

# International Journal of Nanoelectronics and Materials

IJNeaM

ISSN 1985-5761 | E-ISSN 2232-1535



## Study of the Mechanical and Microstructure Properties of Zirconia Reinforced with Glass and Silica

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#### **ABSTRACT**

Artificial teeth were manufactured from zirconia-yttria nanosystems by adding different types of glass to zirconia. The specimens were prepared from tetragonal zirconia crystals stabilized with 3% yttria ( $Y_2O_3$ ) with the addition of soda-lime glass, Pyrex (1,5,10,15)%, and silica (1,2,3,4,5)% in different proportions. The specimens were formed using a pressure-balanced uniaxial hydraulic press (3 tons) the zirconia was sintered at (1500 °C) and the glass-containing systems were fired at (1100, 1200, 1400) °C for (2 hrs.). The structural properties (3 transported at (3 trans

Keywords: Zirconia, Pyrex, Artificial saliva, Dental ceramic

#### 1. INTRODUCTION

Zirconia has become one of the most important materials in dental restorations because of its excellent biocompatibility, non-toxicity, and mechanical and aesthetic properties that simulate those of natural teeth. [1,2].

The aesthetic properties, strong mechanical strength, ease of use, and biocompatibility of glasses and glass ceramics make them important materials in dental restorations. These ceramics, depending on their chemical, physical, and optical properties, can be bonded to natural teeth or to implant abutments and used as inlays, onlays, veneers, partial or full crowns, and bridges.

Dental glass ceramics must be as biocompatible as possible while also achieving or exceeding the properties found in natural teeth in terms of appearance, wear, mechanical strength, and chemical durability in the oral environment. [3].

Color is one of the most essential components of the dentistry industry's attractive appearance. Since dental materials are continuously exposed to various foods and drinks in the oral environment, they need to have the best color stability possible [4]. A smile to be one of the most

important interactive communication skills of a person. [5,6,7].

There has been a description of a new shade guide that is intended exclusively for choosing tooth color, Shade tabs in the color space of real teeth are consistently placed in this system [8,9,10,14].

Conventional color determination systems are tools that assist physicians in determining the color of natural teeth. A contemporary system or manual for the corporation (vita 3D\_master) is based on a comprehensive standard coverage of the tooth's color space, considering the three-dimensional color test, which is based on hue, chroma, and value, using traditional guides, the tooth's color is determined by placing all of the guide's bands next to the patient's teeth and comparing them to the tooth's body. This allows for the selection of the appropriate symbol in terms of hue, which is followed by the determination of the color's intensity, which yields the hue (L.M.R) is examined to see if the natural tooth color is yellowish (L) or reddish (R). Finally, the tooth is checked [11,12,13,15].

#### 2. EXPERIMENTAL

#### 2.1 Material and Method

Specimens were prepared from zirconia-yttria nano powder (American Nanomaterials Research Corporation, 20 nm particle size, 99% purity, pure white powder color). By adding soda lime glass, Pyrex, and silica, a toxicity test was conducted for the glass and the results showed that it is non-toxic as a material for preparing dental ceramics (Environmental Research Center University of Technology-Tissue culture and immunohisto chemical laboratories).

Several systems were prepared in the proportions shown in Table 1. It was formed using the dry alum method by adding (1.5) g of polyvinyl alcohol as a binder. It was compressed by uniaxial hydraulic pressure with a pressure of (3) tons and shaped into a circular disc with a thickness of (4 mm) and a diameter of (20 mm). The soda-lime glass was fired at 1100 °C, Pyrex was fired at 1200 °C, and silica was fired at 1400 °C for 2 h. Dimensions were measured before and after the firing process.

Table 1 Chemical analysis of glass

Glass	SiO <sub>2</sub>	CaO	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	B <sub>2</sub> O
Soda-lime	69-74%	5-12%	12-16%	0-6%	0-3%	-	-
Pyrex	81%	-	4.0 %	-	2.0%	0.5%	13.0%

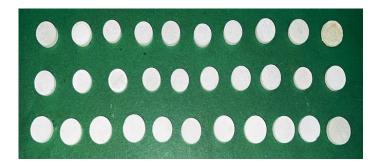


Figure 1. The specimens of dental ceramic.

