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The impact of fabrication techniques and materials used on the surface integrity of Ceramic Dental Crowns

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Abstract
Objective: The rise of digital technologies and materials advancement have introduced a range of restorations used in fixed restorative dentistry with different properties. This study aimed to evaluate and compare the micro hardness and surface roughness of four distinct ceramic materials using three different fabrication methods. Methods: Four different ceramic materials were used in this study (n=48): IPS e. Max, e. Max CAD, 3D-printed Nano ceramic, and zirconia- reinforced lithium disilicate. These materials were fabricated using three different techniques: CAD-CAM, pressing, and 3D printing. The materials investigated were artificial aged via thermocycling. Subsequently, Vicker's micro hardness values were obtained using micro hardness tester, and the surface roughness values were evaluated using atomic force microscopy. Statistical analysis was performed using Kruskal Wallis test. Results: Both CAD-CAM and conventional (press) Subtractive fabrication technique exhibited the highest micro hardness values ($p \leq 0.05$) but the press IPS e. Max had a lowest surface roughness ($p \leq 0.05$) when compared to the other fabrications techniques, namely CAD CAM and 3D printing. Among the materials evaluated, ZLS demonstrated the highest micro hardness and surface roughness values ($p \leq 0.05$) in comparison to IPS e. Max, e.max CAD and 3D- printed Nano ceramic. Conclusion: The use of different materials and fabrication techniques can result in restorations with varying surface properties. Therefore, the clinical decision regarding the selection of a restoration type should be made with careful consideration of these differences.

Keywords: ZLS (zirconia reinforced lithium silicate), 3D printing, CAD-CAM, Micro hardness, Surface roughness

Introduction
Surface characteristics are part of the most essential variables to evaluate when selecting a restorative material. Hardness is an excellent predictor of mechanical characteristics in dental materials and is defined as the material's resistance to prolonged indentation or penetration. Ceramic materials' hardness influences their machinability, polish ability, and wear resistance, and it varies over time by aging, water absorption, and surface reactions [1]. The specimen surface's hardness indicates the material's surface strength and rates its resistance to abrasion. More scratches, surface deterioration, and dimensional changes may occur when mechanically brushing or grinding hard food materials with low hardness [2]. Several tests, such as the Knoop's, Vickers, and Martens tests, can be used to determine hardness. Nonetheless, Vickers micro hardness tests are typically used by researchers to estimate the hardness value of dental material [3]. As previously described by (Li et al., 2021), when the hardness reduced, the roughness increased due to inverse relationships, making the correlations between the two surfaces' features plausible [4].

An essential feature that all dental materials, including glass ceramics, share with common is surface roughness. It influences bacterial adhesion as well as the final look of restorations. Ceramic restorations with a high degree of roughness on their intaglio surface are chosen for bonding with hard dental tissues since they increase the strength of the bond. On the opposing side, the outer surface of a dental ceramic restoration requires to be as smooth as possible in order to prevent or lessen the buildup of plaque, bacterial adherence, irritation of the gingiva, or eventually dental decay [5]. Roughness is a collection of defects or microscopic indentations that define a surface and have an impact on wetting, adhesion, and brightness [6].

One of the best techniques for investigating the surface morphology and topography of dental materials is AFM [5]. For preparing an extra coronal restoration, two techniques are often available: subtractive and additive. Lithium disilicate restorations can be made using the subtractive technique in two ways: pressing with ingots using the lost wax technique and milling using a CAD-CAM system. The above techniques have some drawbacks as involving expensive manufacturing instruments, high production costs, and constraints in complex milling shapes [7]. The other technology, additive manufacturing (AM), depends on computerized CAD design models and use standardized materials for producing specific 3D products using preprogrammed mechanized operations. While compared to subtractive techniques, 3D printing can give more accurate restoration as well as minimize material loss significantly [8]. Additive manufacturing (3D printing) produces physical products from geometrical models by serially adding material. This 3D approach has grown dramatically in recent years. In 1980, Charles Hull created the first 3D printing process model [9].

