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RESEARCH ARTICLE

Investigation of mechanical stability of lithium disilicate ceramic reinforced with titanium nanoparticles

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ABSTRACT

The use of lithium disilicate (LD) glass-ceramic in dentistry is increasing due to its proper mechanical properties and aesthetic appearance. On the other hand, due to the forces acting on dental restorations, materials with mechanical properties as well as suitable tooth color are very important. Titanium nanoparticles (Ti-NP) as a material with suitable mechanical properties can be a used for the production of dental ceramics with higher strength by combining with the LD. The aim of this study was to determine the compressive strength of LD ceramics reinforced with Ti-NP in comparison with conventional LD. This experimental study was performed on 60 LD ceramic blockss containing the desired nanoparticles, Ti-NP with an average particle size of 50-70 nm with the content of 1 wt%, 2 wt% and 5 wt% were pressed in a stainless-steel mold under force of 200-250 MPa and then sintered in the electric furnace. The highest compressive strength of the standard sample groups compared to the commercial LD brand Emax (449±42.920 MPa) was for samples with 5 wt% Ti-NP (341.67 MPa ($p < 0.001$). The addition of 2 wt% and 5 wt% Ti-NP increased the compressive strength of LD ceramic compared to the control group but decreased significantly compared to the conventional commercial sample. The addition of 5 wt% Ti-NP increased the compressive strength of LD ceramic compared to the control group.

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INTRODUCTION

The use of lithium disilicate (LD) glass-ceramics in dentistry is increasing and the fabrication and synthesis of this material is very valuable due to its special mechanical and aesthetic properties and the complexities of its fabrication [1-4]. On the other hand, due to the forces acting on dental restorations, materials with mechanical properties as well as suitable tooth color are very important. Titanium nanoparticles (Ti-NPs) as a material with suitable mechanical properties that is also used in dental veneers as a new material for making dental ceramics with higher strength by combining with the synthesis of LD [5-7]. Today, ceramic materials are widely used in dentistry due to their beauty and good biocompatibility [8-11]. Different types of ceramic systems are available to dentists, which are divided into several categories according to the manufacturing process including pressable [12], slip-casting [13], milling [14] and sintering [12-18]. Also, according to their chemical composition, ceramic materials can be divided into three categories such as glass matrix ceramics, polycrystalline ceramics and matrix resin ceramics [19-21]. As a general rule, aesthetic properties are provided by the glass matrix phase, and the crystals improve their mechanical properties. Therefore, the aesthetic properties of glass matrix ceramics are better than polycrystalline ceramics, but the mechanical properties such as compressive and flexural strength of these ceramics are less [22-28].

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Recently, glass-ceramic systems have received a lot of attention due to their beauty and good mechanical properties. Prior to 1990s, lucite-reinforced glass-ceramic was marketed under the brand name IPS-Empress Lichtenstein (Vivadent and Ivoclar). These materials have the same wear resistance and abrasion properties as natural teeth, but due to their low flexural strength (112±10 MPa), they are not suitable for use in dental bridges [29-35]. The LD glass ceramic was manufactured by the same manufacturer, which includes quartz, LD, phosphorus, alumina and potassium oxide. The IPS E.max Press was introduced as a castable LD glass which is available in block form and LD crystals in its composition prevent the spread of cracks and cause more translucency and better visual and mechanical properties [36-42]. In terms of mechanical properties, LD ceramics are brittle materials and are sensitive to bending forces. Proper mechanical strength assessment is an important mechanical property in evaluating the performance of brittle materials and flexural strength of ceramic materials is one of the most important factors in the success of fixed dental restorations [43-51].

These ceramics have a medium flexural strength (between 400 and 600 MPa) as well as a medium fracture toughness (between 2 and 5.5 MPa. $m^{1/2}$) and can only be used as single-posterior crowns or anterior 3-unit bridges. Very few studies have been performed on the addition of Ti-NP to dental LD ceramics. In the present study, Ti-NP are added to LD ceramics and the effect of these nanoparticles on compressive strength can be evaluated.

The hypothesis can be that Ti-NP have no effect on the compressive strenth of LD ceramics. Considering the advantages of Ti-NP such as high hardness, strength and also considering the positive effect of these nanoparticles in studies on other biomaterials, we evaluated the effect of addition of Ti-NP on LD ceramics. Determination of compressive strength of LD ceramics reinforced with Ti-NP in comparison with commercial LD using X-ray diffraction (XRD) and scanning electron microscope (SEM) analysis.