Table 2 The specimens immersed in artificial saliva, tea, and lemon (citric acid)

Series	Mixture	Code	Series	Mixture	Code
1	Z-G	Z	6	Z-P (10%Pyrex)	ZP0
2	Z-G (5% soda- lime glass)	Z-G5	7	Z-P (15% Pyrex)	ZP5
3	Z-G (10% soda- lime glass)	Z-G10	8	Z-S (1% Silica)	ZS1
4	Z-G (15% soda- lime glass)	Z-G15	9	Z-S (3% Silica)	ZS3
5	Z-P (5% Pyrex)	Z-P5	10	Z-S (5% Silica)	ZS5

#### 2. 2 Preparation of Saliva

The following amounts of artificial saliva were created: (0.002) g ascorbic acid, (0.030) g glucose, (0.580) g NaCl, (0.170) g CaCl<sub>2</sub>, (0.160) g NH<sub>4</sub>Cl, (1.270) g KCl, (0.16) g NaSCN, (0.330) g KH<sub>2</sub>PO<sub>4</sub>, and (0.340) g CaCl<sub>2</sub>.

In 1000 milliliters of distilled water, combine  $Na_2HPO_4$ , 0.200 g urea, and 2.700 g mucin (Bacto-Mucin Bacteriological). A phosphate buffer containing (26.4) mL (0.06) M  $Na_2HPO_{42}H_2O$  and (7.36) mL (0.06) M  $KH_2PO_4$  was added to the solution to titrate it further.

#### 3. MICROSTRUCTURE

#### 3.1 Structural Properties

Scanning Electron Microscope (SEM)

It produces a high-resolution image of the specimen with a three-dimensional appearance and a wide depth of focus. A high-energy electron beam was further employed to scan the specimen surface using a raster scan pattern. The specimen atoms and electrons interact to produce signals that are relayed to a screen that shows details about the specimen surface topography, chemical makeup, and other characteristics

#### 3.2 Physical Test of Specimen

The apparent porosity of artificial dental ceramic was measured by the traditional Archimedes method and the water absorption was calculated according to (ASTM-C373), dry specimens with a mass of (Wd) were placed in hot water for (3hrs) to fill the open pores then cooled the specimens to room temperature by leaving them in the laboratory for (24 hrs) and weighted to have specimens with mass (WS) and immersed in water (Wi) so the (A.P), and (W.A) were calculated by using the equations:

$$(A.P)\% = (Ws-Wd)/(Ws-Wi) \times 100\%$$
 (1)

Where:

Wd is the specimen's dry mass (g), WS is the specimen's water-infiltrated mass (g), and Wi is the specimen's water-immersed mass (g)

$$(W.A)\% = (Ws-Wd)/Wd \times 100\%$$
 (2)

#### 3.3 Mechanical Properties

Diametrical tensile strength

A hydraulic piston is used to calculate the diametrical strength. where the specimen is positioned under the piston vertically. Equation (3) was used to compute the diametrical strength by ASTM-C773.

$$\sigma_{D.S=2F/\pi tD}$$
 (3)

Where:

 $\sigma_{\text{D.S}}$  : diametrical strength (MPa)

F : force (N)

D : diametrical of specimens (mm)
T : the thickness of specimens (mm)

#### 3.4 Color Measurements

Color measurements were objectively approved using the colorimeter (Vita 3D-Master). Measurements were made for all specimens after immersion in artificial saliva, tea, and lemon for three months. Colors were measured according to (Vita 3D-Master),

Traditional color determination systems are devices that help the doctor determine the color of natural teeth, as a modern system or guide for the company (Vita 3D-Master), which is a system that relies on complete standard coverage of the color space of the tooth, taking into account the three-dimensional test for color, which is value, chroma, and hue, which depends on determining the color of the tooth in the traditional guides by passing all the specimens in the guide next to the patient's natural teeth and comparing them with the body of the artificial tooth, so the appropriate symbol is chosen in terms of hue, where the hue (L.M.R) is determined, and then it is checked whether is natural tooth color is yellowish (L) or reddish (R) and (M) all shades have the same hue and the same lightness only the chroma is different.

#### 4. RESULT AND DISCUSSION:

Scanning Electron Microscope (SEM)

An electron microscope was utilized to examine the microscope composition specimen. and the images in Figure 3 shows a scanning electron microscope.

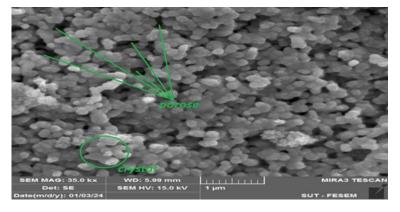
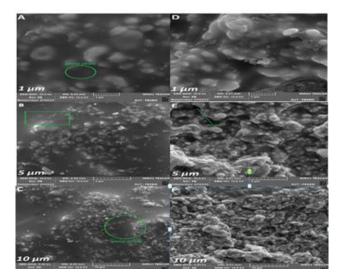


Figure 3. SEM of zirconia-yttria nano system sintering at 1500 °C.

