

Article

Two-Body and Three-Body Wear Behavior of a Dental Fluorapatite Glass-Ceramic

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Abstract: As a veneering porcelain coating of dental prosthesis, two-body and three-body wear behavior of dental glass-ceramic with the main crystalline phase of fluorapatite has not been comprehensively studied. In this work, a self-made fluorapatite glass-ceramic was synthesized and the mechanical and tribological performances of the glass-ceramic were tested, comparing with a commercial feldspathic glass-ceramic. The friction and wear experiments were performed between disk-shaped glass-ceramics and natural teeth in two-body (dry, water, saliva) and three-body (slurry) modes, respectively. Results showed that good mechanical properties of fluorapatite glass-ceramic can be achieved by the sintering process. In both two-body and three-body modes, the fluorapatite glass-ceramic had a smaller friction coefficient and wear rate and caused less damage on antagonistic teeth than the feldspathic glass-ceramic. The greater mechanical properties give fluorapatite glass-ceramic a better wear resistance and reduce the adhesive wear.

Keywords: glass-ceramic; veneering porcelain coating; friction and wear; three-body

1. Introduction

There has been an increase in the application of ceramic restorative materials in dentistry [1,2]. Fluorapatite glass-ceramics and feldspathic glass-ceramic are usually used as veneering porcelain coatings on the zirconia or other base ceramics because of their excellent esthetics and biocompatibility [3]. However, the relatively low mechanical performance of the glass-ceramic compared with zirconia and other base ceramics often leads to excessive wear and fractures [4,5]. Wear is the gradual removal of material as consequence of interaction between surfaces moving in contact, and sometimes induces surface microcrack, which may cause fractures of the ceramic [6]. The ideal prosthetic material, which has a high level of wear resistance, can withstand long-term masticatory pressure, while minimizing the wear of the opposing teeth [7]. There are many factors influencing the wear behavior, such as the properties of the material, the abrasive nature of food, the lubrication environment, and the individual chewing behavior [5,8].

Wear resistance of the ceramics has been studied by many researchers [9–11]. Silva et al. [12] found that lithium disilicate glass-ceramic and feldspathic ceramics have similar wear resistance and are wear friendly to the opposing enamel. Zhang et al. [13] found that the fluorapatite glass-ceramic has lower wear resistance and produced more wear loss of steatite antagonist compared with feldspathic glass-ceramic. Santos et al. [14] showed that zirconia presented more suitable tribological behavior than glass-ceramic veneers. However, clinically the glass-ceramic veneers are indispensable because of their esthetics. Therefore, the mechanical and wear properties of the glass-ceramic need to be improved.

Two-body wear means the direct friction between the restoration material and tooth, containing the dry friction and lubrication in saliva or other liquid mediums [10]. Saliva is a complex mixture composed of water (approximately 99%) and a variety of electrolytes and proteins [15]. Some studies have shown that saliva can play an important role in lowering the friction coefficient and wear loss [16,17], while other researchers reported it may increase the friction and wear [18,19]. Mccrea et al. [20] revealed that friction between occluding teeth is influenced by the quantity or quality of saliva and the presence of restorative materials.

Most of the previous laboratory studies investigating wear of dental ceramics and natural teeth were conducted in two-body mode. However, during mastication the wear process is usually a combination of two-body and three-body modes [21]. Three-body wear process refers to the food particles between the restorations and the opposing teeth. Flour, poppy seeds, rice, or polymethyl methacrylate beads have been used in the in vitro three-body wear tests [21,22]. Therefore, the tribological performance of a dental material should be evaluated in both two-body and three-body modes.

Research on the synthesis and tribological behavior of the material will provide guidance for its clinical practice. However, few studies about the tribological behavior between fluorapatite glass-ceramics and natural enamel in both two-body and three-body modes have been found [23]. We have synthesized a series of fluorapatite glass-ceramics with different mechanical properties by adjusting the chemical composition and heat treatment process [24] and evaluated the effect of initial surface topology, load, and speed on the tribological behavior of the fluorapatite glass-ceramic [25]. In the present study, the mechanical properties of the fluorapatite glass-ceramic were evaluated, and comprehensive friction experiments in two-body (dry, water, saliva) and three-body (slurry) modes were conducted, compared with a commercial feldspathic glass-ceramic. The friction coefficient, wear rate, and wear mechanism were discussed.

2. Materials and Methods

2.1. Synthesis of Fluorapatite Glass-Ceramic

The porcelain coatings usually have thickness of 0.5–2 mm. In order to evaluate the performance of the glass-ceramic, the glass-ceramic was not coated on the zirconia or other base ceramics and was fabricated in disk (with dimensions of 15 mm × 15 mm × 2 mm) and bar (with dimensions of 35 mm × 4 mm × 3 mm) shapes. The SiO₂-Al₂O₃-K₂O-CaO-P₂O₅ system fluorapatite glass-ceramic was synthesized using a melt-quench route [24]. Analytical grade SiO₂, Al₂O₃, Na₂CO₃, K₂CO₃, CaCO₃, CaHPO₄, ZrO₂, CaF₂, TiO₂, CeO₂, Li₂CO₃, B₂O₃, and ZnO powders were mixed for 2 h at the planet type ball mill with a speed of 400 r/min. The compositions of the fluorapatite glass-ceramic in weight percentage were SiO₂ 55.6, Al₂O₃ 14.3, Na₂O 8.5, K₂O 4.1, P₂O₅ 4, ZrO₂ 1.5, F 0.7, TiO₂ 1, CeO₂ 0.8, Li₂O 0.2, B₂O₃ 1, ZnO 3, CaO 5.3. Agate balls with a diameter of 15 mm were used in sealed cylindrical polyamide. The weight ratio of ball-to-powder was 2:1. Then, the mixed powders were melted in corundum crucibles at 1600 °C for 3 h in an electric furnace (SQFL-1700C, Jujing, Shanghai, China). Then, molten glass was quenched in distilled water. The obtained frits were milled to glass powers and sieved using a 48 µm mesh analytical sieve.

