

Mechanical Properties of Polymer-Infiltrated Fluorapatite Glass Ceramics Fabricated from Clam Shell and Soda Lime Silicate Glass

ESRA KUL^{1*}, KHAMIRUL AMIN MATORI², ŞÜKRAN KARADENİZ³, ERSIN ARSLAN⁴, MEHMET ERTUĞRUL⁵

¹Department of Prosthodontics, Faculty of Dentistry at Atatürk University, Yakutiye/Erzurum, 25240, Turkey

²Department of Physics, Universiti Putra Malaysia, Serdang, Selangor, 43400, Malaysia

³Department of Metallurgical and Materials Engineering, Atatürk University, Yakutiye/Erzurum 25240, Turkey

⁴Department of Mechanical Engineering, Faculty of Engineering, İstanbul Aydın University, Küçükçekmece/İstanbul 34295, Turkey

⁵Department of Electrical and Electronic Engineering, Faculty of Engineering, Atatürk University, Yakutiye/Erzurum, 25240, Turkey

Abstract: *Can polymer-infiltrated fluorapatite glass ceramic produced from waste materials, such as clam shell and soda lime silicate glass, be used in prosthetic rehabilitation? The purpose of this study is to investigate the effect of Si/Ca ratio on the mechanical properties of Nb-Bi-Ce doped polymer-infiltrated fluorapatite ceramic networks (PICNs) produced from clam shell (CS) and soda lime silicate (SLS) glass by conventional melt-quench technique, used as a dental ceramic. PICNs comprising Si/Ca at four different weight percentage ratios: 1.27% (PICN-1), 2.15% (PICN-2), 4.12% (PICN-3) and 12.6% (PICN-4) were prepared (n=10). The powder mixtures in four different ratios were compressed in a rectangular prism shaped mold and subjected to equal pressure from all sides in a cold isostatic press, followed by heat treatment at 750 °C for 3 h. Then complete vacuum infiltration was done with a polymer mixture then low temperature firing was applied leading to the formation of the PICNs. Wear behavior and 3-point bending properties were evaluated and specimens were analyzed using scanning electron microscopy (SEM). The friction and wear properties were determined by means of a pin-on-disk tribotester. Since the flexural strength test did not show normal distribution, Kruskal Wallis test was performed in independent groups, $p < 0.05$. There is a significant difference of flexural strength values between the groups ($p = 0.032$), it was determined that the difference was between the PICN-1 and PICN-4 groups ($p = 0.037$). In the analysis of wear scar, abrasion grooves were also observed. The results of this study showed that high Ca and Si content in CS and SLS glass, respectively, encourages the use of waste materials in the production of PICNs intended to be used in prosthetic rehabilitation. The composition of PICNs produced from waste materials affected flexural strength and wear behavior. Increasing the Si/Ca ratio was found to support the mechanical properties of experimental PICN and that experimental PICNs can be considered as high potential candidates for dental applications.*

Keywords: *polymer-infiltrated fluorapatite glass ceramics, clam Shell, soda lime silicate glass, wear, flexural strength*

1. Introduction

Since the late 1960s when Bioglass 45S5 was first introduced by Larry Hench, there has been a great deal of interest in various types of bioglass, widely used in different dental and biomedical applications for their specific biological properties, such as being biocompatible and inert [1-4]. Recently, new ceramic materials of porous polymer-infiltrated ceramic networks (PICNs) have been developed and they were indicated for the construction of minimally invasive restorations, inlays, onlays, veneers, and implant supported crowns [5,6]. Improved mechanical and aesthetic properties of PICN, which are associated with better microstructure, prolong the life of dental restorations [7-9]. Waste materials, such as clam shell (CS) and soda lime silicate (SLS), can be recycled to produce a product beneficial to

*email: esra.kul@atauni.edu.tr

society. CS and SLS comprise high CaO and SiO₂ content, respectively [10].