Microstructure image of a specimen of zirconia-yttria nano systems sintered at 1500 °C, the image shows that the crystals are tetragonal in shape, homogenous in size, and regular in dimensions considering their small size, with no significant crystal growth clear. The zirconia-yttria nano system was sintered at 1500 °C, which prevented granular growth. The small, regular shaped crystals improve their mechanical properties because they have higher

mechanical resistance than the grow crystals. A large percentage of small and medium-sized pores have negative effects on dental artificial because they allow food and liquids to enter the tooth's body, creating a perfect situation for the growth of bacteria and the occurrence of diseases. The zirconia structure comprises small, homogeneously distributed semi-tetragonal to tetragonal crystals. Many researchers are interested in this field [16,25].



**Figure 4.** SEM of zirconia-yttria nano-soda lime system firing at 1100 °C, A, B, C SEM of 5% soda lime glass and D, E, F SEM of 15% soda lime glass.

From microstructure image of specimens of zirconia-yttria nano-soda lime glass system that was fired at 1100 °C. It shows that the surface of the specimen does not contain pores or cracks, as the surface is fully dense. The percentage of addition of soda lime glass was useful, the resulting glassy phase played its required role, and an acceptable result was obtained. This image reinforces the validity of the results obtained in all physical and mechanical tests. which is an important point in artificial teeth manufacturing. Because of its smoothness and lack of topography, the surface appears non-porous, which makes it not an ideal medium for the growth of bacteria.

Surface continuity and lack of pores are a very important factor in artificial teeth. Microscopic images showed complete coverage of zirconia crystals by soda-lime glass. The fracture surface showed that the zirconia crystals were completely covered by glass. This means that the fracture surface passes through the glass rather than the interface between the zirconia and the glass. This is a result of the high adhesion between zirconia and glass.

This means that the firing temperature was suitable, which led the glass to become a liquid with a suitable viscosity to fill the pores and airways in the ceramic body [17,18].

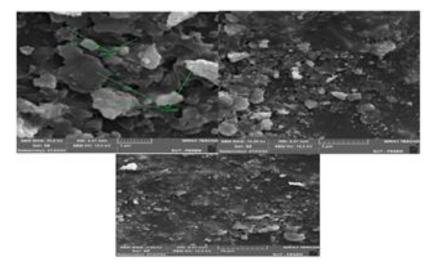


Figure 5. SEM of zirconia-yttria nano-Pyrex system firing at 1200 °C.

From microstructure image of zirconia-yttria nano-Pyrex that was fired at 1200 °C, The system of zirconia-yttria nano-Pyrex. having a small number of pores, rougher surface than zirconia-yttria nano-soda lime glass, and homogeneously distributed, closed, tiny, and unconnected pores that are isolated by zirconia crystals.

Compared to the zirconia-yttria nano-soda lime glass system, although there is not a very high percentage of pores, the surface is very rough, and the fracture passed through the interface between the zirconia and the pyrex, so the pyrex did not completely cover the zirconia crystals after the breakage as a result of the fracture passing through the interface between the zirconia and the pyrex. [19,26].

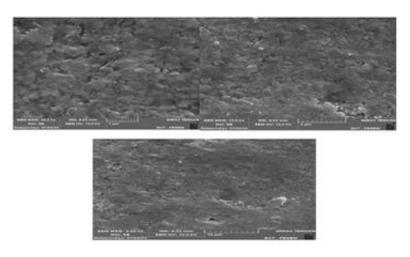


Figure 6. SEM of zirconia-yttria nano-silica system firing at 1400 °C.

From the image of the microstructure of a specimen of zirconia-yttria nano-silica a system that was firing at 1400°C. The images show the presence of a dense surface that results from a glassy phase-filled pore at high temperature at the firing process. This result very good point in artificial dental manufacturing [20,21,27].

### 4.1 Apparent Porosity (A.P) and Water Absorption (W.A)

The apparent porosity and water absorption were measured through equations 1 and 2. In addition, the table 3 shows the effect of adding the percentage of soda-lime glass, Pyrex, and silica on the apparent porosity and water absorption of the zirconia-yttria nano system.