This in-vitro study intends to evaluate and compare the micro hardness and surface roughness of monolithic crowns made from lithium disilicate, 3D Nano ceramic, and ZLS using different fabricating techniques. The null hypothesis suggested that neither the fabricating technique nor the material types for the monolithic ceramic crown would affect their micro hardness and surface morphology.

Materials and Methods

Sample preparation: The sample was designed by the AutoCAD program as a rectangle bar of (10*5*2mm) length, width, and thickness dimensions, respectively (Figure 1). That be saved as a standard tessellation language (STL) file, which is essential for the 3D-printing and the milling systems technique.

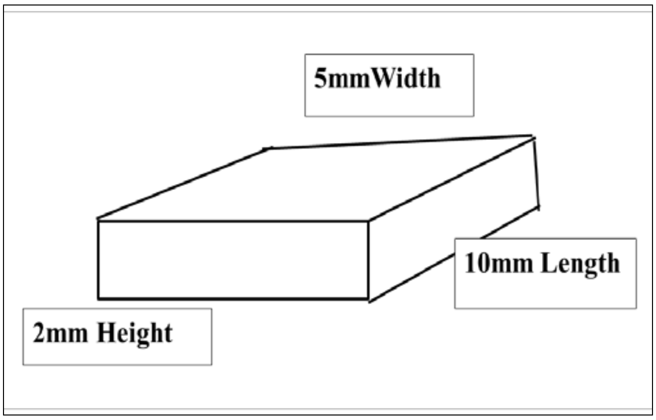

Fig 1: Schematic diagram for the dimensions of prepared sample.

For the conventional technique, a wax template of a rectangular shape bar with the dimension (5×10×2mm) is made by 3Dprinter (Holtsky, Creality 3D Technology Co, Ltd., Shenzhen is a Chinese 3D printer manufacturing company established in 2014), such templates used to produce pressed lithium disilicate samples (IPS e.Max) thru applying the “lost-wax and press-technique” according to the manufacturer’s instructions, in which the wax- templates were fixed by a wax sprue (IPS Multi Wax Pattern Form A; Ivoclar Vivadent) that invested in IPS Press VEST Premium (Ivoclar Vivadent), then put in an oven (KaVo EWL 5645; KaVo, Kloten, Switzerland) to complete melting rate of 5 ◦C min^{-1} from room temperature to 850 ◦C (holding time 60 min) to obtain mold, in which a lithium-disilicate ceramic (IPS e.Max Press; Ivoclar Vivadent) injected. The invested ring will burn out throw a pressing furnace (Programat EP 5010 Ivoclar Vivadent), with an ingot and plunger loaded to complete the pressing rate 60°C min^{-1} from 700 ˚C to 898 ˚C; (holding time 25 min). After cooling, the samples were carefully devested and cleaned from the investing material by air abrasion particles (Germany /Renfert GmbH No.15941305) for 3minutes. The surface will be glazed using IPS e. Max Fluorescence Glaze, and again placed into the sintering-oven and (12) samples made by this technique [10].

While obtaining the ceramic sample of the group e. Max CAD/CAM (IPS e. Max CAD (LT A2, Ivoclar Vivadent, Schaan, Liechtenstein) Dental CAD/CAM system (Yenadent) utilized to generate a rectangular shape bar sample based on the (STL) file prepared first on the same dimension. After the milling process completed, the samples were carefully separated and taken out of the Emax blanks. A football-shaped, fine diamond bur (379-023 M-HP) was used to adjust the margins of each sample to remove any extra material. All samples were sintered in a high-temperature Zetain Sintering Furnace (400-860◦C) for 25 minutes, following manufacturer specifications. Outer contour surface for e. Max CAD samples was polished, cleaned, dried, and then fired with a glaze. So, (12) samples prepared.