The aim of using recycled materials, such as CS (collected as it occurs in nature) and SLS waste, is to reduce the cost of production. Fluorapatite glass-ceramics could be a potential product to be produced from these waste materials and is based on three major compounds: calcium fluoride (CaF₂), alumina (Al₂O₃), silica (SiO₂). Fluorapatite glass-ceramics used in the present study is bioglass-based, and despite being one of the best bioglasses, which are promising excellent dental materials of the future, it has high production costs because of its high melting point [11, 12]. The incorporation of SLS can lower the melting point of the glass system, but also reduces its strength [10]. To overcome this problem and improve the mechanical properties of the glass, other compound oxides, such as CaO and P₂O₅, can be added to the glass system [13]. Besides, the bioglass itself can be crystallized as bioglass-ceramic to improve its physical, structural and mechanical properties with proper heat treatment [14, 15]. When a polymer matrix structure is infiltrated into a sintered porous ceramic, good mechanical and optical properties that allow for a wide application in prosthetic rehabilitation can be obtained and, hence, it could be indicated for monolithic restorations [16, 17]. Since the wear behavior and flexural strength properties of a material is affected by its composition, examination of friction and wear behavior is important in the development of dental materials [18].

The main purpose of the present study is to synthesize CaO and SiO₂ from waste materials, CS and SLS glass, and to compare the mechanical and structural properties of glass-ceramic compositions with different contents, produced by melt- quench technique. The null hypothesis of the study suggests that the mechanical properties of the experimental ceramics are affected by their composition.

2. Materials and methods

To prepare PICN's, 'Anadara Granosa' strain CS from Pantai Cahaya Bulan in Kelantan, Malaysia, and SLS glass from waste glass bottles from a restaurant near UPM Serdang were collected and cleaned. P₂O₅ (99.99%) and Al₂O₃ (99.5%) were supplied from Alpha Aesar, and CaF₂ (99.95%) from R&M Chemicals. CSs were calcined at 900°C for 2 h to obtain CaO and to remove CO₂. Afterwards, both CaO and SiO₂ glass were ground to fine powder, which was then sieved through a 45 μm sieve. A homogeneous mixture was prepared with different weight percentages of CaO, SiO₂, 18 wt% Al₂O₃, 13 wt% CaF₂ and 18 wt% P₂O₅, 4wt% CeO₂, 3 wt% Bi₂O₃, 3 wt% Nb₂O₅ and was melted in a furnace at 1500°C, for 4 h (Table 1). The molten mixture, removed from the furnace, was quenched in cold water to obtain glass frit. Glass frit was dried, crushed and sieved through a 45 μm sieve. The CS and SLS glass, the starting materials of the PICNs, were characterized by energy dispersive X-ray (EDX) analysis performed on a FEI brand NOVA™ NanoSEM 230 field-emission scanning electron microscope (FE-SEM) to examine the percentage of elements present in the samples. The powders were milled in a Retsch PM100 planetary ball milling system (with zirconium containers and balls with a ball-to-powder weight ratio of 3:1) at 200 rpm, for 5 h. The weight ratio of the ground bioglass powder mixed with NH₄HCO₃ particles as a space-holder was adjusted to 5%. To improve bonding between ceramics, methacryloxypropyltrimethoxysilane (MPS) (Sigma Aldrich, St. Louis, MO, USA) was applied, followed by heating at 140°C, for 6 h. The mixtures were then uniaxially compacted under 2.5 tons in a stainless steel mold (20 mm x 10 mm x 4 mm) for 1 min to shape. It was then re-compacted under 200 MPa pressure, for 5 min, using a cold isostatic press. In the two-stage sintering process, during the first step, the space-holder particles were burnt off at 180°C, for 2 h, and, in the second step, the material was kept under fire, at 750°C, for 3 h. Then complete vacuum infiltration was done with a polymer mixture of 99 wt% ethylene glycol dimethacrylate (EGDMA) and 1wt% Polyethyleneimine (PEI) (supplied from Sigma-Aldrich); after vacuum release, the infiltrated blocks were maintained at 70°C for 8 h to form a polymer network, leading to the formation of the PICNs [2, 4, 12, 14]. Ten bar specimens (20 mm x 10 mm x 4 mm) from each of the 4 experimental ceramic materials were prepared and specimen sizes were checked with a micrometer and inspected for chipping at the edges. All specimens were kept in water for 30 days, then, they were allowed to air-dry, at room temperature for 24 h prior to testing. The 3-point bending test was carried out with a universal testing device (Instron) and a 1-kN load cell, according to

ISO 10477:2020 [19]. Each specimen was positioned on a metal fixture with a 10 mm support span and centered under the loading rod. The test was run to failure with a crosshead speed of 0.5 mm/min. The maximum load (N) was recorded by the control software and calculated in MPa (*FS*) using the following formula:

$$FS = \frac{3FI}{2wh^2}$$

where *F* represents the maximum load (N), *I* the support span (mm), *w* the width of the bar at break (mm), *h* the height of the bar (mm).