MATERIALS AND METHOD

The present study was performed as an experimental work in the dental materials research center of Isfahan University of Technology (IUT) in 2020-2022. The data were entered by observation and measurement in a checklist prepared by the authors and the statistical categories of this study included 60 LD ceramics. For mechanical tests (compressive strength) with 12 samples from each group for each test according to the dimensions mentioned in the method and for physical tests was used and also one sample from each group was prepared as control group.

Preparation of test samples

Lithium disilicate ceramic as an Ingot from e.max press (Ivoclar-Vivadent) with shade A1 was considered for this study. In this work, after making a wax pattern in standard dimensions required for compressive strength test (6 × 12 × 12 mm) cylinder for compressive strength analysis by heat and pressing method in the electrical furnace (Programs EP 3010, Ivoclar Vivadent).

The samples were prepared at 900°C. Table 1 shows the main composition of this commercial sample. Fig. 1 shows the emax commercial Ingot for comparison with experimental samples.

In order to prepare ceramics containing the desired Ti-NP as a raw material with a combination of silica powder, calcium carbonate, alumina, zirconia oxide, lithium carbonate and phosphorus pentoxide were used as germination agents according to the Table 2.

Ti-NP with an average particle size of 50-70 nm with hexagonal crystal structure without geometric shape, with a tendency to spherical shape, 98% purity (Purchased from Sigma Aldrich, USA) were prepared. Then, 0 wt% (Sample 1=S1) 1 wt% (S2), 2 wt% (S3) and 5 wt% (S4) were added by digital scale (AC Adapter, Japan) with an accuracy of 0.0001 weight. The materials were ground using automatic mixer and pressed with a pressure and placed inside alumina mold. They were heated in an electrical furnace for 2 hours at 800-900°C to form a homogeneous combination. After the sintering process, to prevent heat shock, it was immediately transferred to the sinter at 450°C and left at this temperature for 2 hours. After this step, by turning off the furnace, the glass blocks inside

Table 1. Compositions and specifications of LD ceramic e.max press (Ivoclar-Vivadent)

Material	Batch	Manufacturer	Composition
IPS e.max press	HT A1/C 14 REF	Ivoclar Vivadent, Schaan, Liechtenstein	SiO_2, Li_2O, K_2O, MgO, ZnO, Al_2O_3, P_2O_5

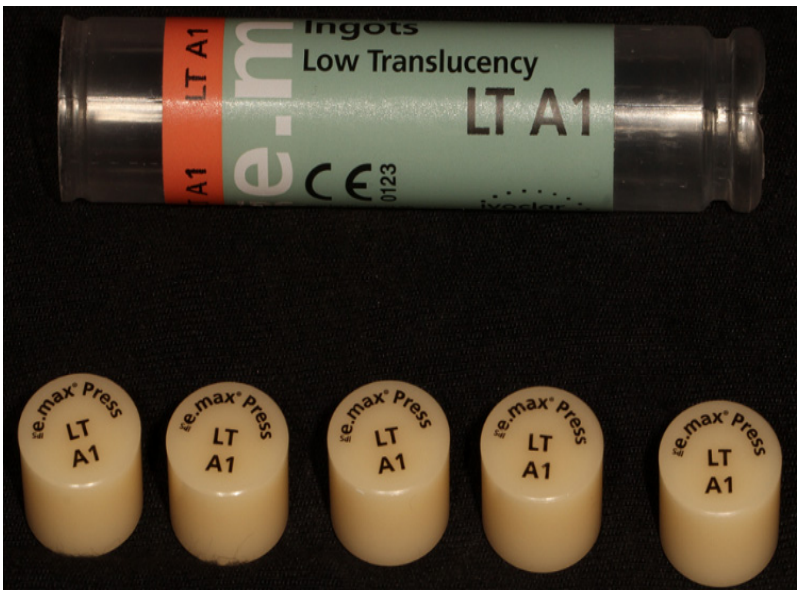

Fig. 1. Commercial samples of S5 LD (Ivoclar-Vivadent Ingot emax)

Table 2. Compositions of the studied groups

Groups	LD composition	Ti-NP (wt%)
Group 1	100	0.05
Group 2	99	0.01
Group 3	98	0.02
Group 4	95	0.05

the furnace were cooled till to room temperature, and thus 5 groups were prepared for the following experiments.

Physical experiments

Physical examinations for the samples, including Scanning electron microscomp (SEM) and X-ray diffraction (XRD) for morphological and phase structure analysis was performed at Isfahan University of Technology, Materials Research Center.

