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Shrinkage Behaviors of Conventional Sintered Dental Zirconia

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سلوك الإنكماش لزركونيا الأسنان التقليدية الملبدة

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Abstract:
Shrinkage is a critical phenomenon throughout the sintering of dental ceramics as it disturbs the structural and physical behaviors. In the present paper, the shrinkage behavior of pre-sintered and sintered zirconia blocks were investigated. The physical attributes such as density and porosity were also evaluated. Three experimental groups of dental zirconia blocks were designed and fabricated from commercial dental zirconia blocks (HT4, 89HT and A214). A total of 12 discs of zirconia samples were then prepared. The density and porosity of pre-sintered and sintered zirconia samples from various brands were computed. The linear and volumetric shrinkages of zirconia samples were computed and evaluated. The outcomes exhibited that the sintering temperature led to the densification of zirconia samples and the relative density values reached up to 97.5%, 94.8% and 93% for HT4, 89HT and A214 respectively. As the density of zirconia samples increased, a dramatic decrease in the pores content after the sintering process was observed. The densification process was accompanied by a degree of contraction. When the linear shrinkage of HT4, 98HT and A214 were 20.27%, 20.74% and 19.58% respectively, they achieved 49.37%, 48.5% and 48.27% as volumetric shrinkage. The shrinking value of all tested zirconia met the clinical application requirements.

Keywords: Dental ceramics, Linear shrinkage, Sintering, Zirconia block.

الملخص
يعد الانكماش من الظواهر الهامة التي تحدث أثناء تلبيد الخزف لما له من تأثير على تركيب وخصائص الخزف الفيزيائية. في هذه الورقة، تم دراسة خصائص الانكماش لكتل الزركونيا المحماة والمتكلسة مسبقا. كما تم تقييم بعض الصفات الفيزيائية مثل الكثافة والمسامية. حيث تم تصميم وتصنيع ثلاث مجموعات لعينات التجارب من كتل زركونيا الأسنان التجارية (HT4، HT89 وA214). بعد ذلك، تم تحضير إجمالي 12 قرصًا من عينات الزركونيا. حيث تم حساب كثافة ومسامية عينات الزركونيا قبل وبعد التلبيد التهائي. كما تم أيضاً حساب ومقارنة الانكماش الخطي والحجمي للكتل الحاصل بعد عملية التسخين أو الاحماء. أظهرت النتائج أن درجة حرارة الاحماء أدت إلى تكثيف عينات الزركونيا ووصلت قيم الكثافة النسبية إلى 97.5%، 94.8% و93% لـ HT4، HT89 وA214 على التوالي. مع زيادة كثافة عينات الزركونيا، لوحظ انخفاض كبير في محتوى المسام بعد عملية الاحماء. كما كانت عملية التكثيف مصحوبة بدرجة من الانكماش. عندما بلغ الانكماش الخطي لـ HT4، HT98 و A214، 20.27%، 20.74% و19.58% على التوالي، بينما بلغ الانكماش الحجمي قدراً يعادل 49.37%، 48.5% و48.27%. وعليه فإن قيمة الانكماش لجميع الزركونيا التي تم اختبارها قد استوفت متطلبات التطبيق السريري أو العيادي.

الكلمات المفتاحية: خزف الأسنان، الانكماش الخطي، الاحماء، كتل الزركونيا

Introduction:

Zirconia prosthodontics are constructed utilizing computer-aided design and computer-aided manufacturing (CAD/CAM) either by hard machining completely sintered blanks or by soft machining pre-sintered blanks The first generation of CAD/CAM, created by Duret and coworkers, coupled controlled machining and milling with intraoral scanning to construct the final crown. This was not the case with the standard CAD/CAM approach,

which only required laboratory work. An alternative approach called networked CAD/CAM processing was developed for dental technology, but its poor precision kept it from being used extensively. While CAD can be used to find 2-D geometry, CAM can be used to process the suggested design and determine the cutting path using various tools. All that CAM is is machine code that is used in copy-milling uses to make a prosthetic out of ceramic [1].

Zirconia is white and opaque like chalk at room temperature and exists in the monoclinic, tetragonal, and cubic phases as the temperature increases. Additionally, zirconia offers abrasion resistance, biocompatibility, chemical stability and has a high melting point, therefore, it is widely used as a heat-resistant material and is known to have very high fracture strength. Polycrystalline zirconia stabilized by adding 3-5 mol% yttria (yttrium tetragonal polycrystals, Y-TZP) is widely used in dentistry [2, 3]. Vafaei, et al. [4] reported that monolithic zirconia restorations have become more popular because of their ideal hue, which results in less wear on the adjacent teeth, their conservative preparation method, and their high rate of long-term clinical success.

Commercially, pre-sintered and entirely sintered zirconia blocks are the two available process phases. Pre-sintered blocks allow for easier, faster, and less tool wear during milling. Pre-sintered blocks are widely used in the global market to fabricate zirconia structures because of these benefits. To provide adequate strength following milling, the material must be sintered at a high temperature when pre-sintered blocks are utilized. The material contracts during the sintering process, strengthening and densifying the framework [5]. Furthermore, the Y-TZP's microstructure is impacted by the temperature and length of sintering. To avoid the formation of a dual cubic-tetragonal microstructure, the procedure should be carried out at low enough temperatures, yet high enough temperatures to produce fully dense materials [6]. Hirano et al. [7] stated that sintering-accompanied shrinkage frequently begins at 900–1000 ◦ and ends at about 1500 ◦C. Consequently, a substitute for zirconia has been investigated and encouraging outcomes have been found [8].