For the ZLS samples prepared by using the STL file created previously thru CAD-CAM system, and sintered by Zetain sintering furnace at 1530°C. Finally, 3D printing Nano ceramic samples prepared by using the STL file created previously thru Auto CAD. Prior to starting the procedure, the Phrozen Mighty 8K 3D printer used the Vat Photo Polymerization technique for their 3Dprinting process, Resin begins as a liquid, but when exposed to light, it undergoes chemical reactions and solidifies, resulting in the formation of a solid object. The technique begins by heating the resin bottle in a hot water jar. It is critical to use all resin in the container and to blend all Nano ceramics into the product. Put water (1L - 1.2L) into a water heater and bring it to a boil; when the water boils, the machine will stop; place the entire bottle inside the water heater, wait 5-10 minutes, then shake rapidly for 30 seconds to 1 minute before placing the resin in the tank of the 3D printing devices and starting the print. After printing, clean the samples in an unheated ultrasonic bath with a 96% ethanol solution for 3 minutes (ultrasonic cleaner power 30 - 35 watt). Resin residues can also be removed with a brush soaked in ethanol (96%), and immediately after alcohol cleaning, wash with tap water for 5-10 seconds, spray with alcohol for a final clean and dry by pressured air. After curing, use a cutting wheel to remove the support structure. Then, blast the white layer with 50 µm glass beads at 1.5 bar pressure for 3 minutes. Check for proper fit and finish the items completely. Finishing and contouring can be done with a carbide cutter or a diamond grinding stone (PH-1011). Finally, forty-eight samples were obtained using three distinct techniques.

Artificial aging: To simulate one year of intraoral use, the 48 manufactured rectangular bars will be thermally cycled utilizing a thermos cycling equipment (Dorsa, Iran) for 5,000 cycles at 5 to 55◦C with a dwell time of 30 seconds [11].

Testing of the samples

Micro-Hardness Test: Using the micro hardness tester DM 8/DM 2 (Yang Yi Technology Co., Ltd, Tainan City 70960, Taiwan), the outer surface of twenty-four rectangular bars-six for each of IPS e. Max, e. Max CAD, ZLS, and 3D printing Nano ceramic-was subjected to a diamond pyramidal indenter with a 300 g load for 10s (hold time) in order to determine the micro hardness values for these materials. Each bar's Vickers hardness number in Kg/mm2 will be obtained by calculating the mean values of the three indentation measurements performed on the center of the bar [12].

Atomic Force Microscopy Test (AFM): AFM used to scan the intaglio surface of 24 rectangular bars in order to use a touch-based method to ascertain the surface topography. The sample was attached to a three-degree-of-freedom scanning piezo using a little piece of double-sided tape on the holder. Silicon cantilevers were touched with a very tiny probe tip (P/N 910 M-NSC 36). The specimen's surface was scanned using cantilevers with spring constants of 0.95 and 1.75 N/m at 3.3 hertz speed [13]. After taking two readings at the center of each bar. The AFM software program was used to determine the SA value.

Statistical analysis: The micro hardness and surface roughness values were submitted to abnormality test so non parametrical analyzing was done and statistical analyzed by Kruskal Wallis followed by Dunn's to compare the effect of materials type and techniques on the micro hardness and roughness values of the ceramic samples that prepared using the IBM SPSS statistics version 25 at ($p \leq 0.05$).

Results: THE effect of technique: A descriptive statistic including the mean and standard deviation of the data was gathered in order to investigate the relationship between the preparation method and the micro hardness value of the samples made from four distinct materials (Table 1). Regarding to the effect of technique on the micro hardness values of the samples of the four different ceramics materials, Kruskal Wallis test showed that a significant difference between techniques at ($p \leq 0.05$) (Table 1). To identify the level of significant that is obtained, Dunn's multiple range test showed that the mean values for subtractive techniques (CAD-CAM and conventional) were significantly higher than 3D printing technique. To determine the relation between the preparing techniques and the surface roughness values of the rectangular bar's samples prepared so a descriptive statistical analysis including the mean and standard deviation values of such data will be obtained (Table 1). Regarding to the effect of technique on the surface roughness value of the samples, Kruskal Wallis test showed that there was significant difference between values at ($p \leq 0.05$) (Table 1). To identify the level of significant that is obtained, Dunn's multiple range test showed that the sample prepared by CAD-CAM and 3Dprinting techniques a significantly higher surface roughness value than Press technique (conventional) which had a lowest value.

Table 1: Descriptive statistical analysis including the mean and standard Deviation for the microhardness values.