Systems	Apparent Porosity	Water Absorption
Z	0.16	0.08
Z-G5	0.14	0.02
Z-G10	0.09	0.01
Z-G15	0.01	0.002
Z-P5	0.1	0.04
Z-P10	0.06	0.02
Z-P15	0.03	0.01
Z-S1	0.15	0.07
Z-S3	0.13	0.05
Z-S5	0.11	0.04

Table 3 showed a decrease in the porosity ratio with an increase in additives soda-lime glass, Pyrex, and silica, the percentage of water absorption and the ratio of apparent porosity are direct related. Because of this, the apparent porosity directly affects how the water absorption rate behaves; As the apparent porosity decreases, the rate of water absorption decreases. As a result of the addition of molecules of these substances additives fill large gaps between zirconia molecules, according to the table.3, the results showed that zirconia-yttria nano-soda lime glass system is more efficient compared to other systems in filling pores between zirconia molecules due to the formation of the liquid phase. At high temperatures, which works to fill

the pores better, it also works on high agglutination, and therefore we get a low porosity ratio. At high temperatures, phase transitions occur in zirconia, which contributes to a decrease in apparent porosity [23,24].

#### 4.2 Diametrical Strength

The diametrical strength was calculated according to equation (3), Figure (7) shows the effect of adding sodalime glass, pyrex, and silica on the diametrical strength of zirconia-yttria nano system.

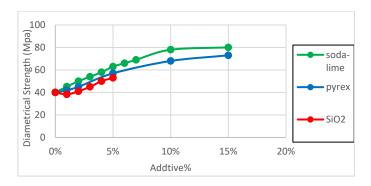


Figure 7. The effect of Soda-lime glass, Pyrex, and Silica addition on the diametrical strength of zirconia-yttria nano-systems.

Figure 7 shows that the diametrical strength increases after adding different types of glass to the zirconia-yttria nano system because of the melting of glass at firing temperatures and the formation of a liquid phase that works to filling the pores and connects the ceramic phases after hardening. A decrease in the percentage of porosity improves the mechanical properties as the pores represent an area of weakness and mechanical defect within the tooth's body. The zirconia-yttria nano soda lime glass system has the highest diametrical strength reaching 80 Mpa because it has a lower porosity. Referring to the scanning electron microscope images and nano zirconia-

yttria Pyrex system and nano zirconia-yttria silica system have the lowest diametrical strength, the soda-lime glass's efficiency was higher in filling the pores and forming a solid, strong, cohesive ceramic body,, diametrical strength test was conducted on the systems before and after immersion in artificial saliva for 3 months, and the results are shown in Table 4, from a table showing that there was no change in the diametrical strength. This means that the diametrical strength was not affected by the immersion of the teeth [17].

**Table 4** Effect of soda-lime glass, Pyrex, and silica addition on diametrical strength of zirconia-yttria nano system before and after immersion

Systems	Before immersion (MPa)	After immersion (MPa)
Z	40	41
Z-G5	63	62
Z-G10	78	76
Z-G15	80	82
Z-P5	57	59
Z-P10	68	67
Z-P15	73	71
Z-S1	38	39
Z-S3	45	46
Z-S5	53	52

#### 4.3 Color Stability

Using Vita Shade 3D\_MASTER, three values were obtained for the color measurement (L.M.R), where (L) denotes the yellowish or reddish color (R), (M) When matching the specimen to the groups in the guide, the color intensity Chroma illustrative in table 5, where the Chroma represents the color saturation, is shown. This is considered the reference point where the color is neither excessively yellowish nor toward redness, as it is considered the closest

color to the color of the natural teeth common among patients. Following the determination of the color intensity.

Chroma Specimens Chroma Hue **Specimens** Hue Z 2 M2 Z-P10 M1 Z-G5 1 M1 Z-P15 1 M2 Z-G10 1 M1 Z-S1 2 M1 Z-G15 1 M2 Z-S3 1.5 M1 Z-P5 1.5 2LZ-S5 1.5 M1

Table 5 Chroma and hue of artificial teeth after immersion

From Table 5 The Vita 3D\_master shade device's results showed that the symbols and numbers indicated colors that were similar to those of natural teeth. Zirconia-yttria nano specimens showed a slight change in color from bright white to white closer to the degree of natural teeth after being immersed in tea for 90 days. The systems that contain glass, are the least colored due to its properties. The system (Z-G5, Z-G10, Z-G15) that contains glass the least colored because it is less porous and does not absorb liquids. In addition, because of the glass's low adhesion, the colors do not adhere to it and the glass cannot be tinted yet, Glass cannot be colored after firing except at the beginning of the manufacturing process, as the results of the system that contains glass do not change color easily appear, as it takes longer than the rest of the materials. Finally, the specimens that were immersed in saliva and lemon did not cause any dyeing. The glass covered the teeth from the outside. The specimen's zirconia-yttria nano are considered among the specimens most susceptible to staining because they do not contain glass. Results showed that samples containing glass were less likely to change color after immersion. [22].