Compared to pre-sintered zirconia, full-sintered zirconia is stronger, less porous in volume percentage, and more resistant to hydrothermal aging. Furthermore, since no additional heat treatment is necessary to produce a dimensional change, the fully sintered zirconia can be milled to the final specified dimensions [9]. Unfortunately, extensive milling times and rapid tool wear are caused by the high strengths of the dense completely sintered blocks. However, to reach their greatest strength, the pre-sintered blocks need to be sintered after milling, despite the fact that they are simple to shape. Therefore, sintering shrinkage must be considered before milling when using pre-sintered blocks [10]. To compensate for this shrinkage, a larger design is prepared in advance during machine milling, following which the manufacturing process is carried out. Thus, the precision may be reduced, and the thin part can easily break. It is crucial to predict the shrinkage properties of zirconia ceramics to produce final sintered products with near-net shape and no defects. As a result, a great deal of research has been done on the fit and sintering shrinkage of zirconia prosthodontics [4-6]. Knowing the precise volume shrinkage for each zirconia blank is essential to maximizing the fitting of the restoration. Therefore, the purpose of this study was to determine and compare the density, porosity and sintering shrinkage of three commercially available dental zirconia.

Methodology

Materials

The materials used in this study were provided by Jaghbub Dental Lab, Benghazi and Alfaieq Dental Center, Misurata. They were purchased from the local market. The details of the collected products are presented in Table 1.

Table 1 Description of experimental materials.

Specification	Materials		
Product name	Dental Zirconia Blank	Balaam Zirconia	Everest Zirconia
Product Number	D98-12	HT-W	-
Product Code	HT4	98HT	A214
Structure	Monolayer	Multilayer	Multilayer
Block Dimension	98*14	98*14	98*14
Place of Production	China	China	S. Korea

- **Specimen Preparation**

A total of 12 discs of zirconia samples were made from three yttrium oxide-stabilized zirconia blocks, which are frequently employed for the fabrication of prosthodontics. The samples were made utilizing computer-aided

design (CAD) software (Exocad GmbH, Darmstadt, Germany) to create 12.0 × 5.0 mm^3 circular samples per disc. A computer-aided milling (CAM) machine (Roland DWX 52D) was employed to cut the discs (Figure 2). Every zirconia block underwent furnace sintering (Tabeo-1/m/zircon-100) at 1530∘C for 13 hours. One lab technician completed this task in accordance with the manufacturer's instructions. The sintering details are displayed in Figure 1. The tested samples before and after sintering are shown in Figure 2.

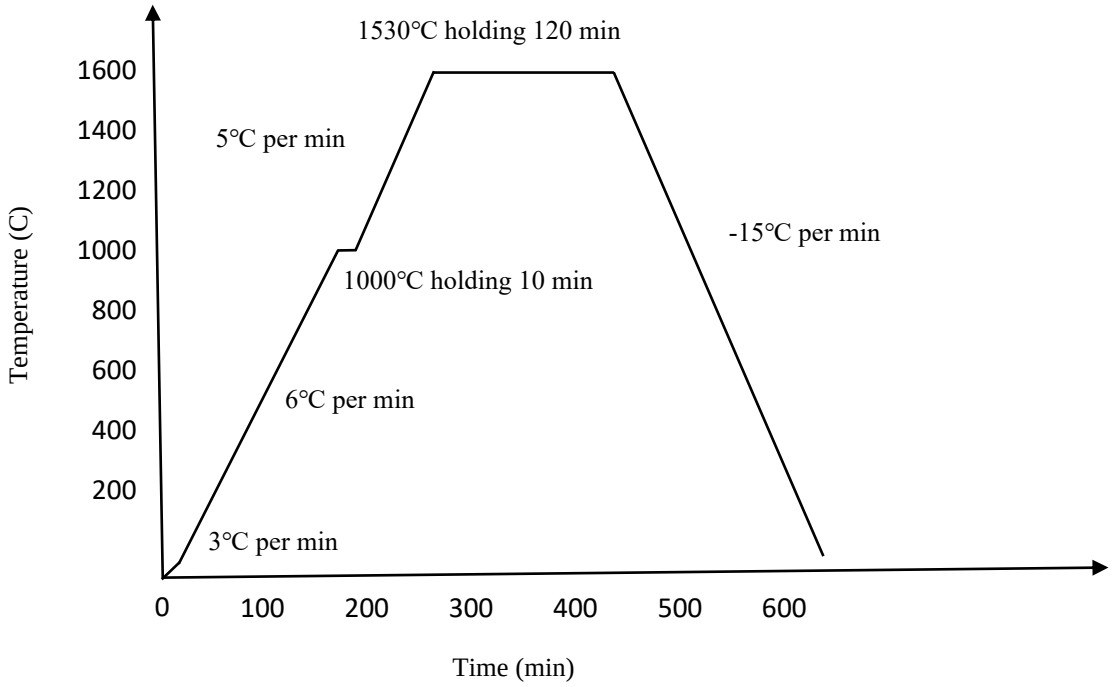

Figure 1 Sintering profile as recommended by the manufacturer.

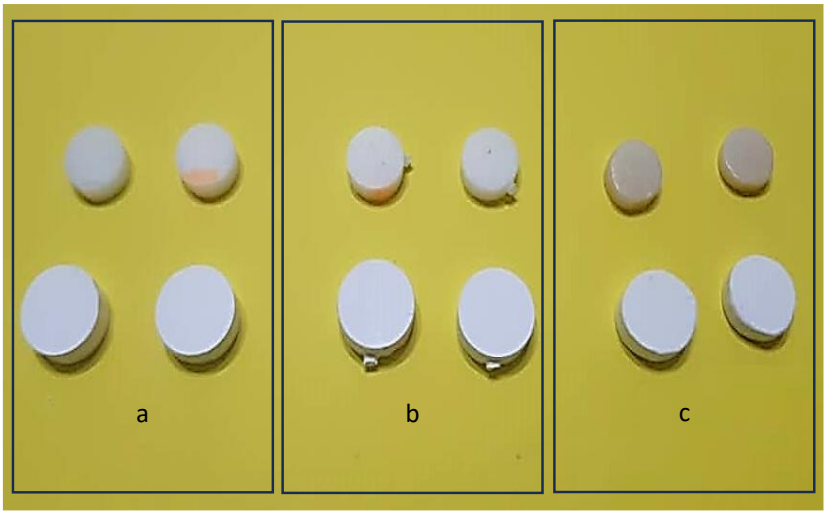

Figure 2 Zirconia samples used in this study, a: HT4, b: 98HT, c: A214.

