52	0.91

Table 2: Mean and standard deviation of zirconia flexural strength with wet grinding in both zirconia specific bur and diamond bur, with and without heat treatment.

Table 3 shows the mean values of zirconia flexural strength in preparation by wet and drygrinding with diamond and zirconia bur.

Based on the results of this table and according to the results of t-test, Performing the heat treatment process following dry zirconia grinding will improve the flexural strength of the samples. Also, the flexural strength of prepared samples in wetgrinding with zirconia specific bur, regardless to received or not heat treatment, was higher than that of diamond bur method. However, there was no significant difference in the flexural strength values of prepared samples with zirconia specific bur in wet or dry or in both conditions with and without heat treatment.

Group	Number	Type of Milling	Thermal Correction	Standard Deviation	Mean	p value
Recycled zirconium by dry abrasion	15	Diamond	Without Correction	41.75	233.99	0.001
	15	Zirconia		23.71	186.49	
	15	Diamond	With Correction	33.94	254.25	0.001
	15	Zirconia		37.5	205.61	
Recycled zirconium by wet abrasion	15	Diamond	Without Correction	30.64	228.55	0.01
	15	Zirconia		30.74	200.65	
	15	Diamond	With Correction	45.46	256.2	0.001
	15	Zirconia		27.53	201.79	

Figure 3: Mean and standard deviation of zirconia flexural strength in wet and dry preparation with diamond and zirconia bur by heat treatment in terms of Mpa.

The results of t test showed that the thermal correction process had no significant effect on the flexural strength of the samples prepared by dry or wet grinding with diamond bur. [Table 4]

Group	Number	Thermal Correction	Milling Type	SD	Mean	p Value
Recycled Zirconia	15	No Correction	Dry	23.71	186.49	0.16
	15		Wet	30.74	200.65	
	15	With Correction	Dry	37.5	205.61	0.75
	15		Wet	27.53	201.79	

Figure 4: Mean and standard deviation of the zirconia flexural strength in the preparation by dry or wet grinding with diamond bur in Mpa.

In Table 5, the mean and standard deviation of the zirconia flexural strength, prepared separately, with different methods provided receiving or not the heat treatment compared to the control group.

Group	Number	Case/Control	SD	Mean	p Value
Dry turning, with Zirconia milling without thermal correction	15	Case	41.75	233.99	0.04
	15	Control	27.27	206.99	
Dry turning, with Diamond milling without thermal correction	15	Case	23.71	186.49	0.03
	15	Control	27.27	206.99	
Wet turning, with Zirconia milling without thermal correction	15	Case	30.64	228.55	0.05
	15	Control	27.27	206.99	
Wet turning, with Diamond milling without thermal correction	15	Case	30.74	200.65	0.55
	15	Control	27.27	206.99	
Dry Turning, with Zirconia fuse with thermal modification	15	Case	33.94	254.25	< 0.001
	15	Control	27.27	206.99	
Dry Turning, with Diamond Cutting with thermal modification	15	Case	37.5	205.61	0.9
	15	Control	27.27	206.99	
Wet Turning, with Zirconia flask with thermal modification	15	Case	45.46	256.2	0.002
	15	Control	27.27	206.99	
Wet Turning, with Diamond Cutting with thermal modification	15	Case	27.53	201.79	0.6
	15	Control	27.27	206.99	

Figure 5: Mean and standard deviation of zirconia flexural strength, by separation of preparations with different methods and receiving or not receiving heat treatment, in experimental and control groups, in samples of zirconia recovered in terms of Mpa.