#### 5. CONCLUSION

- The effect of artificial saliva, tea, and lemon on artificial teeth prepared from the zirconia yttria nano system was studied by adding different ratios of glass.
- 2. The results showed that the mechanical properties of diametrical strength were not affected after being immersed in artificial saliva and tea for 90 days.
- 3. The color properties were improved by adding glass to the zirconia yttria nano system. It was noted that artificial saliva and lemon after immersion did not affect the artificial teeth. A very slight change in the color of the artificial teeth was also observed after immersion in tea for three months.
- 4. The results showed that the zirconia yttria nanosoda-lime glass system is the best due to the very low porosity of the soda-lime glass and the low adhesion property, so a slight color change was observed.
- 5. Zirconia is more prone to discoloration than glass. glass is transparent, devoid of pores, and has a smooth surface. It has a very low tendency to discoloration and a very low wettability.

#### **ACKNOWLEDGMENTS**

The authors extend their appreciation to the researchers supporting my work (Dr. Azhar M. Haleem and Dr. Nehia N. Hussein).

#### **REFERENCES**

- [1] S. M. Saad, N. N. Hussien, and E. M. Hadi, "Enhancing the mechanical qualities of dental ceramics based on nano Zirconia and Yttria and other additive materials," 2024, p. 050001. doi: 10.1063/5.0183665.
- [2] H. Lin, C. Yin, and A. Mo, "Zirconia Based Dental Biomaterials: Structure, Mechanical Properties, Biocompatibility, Surface Modification, and Applications as Implant," Frontiers in Dental Medicine, vol. 2. Frontiers Media S.A., 2021. doi: 10.3389/fdmed.2021.689198.
- [3] S. J. Saint-Jean, "Dental Glasses and Glass-ceramics," in *Advanced Ceramics for Dentistry*, Elsevier Inc., 2014, pp. 255–277. doi: 10.1016/B978-0-12-394619-5.00012-2.
- [4] S. M. Majeed, D. S. Ahmed, and H. S. Ahmed, "Studying the effect of MWCNTs/zirconia nano composite in reducing tumor cells," in *AIP Conference Proceedings*, American Institute of Physics Inc., 2019. doi: 10.1063/1.5138551.
- [5] F. N. Hattab, M. A. Qudeimat, and H. S. Al-Rimawi, "Dental discoloration: An overview," *Journal of Esthetic and Restorative Dentistry*, vol. 11, no. 6, pp. 291–310, 1999, doi: 10.1111/j.1708-8240.1999.tb00413.x.
- [6] J. M. Moosa, H. G. Abdulzahraa, S. A. Zaidan, and H. Y. Abed, "Reinforcing glaze layer of restorative dental zirconia by adding nano alumina ceramics," *NeuroQuantology*, vol. 19, no. 6, pp. 73–78, 2021, doi: 10.14704/nq.2021.19.6.NQ21071.
- [7] S. J. Saint-Jean, "Dental Glasses and Glass-ceramics," in *Advanced Ceramics for Dentistry*, Elsevier Inc., 2014, pp. 255–277. doi: 10.1016/B978-0-12-394619-5.00012-2.
- [8] Tooth discolouration and staining.
- [9] J. J. Ten Bosch and J. C. Coops, "Tooth Color and Reflectance as Related to Light Scattering and Enamel Hardness," *J Dent Res*, vol. 74, no. 1, pp. 374–380, 1995, doi: 10.1177/00220345950740011401.