Methods

- **Density Determination**

After collecting the pre-sintered and sintered samples, a digital caliper was used to measure the exact dimensions (thickness and diameter). The specimens were then weighed with a digital balance (ME204E, Mettler Toledo, USA) The apparent density of the samples was determined by the sample's weight divided by the sample's volume. The theoretical density value is 6.51 g/cm3 which is utilized for computing the relative density [11].

- **Porosity Evaluation**

The pre-sintered samples have a porous nature aiding in reshaping the material to the final form. Densification can be achieved through sintering to densify a low-density performance. Therefore, the bulk porosity of the pre-sintered and sintered zirconia ceramics is calculated based on their relative density using the subsequent formula [12]:

$$p = (1 - \rho) \times 100\% \quad (1)$$

where*: p* is the bulk porosity and ρ is the relative density.

- **Assessment of Sintering Shrinkage**

The pre-sintered and sintered blocks' lengths and weights were measured using a micrometer caliper and weighing apparatus. These measurements, which were made three times over, were utilized to compute the linear and volumetric sintering shrinkage of each specimen using the following equation [10, 13, 14]:

$$\Delta L = (L_0 - L) / L_0 \times 100 \quad (2)$$

where: ΔL: linear sintering shrinkage (%), L_0: the length of the specimen before sintering, L: the length of the specimen after sintering

Results and Discussion

- **Characterizing the surface of the pre-sintered and sintered samples**

For constructing fixed prosthodontic restorations, such as veneers, crowns, onlays, and inlays, CAD/CAM techniques have been widely employed [11]. The LavaTM system (3M ESPE, Seefeld, Germany), Kavo Everest® (Kavo, Biberach, Germany), and Cercon® Smart ceramics (DeguDent, Hanau, Germany) are a few commercial CAD/CAM systems that use zirconia-based ceramics. These ceramic systems come with zirconia blocks that are available in pre-sintered formula. The pre-sintered blocks are simple to shape, but to reach their maximum strength, they have to be sintered after milling due to powder deformation [15].

The zirconia surface (sintered and pre-sintered) was investigated and magnified using a digital camera at 150 × as shown in Figures 3-5. Under such magnification, pores, and impurities on the surface of pre-sintered zirconia can be observed (Figures 3a-5a). These defects could be disappeared after sintering as displayed in Figures 3b-5b. Reduction in voids and impurity elimination after exposure to the sintering temperature is expected leading to contraction. Fully sintered zirconia is stronger, has a lower volume proportion of holes, and is more resistant to hydrothermal aging than pre-sintered zirconia. Furthermore, since no additional heat treatment is necessary to produce a dimensional change, the fully sintered zirconia can be milled to the final specified dimensions [10]. Grain size and the amount of cubic phase are determined by the temperature and length of sintering. In addition, it has been skillfully programmed that the grain size would increase as the sintering temperature rises. Larger grains with fewer boundaries can be more translucent zirconia, but this increases their susceptibility to transformation, which could enhance their optical qualities but detract from their stability [16].