Table 1. Chemical composition of the experimental glasses

Batch	Weight percentage (wt.%)			
	PICN-1	PICN-2	PICN-3	PICN-4
CS	18	13	8	3
SLS	23	28	33	38
CaF ₂	13	13	13	13
P ₂ O ₅	18	18	18	18
Al ₂ O ₃	18	18	18	18
CeO ₂	4	4	4	4
Bi ₂ O ₃	3	3	3	3
Nb ₂ O ₅	3	3	3	3
Polymer	5	5	5	5

Wear tests were performed on a Tribotechnic pin-on-disc tribometer, in dry conditions and under uniform environmental conditions. The main wear parameters affecting the test were optimized as: a 6-mm diameter tungsten carbide (WC) ball, 4 N load, 20 mm/s speed and test distance of 50 m¹. The experiment was repeated three times under the same parameters. The wear scars and the structures of specimens were examined by SEM, whereas the WC balls were examined by an optical microscope to analyze the effects of wear mechanisms involved in the process (Figures 1 and 2). The surface average roughness, *Ra* and the surface profiles of the wear tracks on the layers were measured with a Mahr profilometer. Measurements were made from different directions and the mean *RA* value was determined. The wear volume was calculated using the profiles obtained from the wear track cross-section, and thus the wear rate was attained. It is shown in eq.1 and eq.2.

$$WSV \text{ (mm}^3\text{)} = ta \text{ (mm}^2\text{)} \times RMA \text{ (mm)} \quad (1)$$

$$WR \text{ (mm}^3\text{/Nm)} = (WSV \text{ (mm}^3\text{)}) / (F \text{ (N)} \times sd \text{ (m)}) \quad (2)$$

where; *WSV*: worn surface volume, *ta*: trace area, *RMA*: reciprocating motion amount, *WR*: wear rate, *F*: load, *Sd*: sliding distance

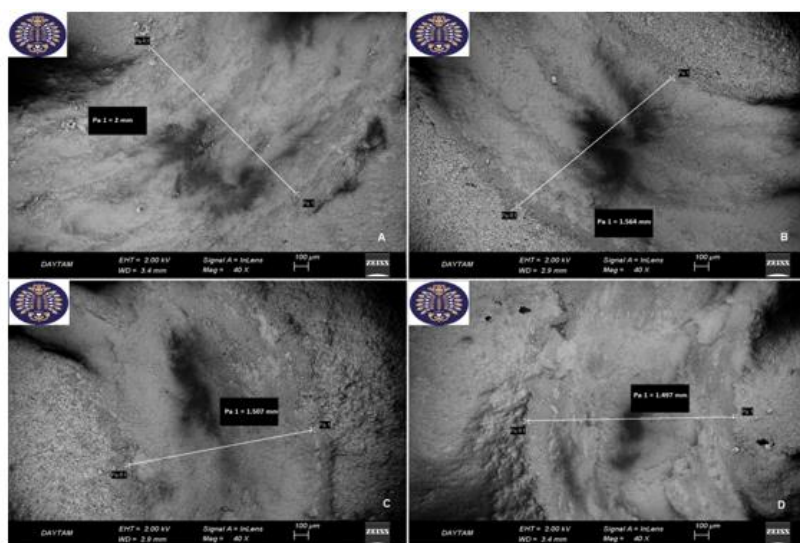


Figure 1. SEM images of the wear track for (a) PICN-1, (b) PICN-2, (c) PICN-3, (d) PICN-4. It is seen that, with increasing Si content (from a to d), the less wear is observed