- [10] A. Joiner, N. M. Jones, and S. J. Raven, "INVESTIGATION OF FACTORS INFLUENCING STAIN FORMATION UTILIZING AN IN SITU MODEL," 1995.
- [11] N. N. Veček, M. Par, E. K. Sever, I. Miletić, and S. J. Krmek, "The Effect of a Green Smoothie on Microhardness, Profile Roughness and Color Change of Dental Restorative Materials," *Polymers (Basel)*, vol. 14, no. 10, 2022, doi: 10.3390/polym14102067.
- [12] E. Cal, P. Güneri, and T. Kose, "Comparison of digital and spectrophotometric measurements of colour shade guides," *J Oral Rehabil*, vol. 33, no. 3, pp. 221–228, Mar. 2006, doi: 10.1111/j.1365-2842.2005.01560.x.
- [13] T. P. Van Der Burg&, J. J. Ten Bosch, P. C. F. Borsboom, and W. J. P. M. Kortsmit, "A comparison of new and conventional methods for quantification of tooth color".
- [14] W. M. Johnston and E. C. Kao, "Assessment of Appearance Match by Visual Observation and Clinical Colorimetry," 1989.
- [15] A. Joiner, "Tooth colour: A review of the literature," *J Dent*, vol. 32, no. SUPPL., pp. 3–12, 2004, doi: 10.1016/j.jdent.2003.10.013.
- [16] S. M. Saad, M. Hadi, and N. N. Hussien, "Zirconia-Yttria System for Manufacturing Ceramic Dental," 2022.
- [17] A. M. Bader and E. M. Hadi, "Integrated modification of silicon carbide ceramic armor via kaolin and Sodalime glass for enhanced ballistic protection: Fabrication, characterization, and performance analysis," Proceedings of the Institution of Mechanical Engineers, Part E: Journal of Process Mechanical Engineering, 2024, doi: 10.1177/09544089241232801.
- [18] A. Shearer, M. Montazerian, B. Deng, J. J. Sly, and J. C. Mauro, "Zirconia-containing glass-ceramics: From nucleating agent to primary crystalline phase," *International Journal of Ceramic Engineering and Science*. John Wiley and Sons Inc, 2024. doi: 10.1002/ces2.10200.
- [19] K. W. Song, I. S. Park, G. S. Shin, S. K. Lyu, M. H. Lee, and T. S. Bae, "Fracture strength of borosilicate glass melt infiltrated zirconia 3-unit bridge," *International Journal of Precision Engineering and Manufacturing*, vol. 14, no. 9, pp. 1607–1613, Sep. 2013, doi: 10.1007/s12541-013-0217-5.

- [20] T. Nakamura *et al.*, "The effect of adding silica to zirconia to counteract zirconia's tendency todegrade at low temperatures," *Dent Mater J*, vol. 30, no. 3, pp. 330–335, May 2011, doi: 10.4012/dmj.2010-142.
- [21] M. Lakusta *et al.*, "The Effect of a Small Amount SiO2 on Sintering Kinetics of Tetragonal Zirconia Nanopowders," *Nanoscale Res Lett*, vol. 12, 2017, doi: 10.1186/s11671-017-2178-6.
- [22] K. M. Lehmann, A. Devigus, C. Igiel, and M. Weyhrauch, "Article in European journal of esthetic dentistry: official journal of the European Academy of Esthetic Dentistry," 2012. [Online]. Available: https://www.researchgate.net/publication/230712 2722.
- [23] A. O. Zhigachev, V. V. Rodaev, and D. V. Zhigacheva, 2019 "The effect of titania doping on structure and mechanical properties of calcia-stabilized zirconia ceramic," Journal of Materials Research and Technology, vol. 8, no. 6, pp. 6086–6093, Nov., doi: 10.1016/j.jmrt.2019.10.002.
- [24] S. Zinelis, A. Thomas, K. Syres, N. Silikas, and G. Eliades, 2010" Surface characterization of zirconia dental implants," Dental Materials, vol. 26, no. 4, pp. 295–305, Apr., doi: 10.1016/j.dental.2009.11.079.
- [25] E. M. Hadi and H. J. Abdul-Hussien, "Preparation of ceramic foam from porcelanite by using simple direct foaming method," in AIP Conference Proceedings, American Institute of Physics Inc., Jul. 2019. doi: 10.1063/1.5116934.
- [26] R. Abdullrazzaq, M. F. Hadi Al-Kadhemy, and E. M. Hadi, "Preparation Foam Brick from Iraqi Local Clay," in Journal of Physics: Conference Series, IOP Publishing Ltd, Mar. 2021. doi: 10.1088/1742-6596/1818/1/012071.
- [27] F. H. Gata and E. M. Hadi, "Preparing a thermal mortar from local Iraqi raw materials and studying thermal properties," in AIP Conference Proceedings, American Institute of Physics Inc., Mar. 2023. doi: 10.1063/5.0110144.