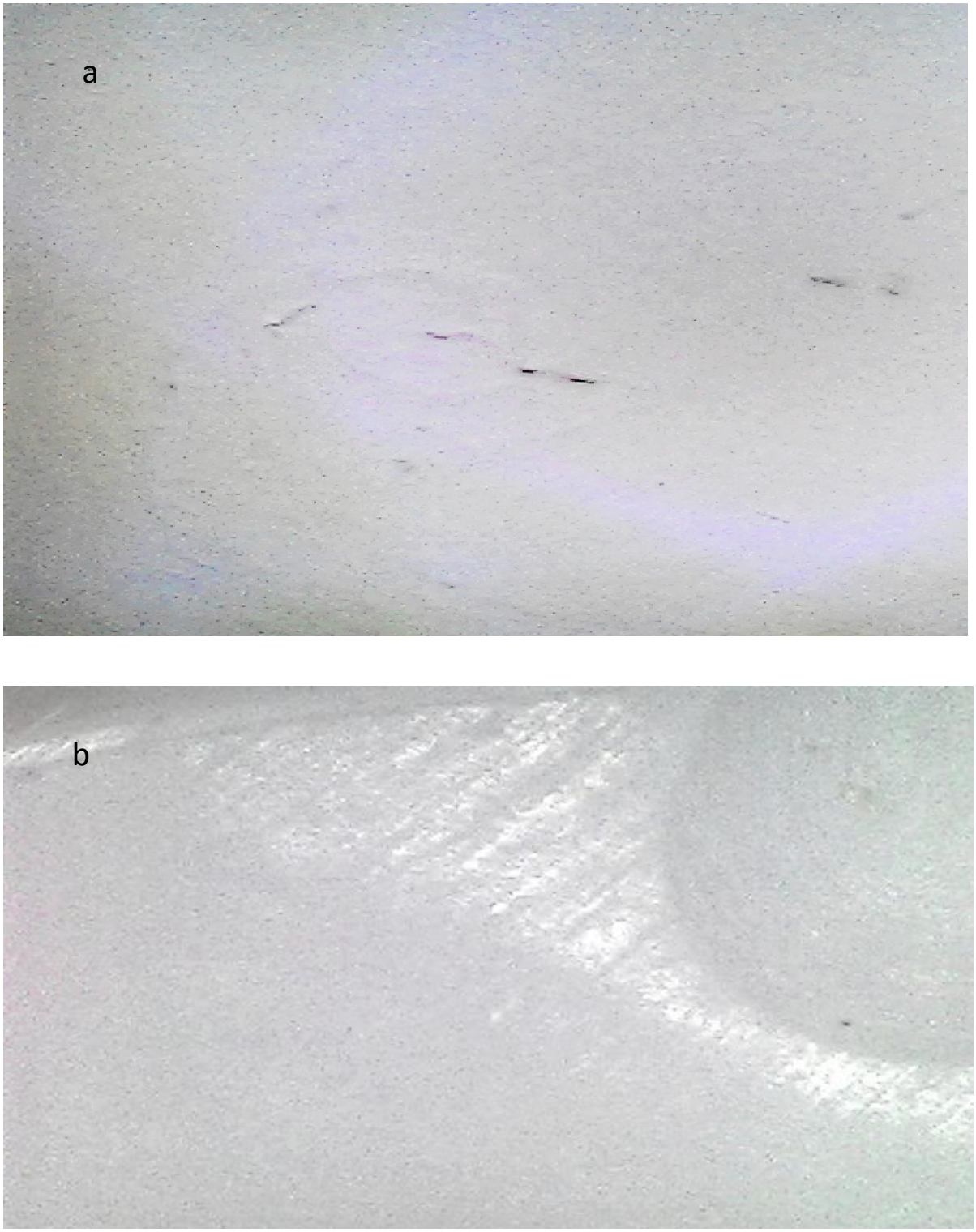

Figure 3 Microstructure of the surface of (a) pre-sintered and (b) sintered 89HT zirconia at magnification of 150 ×

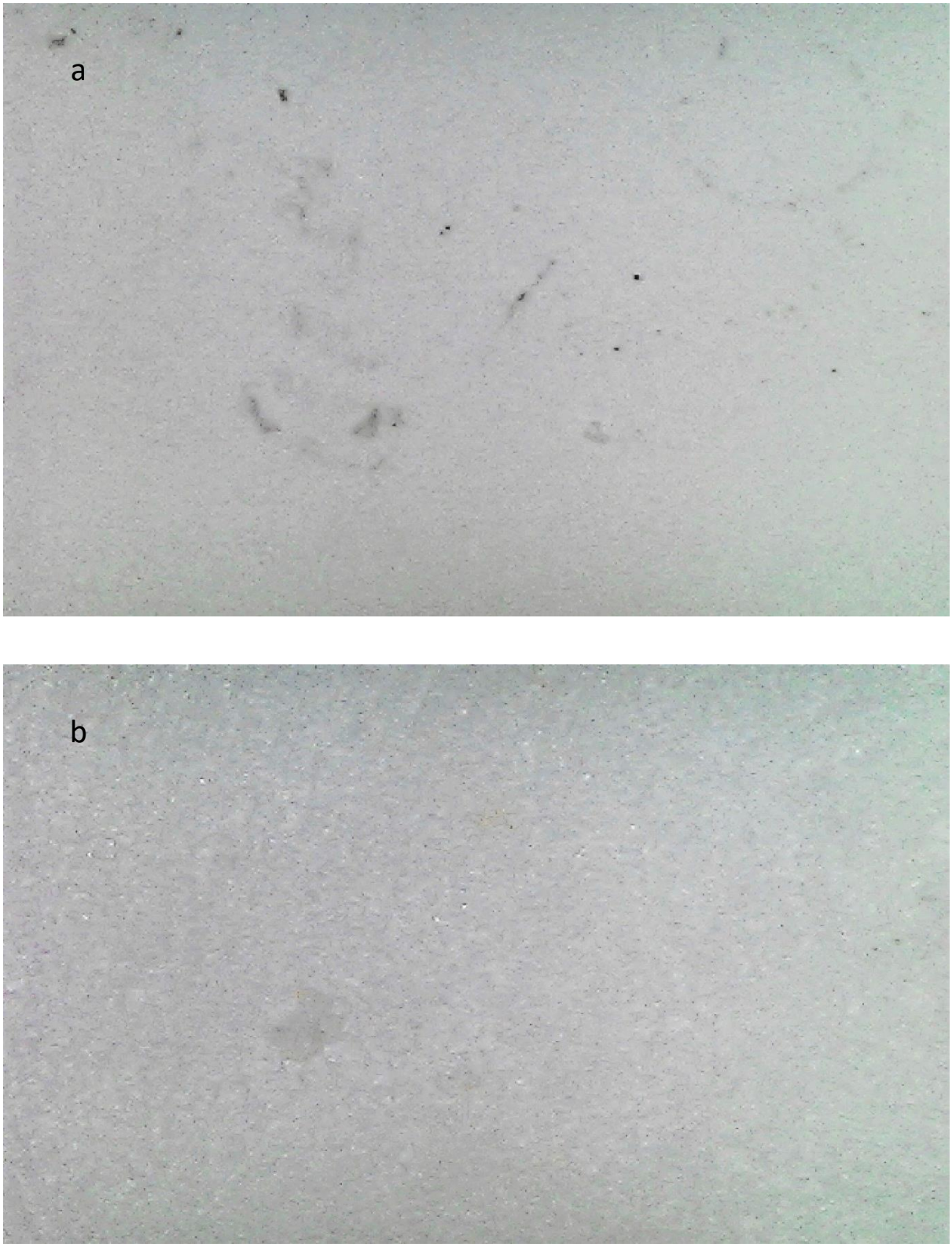

Figure 4 Microstructure of the surface of (a) pre-sintered and (b) sintered HT4 zirconia at magnification of 150 ×