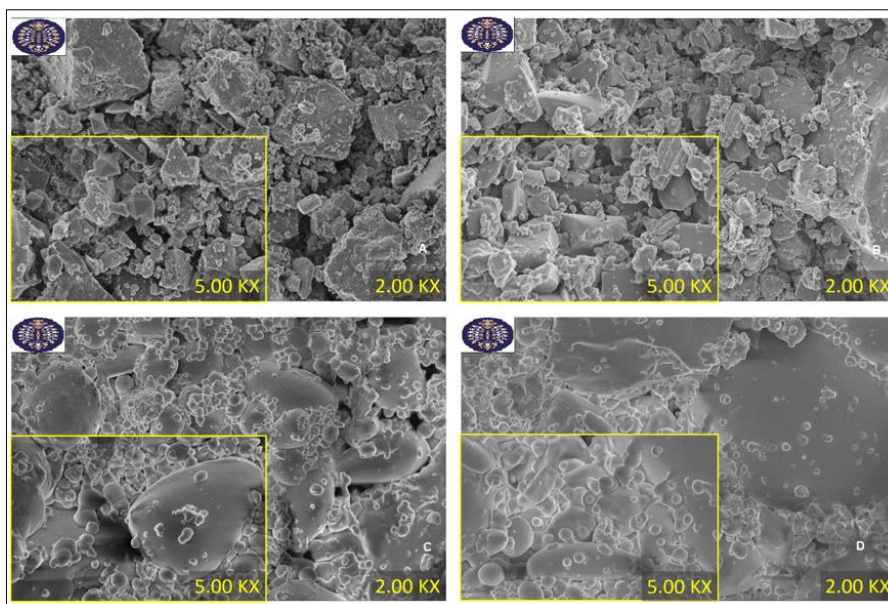


Figure 2. SEM images of groups; (a) PICN-1, (b) PICN-2, (c) PICN-3, (d) PICN-4. It is seen that, with increasing Si content (from a to d), the more the pores are filled and more homogeneous the ceramic structure becomes

3. Results and discussions

Since the data of the results of the flexural strength test did not show normal distribution, Kruskal Wallis test was performed in independent groups, $p < 0.05$. The difference of flexural strength values between the groups was significant ($p = 0.032$), with the difference between the PICN-1 and PICN-4 groups was ($p = 0.037$) (Table 2, Figure 3). The average coefficient of friction (COF) values that recorded during all the test are given in Table 3. COF values of the four groups were close and similar to each other. The extents of wear damages were similar in PICN-2, PICN-3 and PICN-4, but it was more intense in PICN-1. The low amount of SiO_2 in the groups was observed to have increased the wear rates and decreased surface roughness, accordingly, Group 1 showed more damages at the surface, with wide wear scar. As can be seen in the SEM images of worn surfaces given in Figure 2, the width of worn surface is 2 mm in PICN-1, 1.564 mm in PICN-2, 1.507 mm in PICN-3 and 1.497 mm in PICN-4 (Figure 5). The same abrasive wear mechanism can be said to have occurred in all groups. The experiment was repeated three times under the same parameters and the results were very similar for all groups. In abrasive wear, due to the abrasion of the contacting surfaces, and the consequent detached particles, the area inside the wear track is characterized by severe material damage and wear grooves. In the analysis of wear scar, abrasion grooves, ridges and chips were also observed. The wear mechanism can also evolve from a two-body to a three-body wear. Yet, in all cases, material transfers from the batches to the balls, which characterizes the adhesion process, could be observed even with the naked eye, during the abrasion tests (Figure 4).

Table 2. Results of statistical analysis of flexural strength test

Group	Mean MPa	Standard Deviation MPa	Minimum MPa	Maximum MPa
PICN-1	59.7	17.8	31.0	89.2
PICN-2	87.3	27.1	40.6	126.4
PICN-3	109.3	51.8	42.4	183.8
PICN-4	120.9	56.3	40.4	210.4

The use of glass ceramics in prosthetic dentistry is preferred due to the aesthetic appearance, mechanical strength, bio-compatibility and functionality [11, 20]. Today, the use of waste materials,

such as fly ash, eggshell, SLS glass and CS in production of glass ceramic composites attracts the attention of researchers [10, 13].