XRD analysis

Phase characterization study by XRD was used to study and determine the crystals size of the powders. The XRD pattern used for phase analysis and to study the grain size and particle size of nanomaterials. This is possible through the processing and analysis of X-rays returning from the sample surface. Phase analysis was performed by Philips PW1730 and the data were displayed by X pert software.

Phase of crystals by X-ray powder diffraction (XRD; D2-Phaser, Bruker AXS, Germany) with Cu Kα radiation (λ = 1.5418 Å) operating at 30 kV and 10 mA were identified. Data were recorded above 2θ and in the range of 10-90° with a step increase of 0.02° and a time interval of 0.1 seconds in each step.

SEM analysis

The prepared four samples as Ingot were coated with Au and then using scanning electron microscopy (SEM) analysis used to investigate the morphological changes of the powders and samples via LEO Netherlands device.

Mechanical tests

The mechanical tests considered for the

specimens, including compressive strength were performed with the following structure. To perform this test, 12 samples of Ingot (cylinders) with a length of 6 mm and a diameter of 12 mm were fabricated. The samples are then compressed under load by Uinversal Testing Machin (UTM) (KOOPA, Iran) with a crosshead speed of 0.5 mm/minute and the force required for failure is recorded and after being placed in the following formula (ISO-1992) the compressive strength (MPa) was calculated and reported using following Equation 1.

Compressive strength (CS) MPa = Force (F)/ Area (A for cylindrical shape). (Eq. 1)

F is the load at the breaking point (N) and A is the cross section of the cylindrical specimen (mm).

Ethical considerations

In this study, the following ethical considerations were considered. Before starting the research, the researcher registered the title of her research in the university and obtained the necessary license in this field. This research with the ethics code IR.IAU.KHUISF REC.1399.230 was registered on 2021. In this study, honesty and trustworthiness in using scientific sources were observed. This study examined 12 samples in each group and the aim is to determine the compressive strength of LD ceramic reinforced with Ti-NP compared to conventional LD in dentistry application. Because the sample size in this study is 12, non-parametric tests were used throughout this dissertation.

RESULTS AND DISSCUSION

According to XRD pattern, the amorphous structure was observed in S1, S2 and S3, which is glassy and amourphous structure. The presence of sharp peaks indicated high crystallization, in which crystallization was a done with high purity of the particles. The spherical and regularly dispersion of Ti-NP in the LD can be seen in the XRD pattern. The closest specimen to the e maz was the sample 3. As the Ti-NP content increases from the S1 to the S4 specimen, the peaks observed in the XRD pattern become denser and their dispersion decreases, which increases the mechanical properties and increases the chemical stability as shown in Fig. 2. With increasing Ti-NP content in S3 sample, chemical and mechanical stability has increased. In samples S1, S2 and S4 the peaks are more scattered and sample S4 was most similar to the sample e max and in sample S2 a peak is seen at an angle of 65° as can be seen in Fig. 2. The sample S1, the Ti-NP were dispersed needle-shaped and with increasing concentration of Ti-NP, particle dispersion increased and particle size decreased. With the addition of Ti-NP, the amount