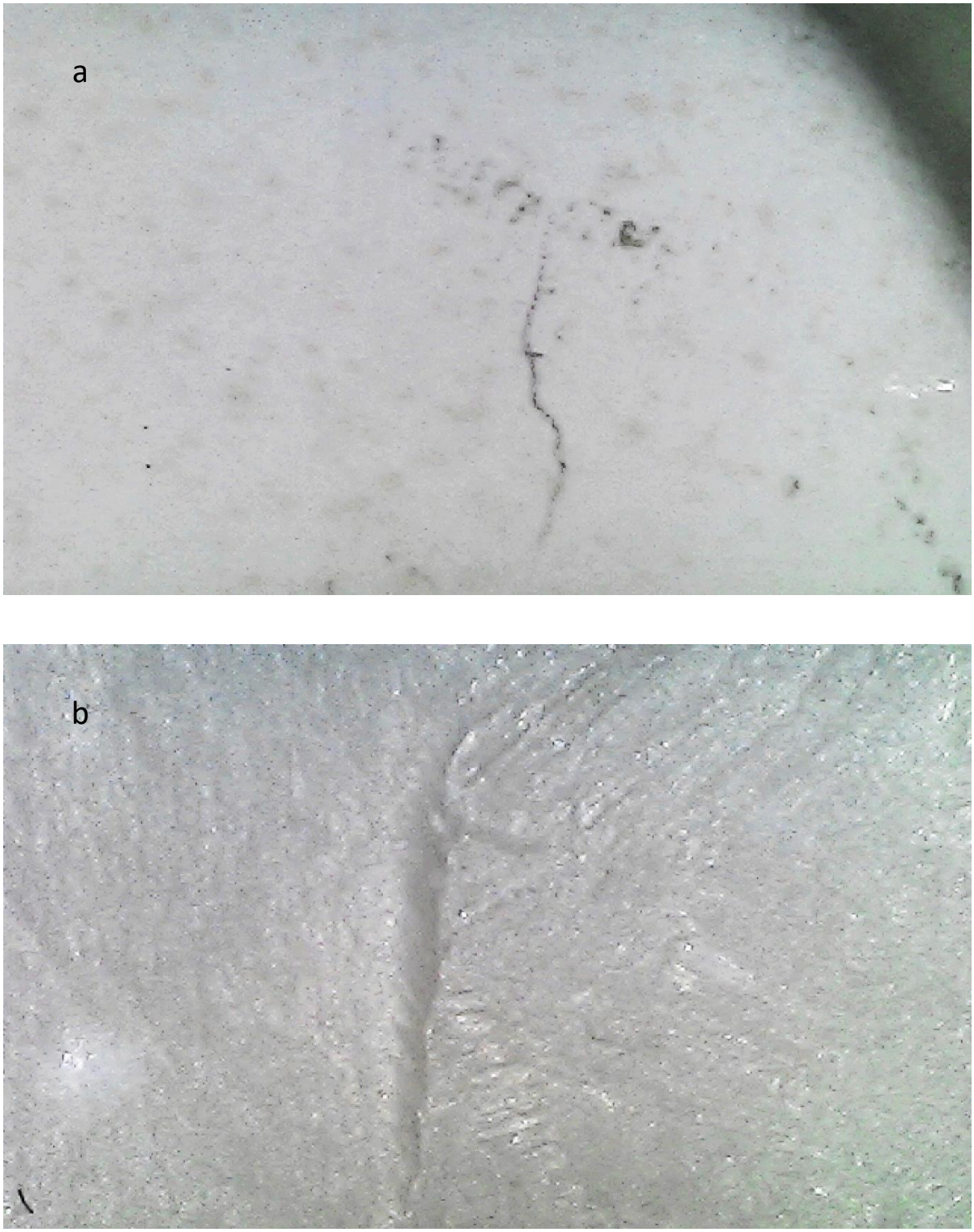

Figure 5 Microstructure of the surface of (a) pre-sintered and (b) sintered A214 zirconia at magnification of 150 ×

- **Density Determination**

Figure 6 shows the alternations in the apparent density of pre-sintered and sintered zirconia. It can be observed that the density of pre-sintered samples is much lower than that of the sintered ones. Alternatively, there is no notable variance among the pre-sintered specimens. The reduction in density values of pre-sintered zirconia could be attributed to unchanged the original shape of the particles as a result of low sintering temperature leading to low densification. At this stage, the sintering process is dominated by the surface diffusion mechanism. These outcomes match well with those of earlier research [17] where low sintering temperature yielded poor density. As

depicted in a different study, the morphological analysis revealed that the samples that were pre-sintered at temperatures of 900 °C and 1000 °C have a rough surface with visible porosity [18].

Following the sintering process, no discernible variation in the densities of the sintered blocks belonging to the different experimental groups was found. The densification of zirconia samples increased with an increase in sintering temperature [5], therefore, at 1530°C, the HT4, 89HT and A214 zirconia exhibited similar trends, i.e., the relative density values reached up to 97.5%, 94.8% and 93% respectively as shown in Figure 7. This is most likely because all of the samples were sintered under similar conditions [10]. At a higher sintering temperature of 1530 °C, the zirconia powder bonds quickly and forms a network. Porosity decreases as a result of diffusion mechanisms coming into play during the heating process at high temperatures. The powder's enormous bonding is facilitated by the energy imparted to its particles [17]. Others who deduced that raising the sintering temperature considerably enhanced the density and translucency also reported similar results [4, 19]. Despite having superior mechanical characteristics, totally dense blanks are less common than partially sintered blanks because of their lengthy milling periods and dense material's hardness, particularly when it comes to producing fixed restorations [1, 18]