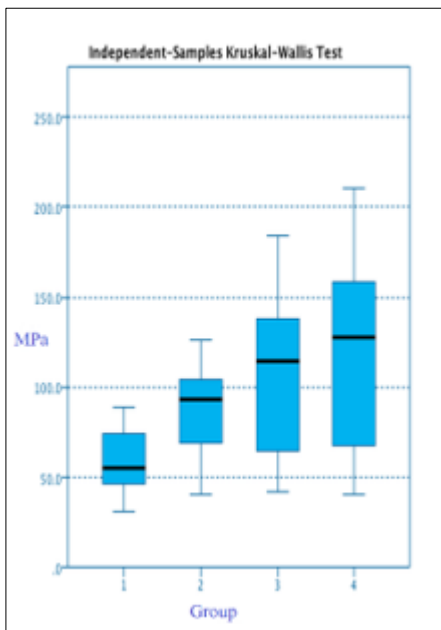


Figure 3. The results of flexural strength test of different groups [4]

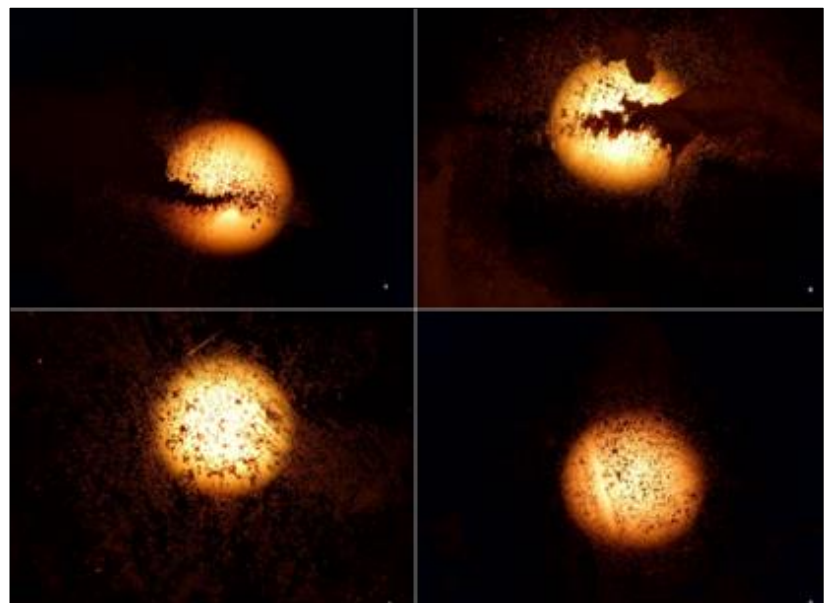


Figure 4. Optical microscope images of the ball for (a) PICN-1, b) PICN-2, (c) PICN-3, (d) PICN-4. It is seen that, with increasing Si content (from a to d), the less material transfer from the batches to the balls occurred [4]

Table 3. Surface Roughnessⁱ, COF and wear rate of groups [3]

Group	Surface roughness (μm)	COF	Wear Rate (mm ³ /N.m)
PICN-1	4.461	0.410	6.4087 E ⁻⁰²
PICN-2	3.994	0.548	3.9944 E ⁻⁰³
PICN-3	2.053	0.542	3.6804 E ⁻⁰³
PICN-4	1.9	0.529	3.3233 E ⁻⁰³