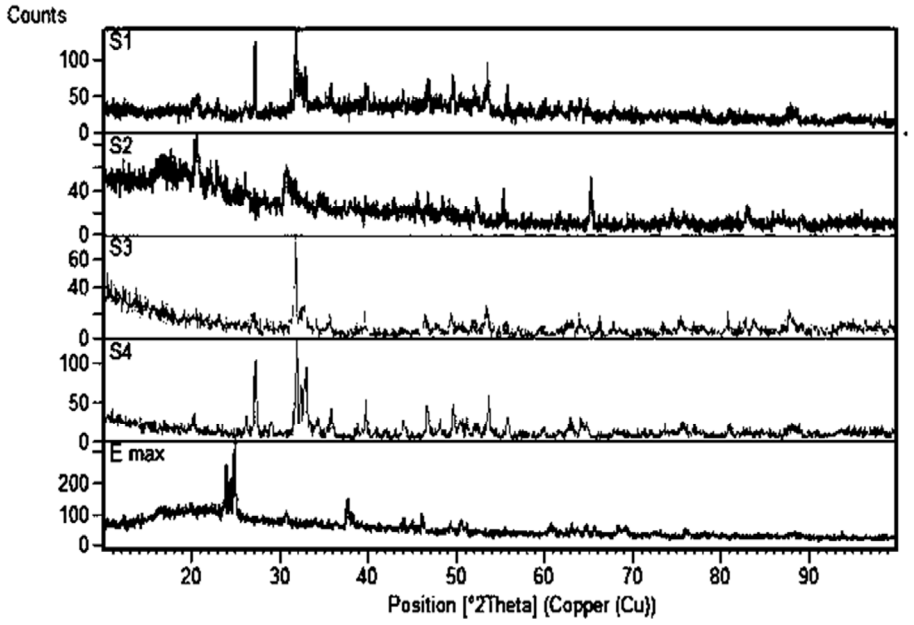

Fig. 2. XRD pattern of powders S1: lithium disilicate (control group), S2: lithium disilicate reinforced with 1 wt% Ti-NP, S3: lithium disilicate reinforced with 2 wt% Ti-NP, S4: lithium disilicate reinforced with 5 wt% Ti-NP, and S5: commercial lithium disilicate

of crystallization increased in the matrix and the amount of porosity decreased, and in the S4 sample the amount of porosity is much less than in the S1 sample. The SEM images shows the S1 specimens of the LD have needle crystals and the germination of the crystals is visible as shown in Fig. 3 (a-d). This specimen had relatively high porosity, which reduced its mechanical strength.

Fig. 4 shows the commercial lithium disilicate compared with the synthesized powders in this study.

The SEM images show the nanoparticles of the ceramic component and the morphology was quasi-spherical with the particle size less than 80-50 nm. SEM images shows that the nanoparticles tend to stick together and create agglomeration due to the decrease in surface energy of the particles, which reduces the mechanical strength of the microstructure as shown in Fig. 3 (a-d) and Fig. 4.

The highest average compressive strength in the standard commercial groups of e max standard (449.42 ± 10 MPa), 5 wt% Ti-NP (341.67 ± 10 MPa), 2 wt% Ti-NP (83 ± 10.573 MPa), 1 wt% Ti-NP (258.92 ± 5.5 MPa) and control (211.83 ± 5.55 MPa) were observed. At 95% confidence level, Kruskal-Wallis test was significant ($p<0.001$) and the mean compressive strength was significantly different in the five groups. Many researhcers used Ti-NP in combination with organosilane allyltriethoxysilane (ATES) to improve the mechanical properties of composite dental resins (RBCs, Z100, 3M ESPE). The Ti-NP were dissolved in ethanol solution containing ATES. The modified nanoparticles were washed with pure ethanol and dried before use as a filler. Flexural strength helps with fracture resistance, surface abrasion resistance, as well as creating an excellent surface to prevent bacterial bonding and wear of opposite teeth. In recent years, metal oxide nanoparticles have been extensively studied due to their antimicrobial properties. In particular, Ti-NP is now considered a low-cost, clean photocatalyst with chemical and non-toxic stability and is used for a wide range of environmental applications, including water