Type	Preparing technique		Microhardness	Surface Roughness
Subtractive System	Conventional Technique	6	441±4	33±5
	CAD-CAM Technique	12	452±130	303±197
Additive System	3Dprinting Technique	6	35±3	258±5.5
P value			0.003	0.002

Data expressed as mean ±Sd, Kruskal Wallis test used to identify the significant differences at p value less than 0.05.

The effect of materials used: To determine the relation between the materials used and micro hardness value of the samples which prepared from three different techniques (Press (conventional), CAD-CAM and 3Dprinting), the data were collected and a descriptive statistic including the mean and standard deviation of such data will be obtained (Table 2).

Regarding to the effect of materials used on micro hardness value of rectangular bar samples, Kruskal Wallis test showed that there was significant difference between ceramic materials at ($p \leq 0.05$) (Table 2). To identify the level of significant that obtained, Dunn's multiple range test showed that the samples that prepared from ZLS material have the highest micro hardness values while Nano ceramic was the lowest one. To determine the relation between the materials type (IPS e. Max, ZLS, e. Max CAD, and 3D Nano ceramic) and the surface roughness values of the rectangular bar's samples prepared a descriptive statistical analysis including the mean and standard deviation values of such data will be obtained (Table 2). Regarding to the surface roughness of rectangular bar samples prepared from a four different ceramics material (ZLS, IPS e. Max, 3 Dnanoceramic, and e. MAX CAD). Kruskal Wallis test showed that there was significant difference between ceramic materials at ($p \leq 0.05$) (Table 2)

To identify the level of significant that obtained, Dunn's multiple range test showed that the mean surface roughness value for rectangular sample prepared from ZLS was significantly had a higher roughness value while IPSe. MAX was the lowest one. To understand the effect of material on mechanical and topographical properties of the rectangular bars prepared from different ceramic materials (IPS e. Max, ZLS, e. Max CAD, and 3D Nano ceramic) the mean values for micro hardness and surface roughness testes performed in this study.

Table 2: Descriptive statistical analysis including the mean and standard Deviation for the micro hardness values.

Ceramic materials	N	Microhardness	Surface roughness
e. Max CAD	6	328±6	127±3.7
IPS e. Max	6	441±4	33±4.7
Nano ceramic	6	35±3	259±5
ZLS	6	577±5	524±2.3
P value	24	0.001	0.001

Data expressed as mean ±Sd, Kruskal Wallis test used to identify the significant differences at p value less than 0.05.

Discussion

For subtractive techniques including CAD-CAM and press. The preparing for press technique involves various procedures, each of which may have an impact on the end product. The press technique may change the dimensions of the impression by transporting it to a dental laboratory and subjecting it to temperature variations. Additional distortions may occur as consequence of the time between taking the imprint and pouring the stone cast, the room temperature, the surface wettability of the gypsum product, and disinfection. Errors may occur during the investment and pressing procedures, the use of a die spacer, or the creation of a wax model of the desired crown, and the investment and pressing operations can all cause error [14].

The CAD-CAM technology has grown in popularity and trust among professionals and patients. It improved restoration quality and reduced stress for dentists and lab personnel. Furthermore, current CAD-CAM restorations are more durable and quicker to manufacture than traditional restorations. Despite these benefits, the biggest barrier to adoption is the high cost. Because of financing limitations, dentists in developing countries are still hesitant to utilize CAD-CAM. However, one should not overlook the benefits that CAD CAM provides. When we compare the advantages of CAD CAM restorations to traditional ones, CAD CAM restorations will certainly come out on top. They provide us with high-quality solutions that are quickly and easily fabricated [15].

However, there are several disadvantages to using CAD-CAM, including the possibility of introducing micro cracks during the milling process, the need to replace worn tools, material waste, and the limited ability to replicate surface geometry due to the size of the milling tools and the computer numerical control machine's working axes [16]. Unlike subtractive manufacturing, additive manufacturing is "a process of combining materials to make objects from 3D model data, usually layer upon layer. "It enables the production of smoother, more complex prosthetic devices while using less materials [17].

Micro hardness, which can be defined as resistance to persistent indentation or penetration, is one of the most important mechanical properties. It shows how well the restoration can tolerate mastication force. It is employed to determine a material's resistance to wear and tendency to erode opposing dental structures [18].