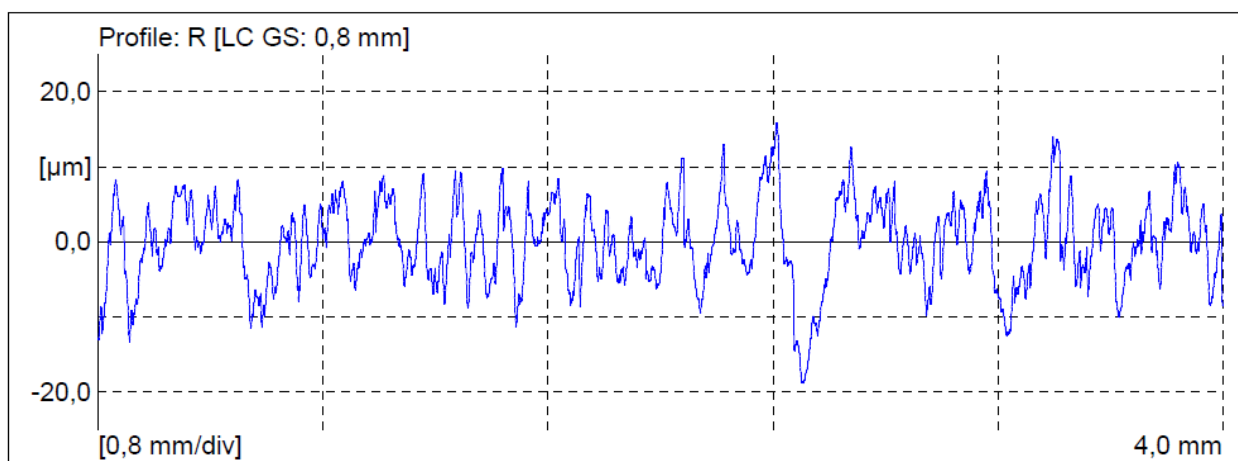


Figure 5a. Surface roughness profiles of PICN-1

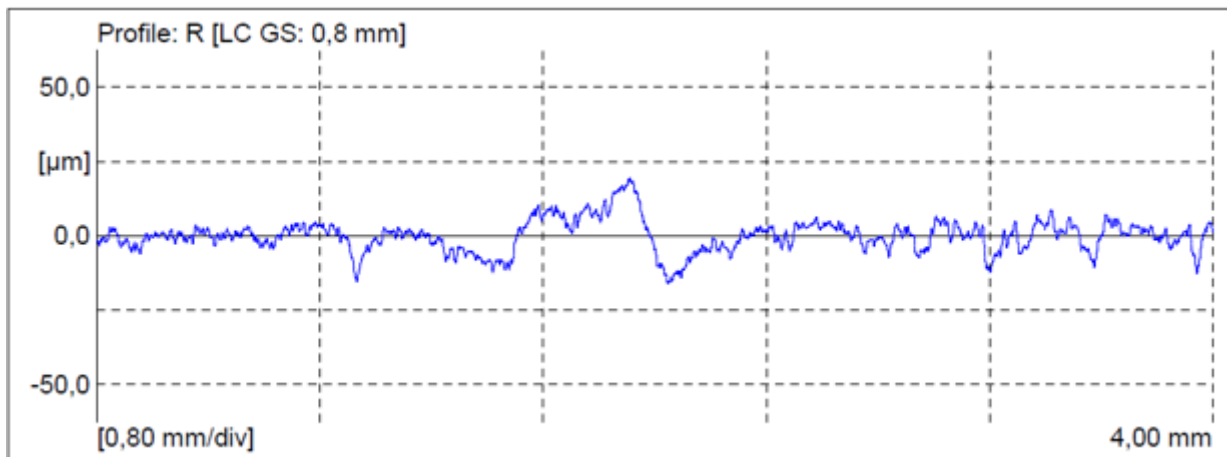


Figure 5b. Surface roughness profiles of PICN-2

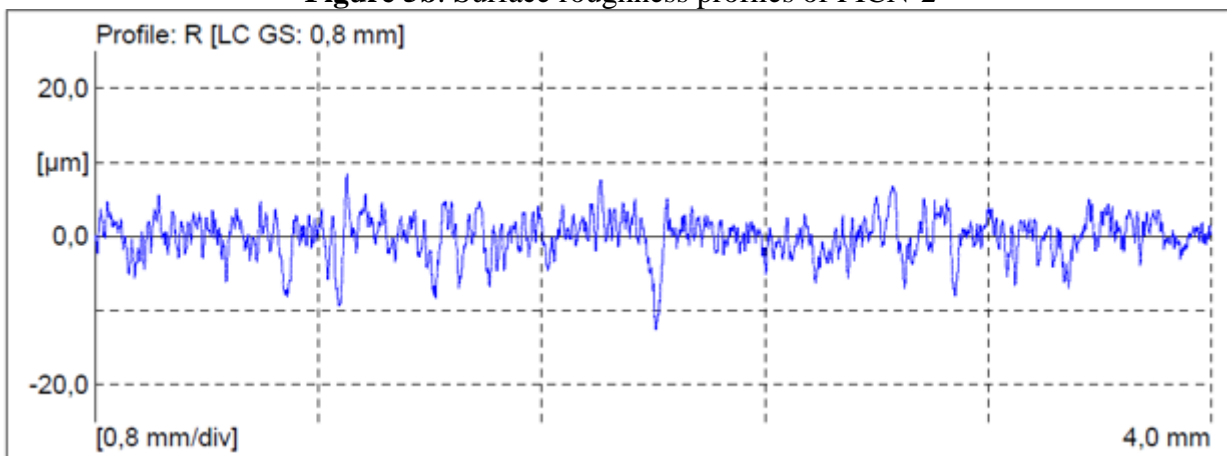


Figure 5c. Surface roughness profiles of PICN-3

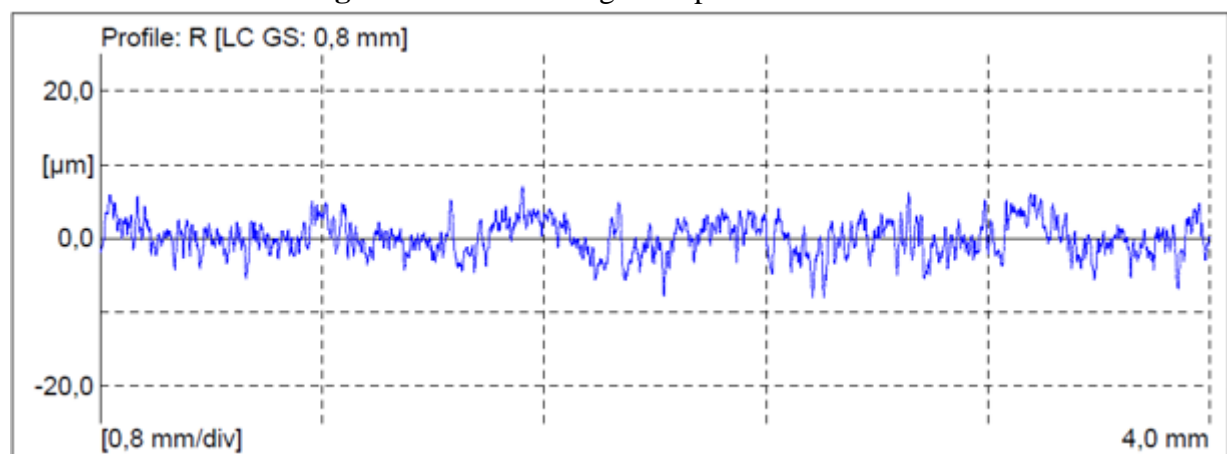


Figure 5d. Surface roughness profiles of PICN-4

As using/reusing waste materials is a new way to minimize the harm to the environment, the present study was aimed at using CaO and SiO₂ obtained from waste materials, such as CS and SLS glass, to produce PICN. Attempts have been made to reduce the high cost of pure chemicals, such as calcite and silicate. Additionally, the limited number of studies on the production of PICN composites by using waste materials as the starting materials also causes such studies to be extensive and exhaustive [10]. According to the results of this study, it can be said that the elements and ratios of PICN play a very important role in the design and production of SiO₂-CaO-Al₂O₃-CaF₂-P₂O₅ bioglass-ceramic systems suitable for dental use. Li et al. [1] reported that the incorporation of CaO to the composition helps promote crystal phase formation. CaO can be considered as the network modifier, which is presented as