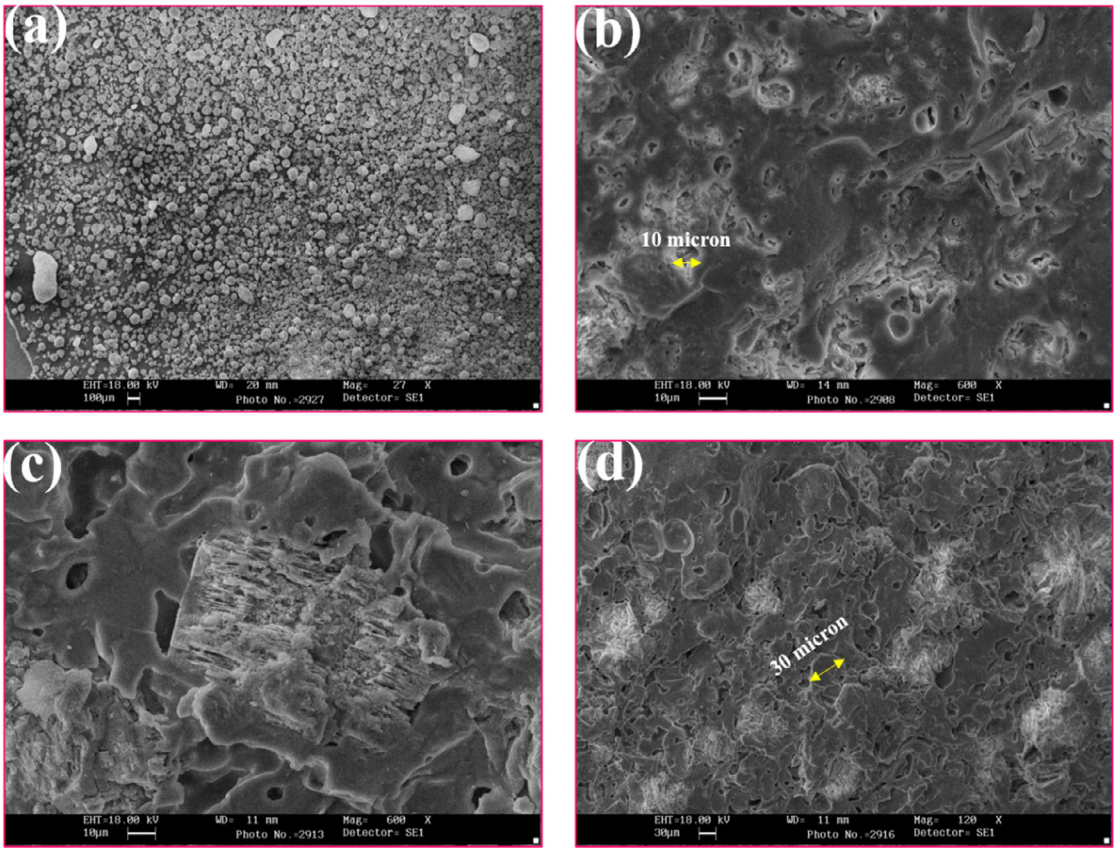

Fig. 3. SEM images of powders S1: lithium disilicate (control group), S2: lithium disilicate reinforced with 1 wt% Ti-NP, S3: lithium disilicate reinforced with 2 wt% Ti-NP, and S4: lithium disilicate reinforced with 5 wt% Ti-NP