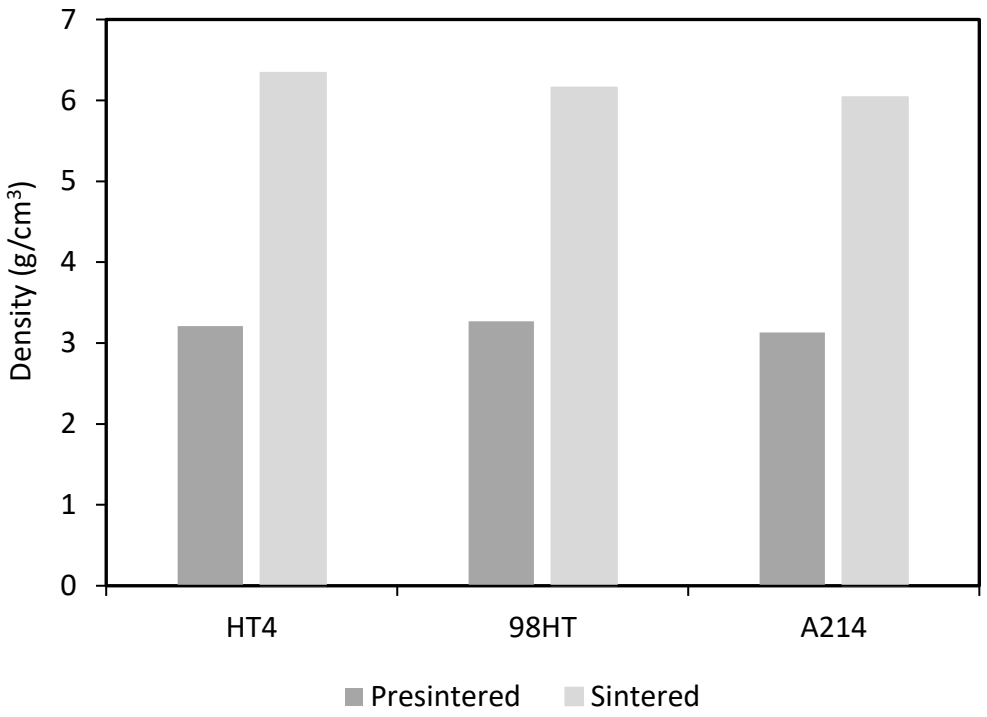

Figure 6 Average apparent densities of the experimented zirconia.

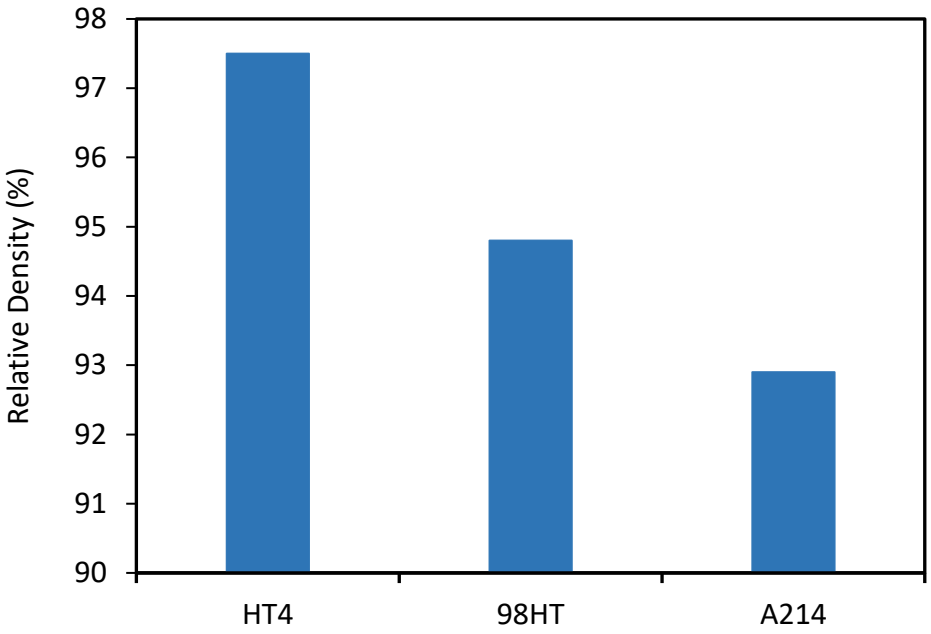

Figure 7 Relative densities of the sintered zirconia.

- **Porosity Content**

A crucial stage in the fabrication of prosthodontics, sintering has a big impact on the materials' characteristics. Heat is transmitted from the surface to the core of zirconia during the sintering process, resulting in a mature zirconia. One important factor influencing the size and density of zirconia particles is the sintering temperature. It has a direct impact on zirconia particle development, porosity, and density [1, 20]. Among the many ceramic materials, zirconia has the highest rate of porosities in the core (relative refractive index: 2.2). It is possible to increase the translucency of zirconia blocks and produce a more desirable color by reducing these porosities and creating a denser substance. Therefore, by altering the sintering conditions, dental professionals can modify the crystalline structure of zirconia and enhance its optical attributes [4].

Porosity is defined as the percentage of void space in a solid [21]. As displayed in Figure 8, the pre-sintered zirconia exhibited a higher porosity level compared to that of sintered ones. It can be noted that there is a dramatic decrease in the pores content after the sintering process of HT4 89HT and A214 zirconia by 95% 89.5% and 86.3 respectively. These results can be explained by the fact that if the temperature or sintering time are increased, zirconia particle size rises. This, in turn, increases the atomic force between the particles, which reduces micro-porosities in the polycrystalline structure of zirconia. Higher homogeneity of the zirconia crystalline structure is the consequence of both the accumulation of nanocrystalline zirconia particles and the shrinkage of porosities between the particles. This eventually causes an increase in translucency and low refractive index light transmission. This is in agreement with Gómez et al. [22] demonstrated a decrease in porosity by raising the temperature to the point where the relative density went from 50% to 99%, supporting this claim. Moreover, it has been shown by others that zirconia restorations are frequently sintered between 1350°C and 1600°C. Over 1600°C temperatures lead to excessive particle development and an increase in pores level. [4, 23].