ions that replace the glassy network [21]. SiO_2 is responsible for the bonding of the glass network and is present in the composition as a network former that helps lower the melting point of the glass [8]. In addition, Al_2O_3 is added to increase the mechanical strength of the glass composition [22]. The presence of CaF_2 as a source of fluoride ion is also important, as the fluoride ion promotes remineralization and can replace the hydroxyl ion in apatite formation. The presence of apatite phase $\text{Ca}_3(\text{PO}_4)_2$, which interacts well with dentin and enamel, is important in the composition of a glass ceramic material that can be used in dental applications. P_2O_5 and CaO are added to this bioglass system to improve its mechanical properties [20]. Alumina is added to increase chemical stability [20] whereas ZrO_2 adds strength, and Tb_2O_3 and 2% CeO_2 have a slight effect on the overall optical properties of the material [21]. Fluorapatite glass-ceramics doped with small amounts of niobium oxide can crystallize into a very fine binary microstructure of forsterite polygonal crystals together with fluorapatite spherical crystals [15, 23]. In multicomponent bioactive silicate glasses, calcium is an important component in increasing bioactivity and when incorporated as fluoride, it causes a decrease in the glass transition temperature by increasing crystallization temperature in the presence of phosphorus pentoxide and efficiently improves the process thereby [24]. Nb-doped fluorapatite glass ceramics sintered at low temperatures have been shown to be excellent candidates for producing bioactive ceramic scaffolds [23]. Normally, glass has low mechanical properties. However, its mechanical properties can be improved by adding other elements to the glass system and by sintering the glass ceramic system at an optimum temperature. In our previous study, we reported that increasing the Ca/Al ratio had supported low temperature sintering of fluorapatite glass-ceramics, which are conventionally difficult to sinter [10].

With novel PICNs created with computer-aided design and computer-aided manufacturing, [25] restorations with sufficient strength, good internal compatibility and less opposing tooth wear can be made [26] as compared to existing ceramics [27, 28]. It can be said that there has been a shift more towards polymer-based ceramics among newly developed aesthetic materials in dentistry. Therefore, there is a need for developing new composite systems to support the emerging dental material market.

The aim in the present study was to evaluate the mechanical properties of the new material we produced and to determine the content parameters that affect the mechanical properties of this material group [2, 29]. The null hypothesis of this study was accepted that the optimal performance of PICN dental material is affected by ceramic network composition. Increasing the Si/Ca ratio improved the mechanical properties of experimental PICN.

The first all-ceramic dental ceramics with polymer infiltration was In-ceram, developed by Sadoun and commercialized by Vita Zahnfabrik, in 1989. Still, the 20% shrinkage due to sintering associated with the use of all-ceramic systems and the presence of coarse grains resulting in an open fine-pore structure throughout the alumina body are major problems that need to be overcome. Such structure, under the influence of capillary forces, results in almost completely dense structures, when fully wetted with the polymer. And it has found application in the clinic both as crowns in the anterior and posterior regions of the mouth, and as single-unit anterior bridges and two-unit posterior bridge structures [30]. It can be designed by incorporating a second low elastic modulus phase into a ceramic to toughen dental materials and to improve the damage tolerance of restorations against resulting defects [31]. Argyrou et al. [17] showed that nanoceramic resin had larger 3-point flexural strength values as compared to PICN and there was no statistical difference between the mean values of nanoceramic resin (170 MPa, compression) and a leucite-reinforced glass-ceramic (159 MPa, compression), both showed greater values than PICN (124 MPa, compression) and feldspathic ceramic (120 MPa, compression [17]). The mechanical properties of feldspar ceramic can be improved by infiltration of a second phase, the polymer phase, into the porous ceramic structure. A similar study showed that the ratio of porous ceramic to polymer content affects the flexural strength (range 131.1-159.9 MPa), and elastic modulus (16.4–28.1 GPa) of newly produced PICN material [31]. Since a similar correlation was found between the flexural strength and wear behavior of the tested materials and their chemical compositions, the results of the present study show that the mechanical properties depend on the chemical composition of the material.

As a suggestion for future studies, the amount and the type of polymer can be changed to find the optimum polymer content and the optical properties of the PICN material can also be examined.

4. Conclusions

Based on the results of this study, the following conclusions can be made:

- high Ca and Si content in CS and SLS glass, respectively, encourages the use of waste materials in the production of PICNs intended to be used as dental restorative materials;
- the PICNs mechanical properties improved with increasing Si to Ca ratio (wt%);
- of the experimental ceramics used in the study, PICN-3 and PICN-4 were shown to meet the minimum flexural strength of 100 MPa, required by the relevant ISO standard 10477 for use in the clinic.

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