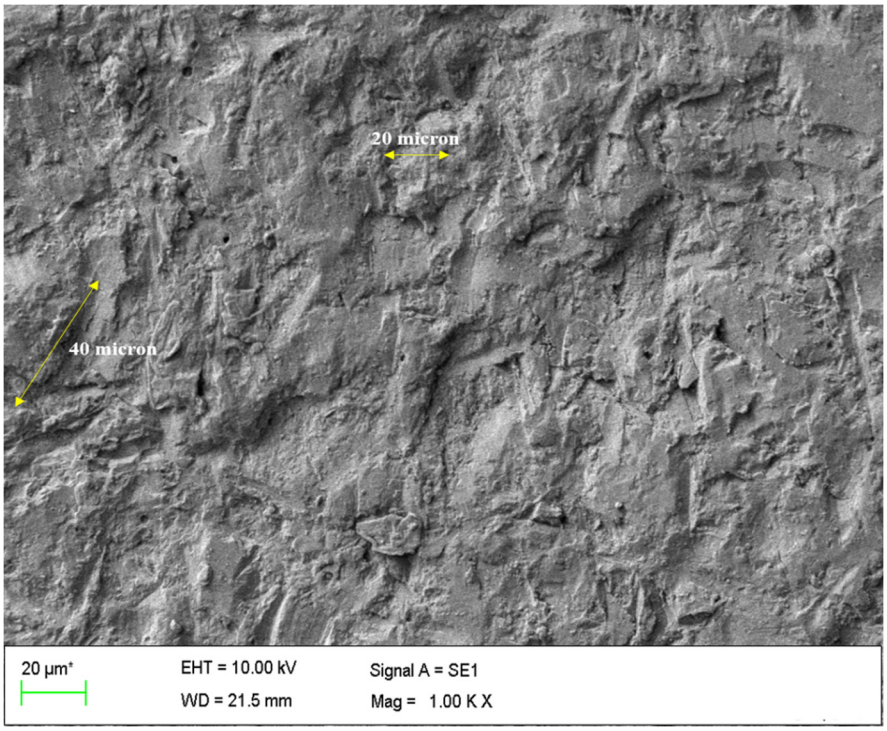

Fig. 4. SEM images of powder S5: commercial lithium disilicate

treatment and air treatment. Ti-NP have excellent and inexpensive mechanical properties, and titanium is the most abundant metal on earth after aluminum, iron, and magnesium. On the other hand, improving the strength of the restoration can increase its applications and can be very cost-effective. The aim of this study was to determine the compressive strength of LD ceramic reinforced with Ti-NP (0 wt%, 1 wt%, 2 wt% and 5 wt%) in comparison with conventional lithium di silicate. Compressive strength indicates the resistance of the material to vertical static loads. The results showed that the addition of Ti-NP (2 wt% and 5 wt%) to LD ceramics significantly increased the average compressive strength compared to the control group. However, it was significantly less robust than commercial LD. These results may be attributed to the dispersion of Ti-NP nanoparticles in the LD ceramic matrix, which negatively affects the degree of conversion, which in turn leads to an increase in the level of the unreacted monomer, which acts as a plastic deformation. Therefore, the content of added nanoparticles is of vital importance. On the other hand, in XRD images, as the amount of Ti-NP increased from S1 to S4, the peaks observed in the XRD diagram became denser and their dispersion decreased, which can increase the mechanical properties and increase the chemical stability. Mohsen et al. [32] added silver nanoparticles to porcelain and found that porcelain modified with silver nanoparticles showed higher fracture toughness compared to the control group. Many researchers study indicate that the addition of silver nanoparticles with percentages (0.0 wt%, 0.5 wt%, 1 wt% and 3 wt%) increases the fracture toughness of alumina ceramics. Ahmed et al. [33] reported that the addition of 1 wt% Ti-NP nanoparticles increased the compressive strength of acrylic resin, normal heat cure acrylic resin and high impact acrylic resin. The final flexural strength of a material indicates its potential for resistance to catastrophic failure under flexural load. The results of this study showed that the addition of 5 wt% Ti-NP increased the compressive strength of LD ceramic compared to the control group. Also, due to the fact that according to SEM images, fewer particles were scattered in a needle-shaped manner and the pore state and porosity were less, more strength can be justified. Ahmed et al. [33] reported that the addition of 1 wt% Ti-NP increased the flexural strength of an acrylic resin normal heat cure acrylic resin and high impact acrylic resin but

increased the flexural strength by increasing the concentration of Ti-NP to 5 wt%. Shirkavand et al. [34] added 1% Ti-NP to acrylic dental resins and observed an increase in tensile strength. Strength assessment is an important mechanical property in evaluating the performance of brittle materials. This mechanical test is more clinically appropriate because the dental restorative materials used inside the mouth are more similar to the force exerted by three-point bending test conditions [52-58]. The role of saliva was not considered in this study as biological investigation. In addition, the oral cavity is a different clinical setting. For example, the presence of water, changes in temperature and pH level in the oral cavity may also significantly affect the characteristics of the restoration. In addition, the present study evaluated the addition of Ti-NP *in vitro*. Therefore, further studies are needed to further explain the effect of Ti-NP on dental ceramics in vivo [59-64]. Although, commercial dental composites available in the market are clinically easy to use, problems such as low fracture strength still limit their applications, especially in posterior teeth. The present study was conducted with the aim of determining the effect of LD type on the mechanical properties of dental composites [65-68]. Many works investigate the reinforcing effect on mechanical properties, apatite-mullite ceramic glass particles were used, which had equal amounts of SiO_2, ZrO_2, and TiO oxides as variables. In recent years, dental composites have become the most important because of the retreat of amalgam and the increasing demand for beauty. It has become a treatment option in clinical dentistry and has been widely used to repair caries and other dental defects. Composite resins are one of the most important dental materials that are also widely used in dentistry [69-70].

CONCLUSION

As the Ti-NP content increases from the S1 to the S4 specimen, the peaks observed in the XRD pattern become denser and their dispersion decreases, which increases the mechanical properties and increases the chemical stability. Addition of 2 wt% and wt5% Ti-NP increased the compressive strength of LD ceramic compared to the control group but decreased significantly compared to the conventional commercial sample. Addition of 5 wt% Ti-NP increased the compressive strength of LD ceramic compared to the control group. The highest average compressive strength in the standard commercial groups of e max standard (449.42 ± 10 MPa), 5 wt% Ti-NP (341.67 ± 10 MPa), 2 wt% Ti-NP (83 ± 10.573 MPa), 1 wt% Ti-NP (258.92 ± 5.5 MPa) and control (211.83 ± 5.55 MPa) were observed. In addition, the oral cavity is a different clinical setting. For example, the presence of water, changes in temperature and pH level in the oral cavity may also significantly affect the characteristics of the restoration. In addition, the present study evaluated the addition of Ti-NP *in vitro*.

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AUTHOR CONTRIBUTION

The author contribution is as Dr. Aida Rajaei work as Dental Resident. Assit. Prof. Kazemian, Dr. Amirsalar Khandan are presented as Aida Rajei supervisor and co-supervisor, respectively.

DECLARE FINANCIAL SOURCE

This research with the ethics code IR.IAU. KHUISF REC.1399.230 was registered on 2021.

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