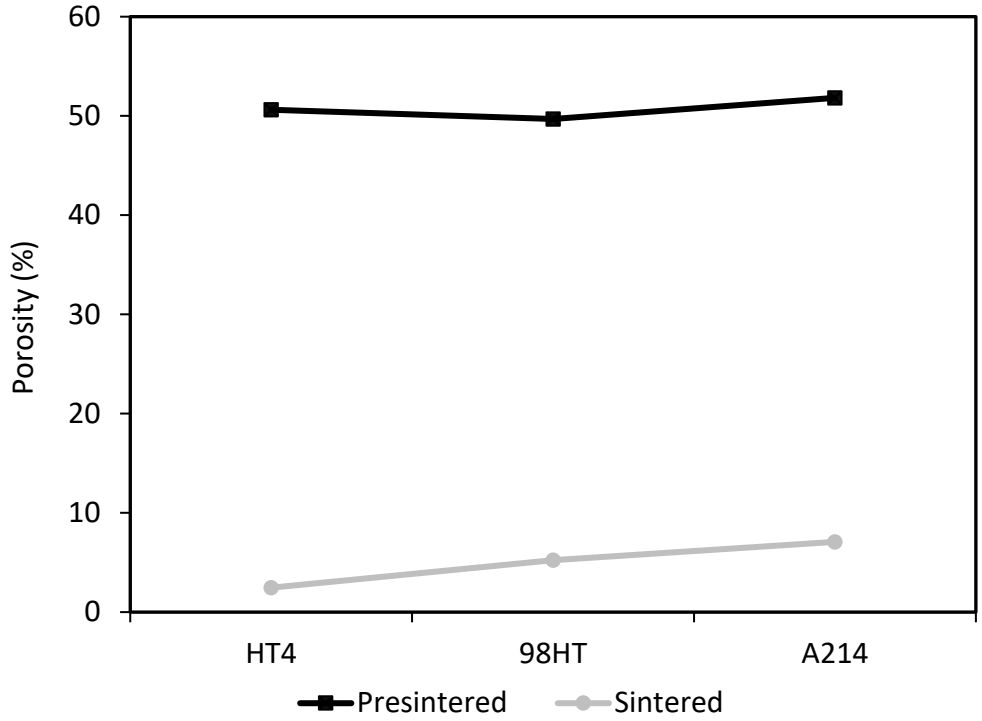

Figure 8 Porosity level of the tested dental zirconia.

- **Sintering Shrinkage**

After the sintering process, the zirconia repair experiences a contraction at roughly 25% and reaches its required dimensions, at which point it achieves its ultimate mechanical characteristics. Because sintering shrinkage prediction and sintering distortion control are critical variables in achieving an exact fit for prosthetic crowns, the shrinkage rates must be estimated and anticipated prior to milling in order to improve the fitting of the restoration. Furthermore, a prosthetic crown's decreased ability to withstand fractures is linked to a poorly fitting margin region [1, 7]. According to Ozden et al. [6], non-uniform shrinkage during sintering could cause the restorations to fit incorrectly.

The linear and volumetric shrinkages of the tested zirconia are depicted in Figure 9. The sintered zirconia samples exhibited a similar manner. When the linear shrinkage of HT4, 98HT and A214 were 20.27%, 20.74% and 19.58% respectively, they achieved 49.37%, 48.5% and 48.27% as volumetric shrinkage. The process of sintering eliminated the empty spaces between the powder particles, resulting in densification and component contraction. As seen in Figures 3-5, a flawless sintered portion was seen at 1530 °C with no cracks or flaws. The most important stage in dimensional variation is sintering, during which shrinkage may occur in the range of 20% to 25% [18]. Such low shrinkage could be attributed to the high powder loading used. Because all of the samples were subjected to identical sintering conditions, a total volumetric shrinkage of almost 50% was discovered after sintering which is in good agreement with previous studies [18, 21]. Another work [14] postulated that when sintering at 1350 °C, the average shrinkage is 18.69%. The mean shrinkage keeps rising to 19.68% when the sintering temperature is raised to 1400 °C. There is only a 5.2% difference in shrinkage between 1400 °C and 1350 °C. After raising the sintering temperature to 1450 °C once more, the mean shrinkage drops to 19.8%. Between 1400 °C and 1450 °C, the shrinkage differential grew to 0.6%. Hirano, et al., [7] concluded that depending on the layer, there are differences in the pre-sintering shrinkage progression degree for zirconia disks. In addition, it is deduced that the rate of shrinkage in the post-sintering process is lower in layers with advanced pre-sintered shrinkage than in layers with slow pre-sintered shrinkage and that the occlusal surface side experiences more shrinkage during post-sintering than the marginal side.

It can be noted that the linear sintering shrinkage of the blocks reduces with an increase in the pre-sintered block densities during the linear sintering shrinkage investigations. The precision of the restoration will be improved if the block has minimal linear sintering shrinkage. Blocks with low linear sintering shrinkage must therefore be made. This will enhance the restoration's fit with the coping that the pre-sintered block produces. Furthermore, an increase in shrinkage by dimension may be interpreted as a sign that the sintering temperature needs to be raised

further to achieve optimal mechanical behavior. When the shrinkage stops, it suggests that raising the sintering temperature is no longer necessary and will just cause the grain size to expand more, which will reduce the toughness of zirconia and is unacceptable [10].

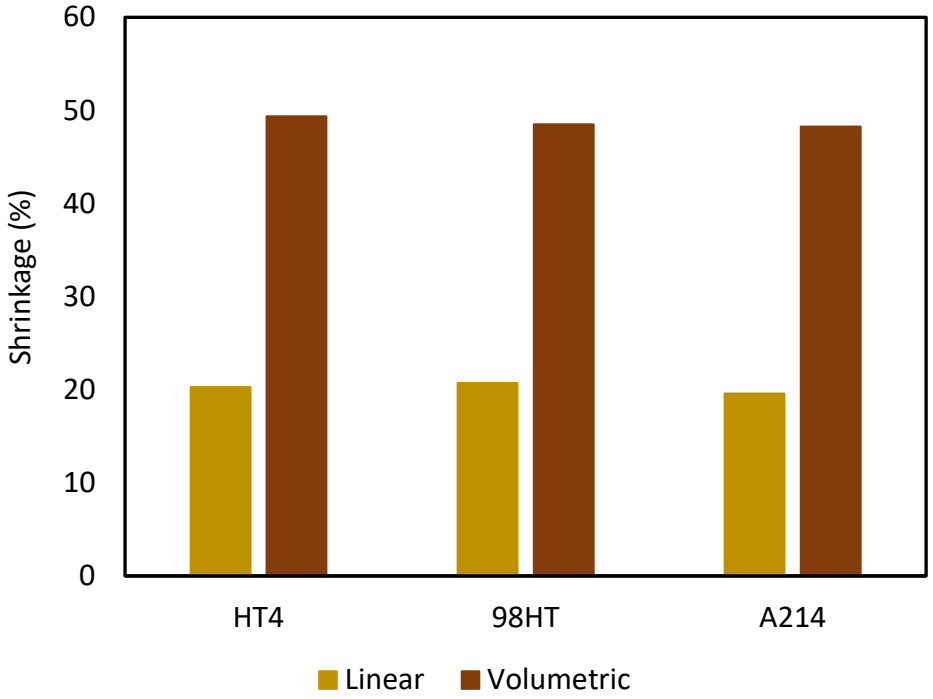

Figure 9 Shrinkage proportion of the processed zirconia.