Discussion

Strength is one of the most important parameters for the clinical success of dental restorations.[13] In this study, the Biaxial Flexural Strength Method was used to determine the strength of recycled zirconia samples in dry and wet grinding with diamond bur and zirconia specially bur in both with and without heat treatment conditions. Due to the fact that during the abrasion of commercial blocks of zirconia, some of it remains unused, and in addition to extra costs, it also causes environmental pollution, therefore, in the present study, the possibility of using these wastes in recycled form in dental restorations was evaluated as disk. On the other hand, x-ray diffraction methods were used to determine the presence and extent of tetragonal zirconia phases and biaxial bending strength calculations were used to determine mechanical properties. According to the results of this study, the flexural strengths obtained in recycled zirconium samples were about one-third of commercial zirconium. According to XRD analysis, the dominant phase is the monoclonal recycled sample, which is likely to change the process of powder preparation for compression and preparation of the disc has caused this change. Perhaps the nanoscale particle size fragmentation can be the result of this process, so it will be useful to do research with particles larger than microns. Based on this, perhaps the use of recycled zirconium cannot have enough commercial zirconium strength for use in dental restorations and it seems that there is a need for more research and more advanced methods. Preparation in dry and wet grinding with diamond bur and special zirconia bur in terms of receiving and not receiving heat treatment improved the values of flexural strength of zirconia in comparison with the control group, which was also significant in most cases. It seems that these increases are due to the removal of the deep gaps and the uniformity of the surface of the zirconia discs after the abrasion, and also due to the increased hardness of the zirconia fracture due to the transfer of the phase from t to m.[14] Despite some research, the effects of abrasion on the zirconia flexural strength values have not yet been determined. Some factors such as surface violence, plastic deformation and the percentage of transported zirconia are effective in this regard. The recent factor also depends on the change in phase T to M, Dependent on abrasion rate and ambient temperature.[15-17] In our study, the use of diamond and specific zirconia bur in zirconia samples recovered under receiving and not receiving heat treatment conditions in most cases resulted in increased zirconia hardness values compared to controlled samples. Of course, in some cases, preparation by dry and wet grinding with diamond bur in with and without heat treatment reduced the values of flexural strength of zirconia in comparison with the control group. Botelho *et al*. (2018) investigated the effects of preparation techniques on the biaxial flexural strength of zirconium materials, and showed that abrasion with diamond bur had no effect on zirconia weakening.[18] That is not consistent with the findings of our study. Considering some cases, reducing the zirconia flexural strength in the application of diamond bur than the control group, it may be assumed that zirconia strength is similar to other dental ceramics affected by surface roughness. Curtis *et al*. (2006) showed that the use of rough diamond bur in zirconia significantly reduced its strength, but the grinding with softened bur had no effect on its strengths relative to control specimens.[19] Our study also showed that in wet and dry grinding methods, with special zirconium bur, heat treatment increased the flexural strength of the samples. In wet grinding with diamond bur, heat treatment also had no significant effect on the increased flexural strength of recycled zirconia discs. Accordingly, in grinding with a special zirconia bur, heat treatment has a definite effect on the improvement of flexural strength, but in the use of diamond bur, these effects were either reversed or not very significant. On the other hand, the changes made in the flexural strength values of recovered zirconium disks in conditions with and without heat treatment have not been remarkable.

In a study by Hjerpet *et al*. (2016), abrasion and air conditioning with silicon carbide papers had a significant effect on the flexural strength of the samples which illustrates the effectiveness of the surface preparation methods of zirconia samples in the strength and superficiality of the material of the zirconia framework material.[20] In our study, according to the manufacturer's guideline, the heat treatment was carried out at 1200 ° C for 2 hours, which may be a reason for the effectiveness of heat treatment on the flexural strength of the samples.

In the study, thermal modification at 930 ° C for 1 minute and at 910 ° C for 1 minute after the preparation by means of air conditioning, turning and polishing resulted in a decrease in the zirconium flexural strength values. According to researchers argue, thermal correction by reverse phase transfer leads to a decrease in the monoclinic phase. Zirconia is not stable over time, and the moisture and heat of the oral environment can cause it to degrade at low temperatures.[21] Guazzato *et al*. (2005) Preparation methods by air conditioning and lacquering can also increase zirconium strength in conditions without heat treatment, while polishing, removes the stresses layer of zirconia and reduces its flexural strength.[22] In the present study, diamond bur of a mean size of 80 microns were used. The reinforcement effects of the abrasion process depend on the percentage volume of modified zirconia in the depth of the surface compression layer and the increase in the thermodynamic stability of tetragonal zirconia grains and the Turning intensity are also effective in this field.[23] According to the results of our study, the process of heat treatment following zirconia dry grinding, regardless of the type of bur (diamond or special zirconia), improved the flexural strength of the samples. In addition, the flexural strength of the samples in wet grinding with special zirconia bur, regardless of receiving or not receiving heat treatment, was higher than that of diamond bur method. Also, there were no significant differences in the flexural strength values of samples prepared with special zirconia bur in dry or wet conditions and in both cases with and without heat treatment.

Conclusion

Overall, it seems that preparation through different methods in most cases increased the strength of zirconium discs and improved performance of zirconia bur compared to diamond bur. The results of this research can be used in clinical conditions and in order to increase the clinical success of zirconia restorations. However, in the recycling category, we need further studies with different particle sizes and different presses of the resulting powders to reinforce the flexural strength.

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