The CAD-CAM technique had the highest micro hardness value (462.2) and the 3D printing technique had the lowest (35.2). This could be because the CAD-CAM technique presses e.Max CAD ceramic ingots under controlled industrial conditions, creating a more homogeneous microstructure. It's also possible that the subsequent machining (grinding) process for the actual fabrication sample could produce a high micro hardness surface [19].

While the 3Dprinting process had the lowest hardness value, this may be due to a number of factors that influence the mechanical properties of printed restorations, such as polymerization degree, build thickness, and the use of reinforcing materials [20]. Stereolithography uses a photopolymerizable substance. The light source, layer thickness, material qualities, photopolymerization initiator type, and presence of other additive components all had an effect on polymerization degree. This means that the mechanical qualities of the finished product will vary according to the manufacturing conditions [21]. Three-dimensionally printed materials are created via a layering procedure, which results in a chemical link between the layers. The mechanical properties of 3D-printed resins are affected by the fabrication technology used. Ibrahim et al. (2020) found that orientation and layer thickness during printing influence the mechanical properties of these materials [22].

The samples made using ZLS had the highest micro hardness value (576.6) and those made with 3D printing Nano ceramic had the lowest (35.2). It has been suggested that adding tetragonal zirconia to the composition of ZLS, another type of glass-ceramic CAD/CAM material, may improve its mechanical properties [23]. The dual microstructure of this multi-component glass-ceramic is composed of a glassy matrix with 10% zirconium oxide and fine lithium Meta silicate with LD crystals. Zirconium oxide is thought to improve the material's mechanical strength. After the last fire cycle, the material, which has been pre-crystallized, will reach its full density [24], and this could result in the ZLS samples having the greatest micro hardness value. The mechanical characteristics of Resin Nano Ceramics (RNC), which are influenced by the distribution and amount of ceramic fillers, may be the reason for the 3D-printed Nano ceramic's lowest micro hardness value. Therefore, the improvement in RNC hardness may be influenced by the increase in ceramic filler content as well as the state of the slurry mixing, where the filler is evenly mixed with the matrix. Agglomeration happens when the ceramic filler and resin are not mixed evenly, and the filler content may have less of an impact on the micro hardness value.

The restoration's surface topography played great role in the formation of dental biofilm, debris, and dyes, causing not only gingival irritation and the risk of secondary caries, but also decreasing the gloss of the restoration, leading to discoloration and/or surface degradation [25]. At the same time, it can vary depending on a number of factors, including fabrication technique and material composition [26].

The samples made using the CAD-CAM approach had the maximum surface roughness (320.4), whereas the samples made with the IPS conventional technique had the lowest value (32.9). The form of the milling tools used in subtractive milling has an impact on roughness in CAD/CAM technology. In contrast to press methods, the milling process uses diamond burs to selectively reduce prefabricated blocks to the desired shape. The surface geometry of ceramic blocks may be altered by the diamond burs used in the milling process, producing a rougher surface [27]. The pressing method, on the other hand, has less shrinkage throughout the process, which lowers surface porosity and, consequently, surface roughness [28], which could account for the lowest surface roughness in the pressing method.

Surface roughness is also significantly influenced by the type of material. The ZLS had the maximum surface roughness (523.7), whereas IPS e. Max had the lowest (32.9). The pre-crystallized composition of ZLS is responsible for its surface roughness. This composition includes tetragonal zirconia fillers with round and submicrometric elongated grains of lithium orthophosphates and Meta silicates embedded in a porous glassy matrix [29].

IPS e. Max Press has the lowest surface roughness because its microstructure is made up of lithium disilicate crystals (approximately 70%), $Li_2Si_2O_5$, integrated in a glassy matrix. The predominant crystal phase is lithium disilicate, which has needle-like crystals. The crystals measure 3 to 6 µm in length. In general, larger and longer crystals. The firing temperature of 890◦C in e. Max Press aligns crystals, resulting in a smooth surface [30].

Conclusion

The use of different materials and fabrication techniques can result in restorations with varying surface properties. Therefore, the clinical decision regarding the selection of a restoration type should be made with careful consideration of these differences.

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