Conclusion

The experimental blocks in this investigation did not exhibit any appreciable variation in their physical characteristics. It was discovered that their densities before sintering affected the shrinkage behavior in both linear and volumetric sintering. Moreover, the porosity contents were negligible after sintering. The shrinking value of all tested zirconia met the clinical application requirements.

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References:

[1] Rao HM, Kumaraswamy M, Thomas D, Boraiah S, Rana KS (2023). Zirconia in Restorative Dentistry. In: Al-Naib UMB, editor. Zirconia – New Advances, Structure, Fabrication and Applications. Croatia: IntechOpen. p. 1-17.

[2] Aboras M, Muchtar A, Azhari C, Yahaya N (2016). Role of Yttrium Oxide Additive in Stabilizing Zirconia for Dental Applications. 1st International Research Conference on Engineering, Science and Humanities Proceedings. 1-7.

[3] Shim JH, Hong M-H (2022). Effect of various surface treatments by the sol-gel method on the bond and flexural strengths of zirconia-porcelain. Korean Journal of Dental Materials. 49:87-96.

[4] Vafaei F, Shahbazi A, Hooshyarfard A, Najafi AH, Ebrahimi M, Farhadian M (2022). Effect of sintering temperature on translucency parameter of zirconia blocks. Dental Research Journal. 19:1-6.

[5] Abbas MKG, Ramesh S, Tasfy SFH, Lee KYS, Gul M, Aljaoni B (2023). Effect of sintering additives on the properties of alumina toughened zirconia (ATZ). MRS Communications. 13:618-26.

[6] Ozden YE, Guncu MB, Aktas G, Canay S (2022). Effect of sintering time on the marginal and internal fit of monolithic zirconia crowns containing 3-4 mol% Y2O3. BMC oral health. 22:493-501.

[7] Hirano M, Nomoto S, Sato T, Yotsuya M, Hisanaga R, Sekine H (2022). Sintering distortion of monolithic zirconia in 4-unit fixed partial denture: Effect of layered structure and vertical milling area. Journal of the mechanical behavior of biomedical materials. 128:1-9.
[8] Emam M, Metwally MF (2022). Effect of coping materials zirconia or polyetheretherketone with different techniques of fabrication on vertical marginal gap and fracture resistance of posterior crowns with composite veneering. BMC oral health. 23:546.
[9] Suttor D, Bunke K, Hoescheler S, Hauptmann H, Hertlein G (2001). LAVA--the system for all-ceramic ZrO2 crown and bridge frameworks. Int J Comput Dent. 4:195-206.
[10] Oh GJ, Yun KD, Lee KM, Lim HP, Park SW (2010). Sintering behavior and mechanical properties of zirconia compacts fabricated by uniaxial press forming. The journal of advanced prosthodontics. 2:81-7.
[11] Alwade FH, Ismail IJ, Ibrahim FJ (2019). Zirconia in Dental and Other Biomedical Applications - An Overview. International Journal of Medical Research & Health Sciences. 8:30-7.
[12] Li J, Wang X, Lin Y, Deng X, Li M, Nan C (2019). In Vitro Cell Proliferation and Mechanical Behaviors Observed in Porous Zirconia Ceramics. Materials. 9:1-9.
[13] Renold Elsen S, Ramesh T (2016). Shrinkage characteristics studies on conventional sintered zirconia toughened alumina using computed tomography imaging technique. International Journal of Refractory Metals and Hard Materials. 54:383-94.
[14] Moosa JM, Abdulzahraa HG, Zaidan SA, Abed HY (2021). Reinforcing Glaze Layer of Restorative Dental Zirconia by Adding Nano Alumina Ceramics. NeuroQuantology. 19:73-8.
[15] Sinka IC (2007). Modelling Powder Compaction. KONA Powder and Particle Journal. 1:1-22.
[16] Denry I, Kelly JR (2014). Emerging ceramic-based materials for dentistry. Journal of dental research. 93:1235-42.
[17] Wahi A, Norhamidi Muhamad N, Sulong AB, AHmad RN (2016). Effect of Sintering Temperature on Density, Hardness and Strength of MIM Co30Cr6Mo Biomedical Alloy. Journal of the Japan Society of Powder and Powder Metallurgy. 63:434-7.
[18] Amat NF, Muchtar A, Yew HZ, Amril MS, Muhamud RL (2020). Machinability of a newly developed pre-sintered zirconia block for dental crown applications. Materials Letters. 261:1-7.
[19] Hao CC, Muchtar A, Azhari CH, Razali M, Aboras M (2016). Influence of Sintering Temperature on Translucency of Yttria-Stabilized Zirconia for Dental Crown Applications. Jurnal Teknologi. 78.
[20] Liu YC, Lin TH, Lin YY, Hu SW, Liu JF, Yang CC, et al (2022). Optical properties evaluation of rapid sintered translucent zirconia with two dental colorimeters. Journal of dental sciences. 17:155-61.
[21] Yu T, Zhang Z, Liu Q, Kuliiev R, Orlovskaya N, Wu D (2020). Extrusion-based additive manufacturing of yttria-partially-stabilized zirconia ceramics. Ceramics International. 46:5020-7.
[22] Gómez S, Suárez G, Rendtorff N, Aglietti E (2016). Relation between mechanical and textural properties of dense materials of tetragonal and cubic zirconia. Science of Sintering. 48:119-30.
[23] Sanal FA, Kilinc H (2020). Effect of shade and sintering temperature on the translucency parameter of a novel multi-layered monolithic zirconia in different thicknesses. Journal of esthetic and restorative dentistry : official publication of the American Academy of Esthetic Dentistry [et al]. 32:607-14.