Disk-shaped glass-ceramic specimens, which were used in friction test, were produced by glass powers that were pressed under 20 MPa pressure using a mold. The pressed powers were heat-treated to nucleate and crystallize, with the heat treatment regime referring to the differential thermal analysis (DTA, STA449F3, Netzsch, Selb, Germany) and X-ray diffractometer (XRD, D500, Siemens, Munchen, Germany) results, which have been depicted previously [24]. The heat treatment regime was: isothermally heated to 1100 °C at a heating rate of 5 °C/min, held for 30 min in the electric furnace, and then cooled naturally.

2.2. Characterization of the Fluorapatite Glass-Ceramic

The microstructures of the fluorapatite glass-ceramic were observed by field emission scanning electron microscope (SEM, JSM-7610F, Jeol, Tokyo, Japan) after being etched with 2.5% HF for 30 s, and crystal phases were analyzed using the X-ray diffractometer (XRD, D500, Siemens, Munchen, Germany). Bar-shaped specimens were made and bending strength was measured by a four-point bending method, as mentioned in reference [26], with loading speed of 0.5 mm/min. The four-point bending flexural test provides values of the flexural stress by loading the bar-shaped specimens with 4 rollers in a material testing machine (Instron 8801; Instron Corp, Canton, MA, USA). The span of the upper inner rollers was 15 mm and that of the lower outer rollers was 30 mm. Bending strength σ_b was represented by the stress when the specimens were fractured, which were calculated by the following equation:

$$\sigma_b = \frac{3Fl}{bh^2}, \quad (1)$$

where, F is the maximum bending force (N), l is the distance between the inner supports and the outer supports (mm), b is the width of the specimen (mm), h is the height of specimen (mm). Strain gauges were adhered on the lower surface of the four-point bending specimens to collect strain value ε during loading, and the elastic modulus was calculated with $\Delta\sigma/\Delta\varepsilon$, as described in reference [27], where $\Delta\sigma$ and $\Delta\varepsilon$ are differences of stress and strain, respectively. A Vickers microhardness tester (HXD-1000TMS/LCD, Optical Instrument Factory, Shanghai, China) was used to measure the hardness of glass-ceramics with load of 4.9 N for 15 s.

2.3. Preparation of Feldspathic Glass-Ceramic and Tooth Specimens

Feldspathic glass-ceramic (Vita VM9) specimens were sintered in accordance with the manufacturer's instructions. The bending strength, elastic modulus, and hardness of the feldspathic glass-ceramic were measured using the same method as previous described. Disk-shaped feldspathic glass-ceramic specimens (15 mm in diameter; 2 mm thick) were produced for the friction experiment. The upper surface of fluorapatite and feldspathic glass-ceramic specimens were polished using aluminum oxide abrasive paper (3M Corp., St. Paul, MN, USA) with sequentially finer grit size (40, 20, and 9 μm). Molars were extracted from 18~32-year-old patients for orthodontic reasons. Each tooth was cut into 2~4 parts using a diamond saw, and the unbroken cusps were chosen in the friction experiment.

2.4. Friction and Wear Tests

The friction tests between the glass-ceramics and natural teeth were conducted in a friction test machine (MMW-1, Shun Mao Inc., Jinan, China). Pin-on-disc loading mode with rotational radius of about 6 mm was employed. The teeth specimens were embedded in the acrylic resin that was fixed on the upper sample holder using a screw. The porcelain specimens were fixed in a steel jig. A total of 1500 revolutions of friction test were conducted on each specimen with the vertical load of 40 N and rotational speed of 150 r/min. Four lubrication conditions were used: dry friction, immersed in water, immersed in nature saliva, and immersed in food slurry. In total, 5 specimens were tested for each set of parameters. Saliva was provided by a healthy male volunteer with no oral disease and was collected prior to experiment between 6 and 7 am. The volunteer had refrained from eating, drinking, and smoking for at least a 12 h period prior to collection. The food slurry consisted of a mixture of 20 g cornmeal grain in 20 mL of distilled water.

Scanning electron microscope was employed to observe the profile of wear scar and elemental composition of the surfaces was analyzed using energy dispersive X-ray spectroscopy (EDX, XMAX50, Oxford Instruments, Abingdon-on-Thames, UK) to evaluate the adhesive wear. Wear volume of the glass-ceramics was measured by a white light interferometer (RTEC Ltd., CA, USA). The wear volume of each tooth was calculated by superimposing the three-dimensional surfaces before and after the

wear test, which were measured using an MTS 3D profiler (MTS Systems Corporation, Eden Prairie, MN, USA).

The friction coefficients and wear results were analyzed with two-way analysis of variance (ANOVA), followed by Tukey's HSD test at a significance level of $\alpha = 0.05$ (SPSS Statistics ver.25, SAS, USA).

3. Results

3.1. Characteristic of the Glass-Ceramic

The microstructures, XRD patterns, and appearance of the fluorapatite glass-ceramic after sintering are displayed in Figure 1, as reported in our previous work [24]. Rod-like fluorapatite crystals were scattered in the glass matrix of fluorapatite glass-ceramic and a minor crystalline phase of anorthite was also detected. The fluorapatite crystals had a mean length of 369 nm, which were determined from more than 100 crystals in SEM micrographs. For feldspathic glass-ceramic, the main crystal is leucite, as reported in reference [13].

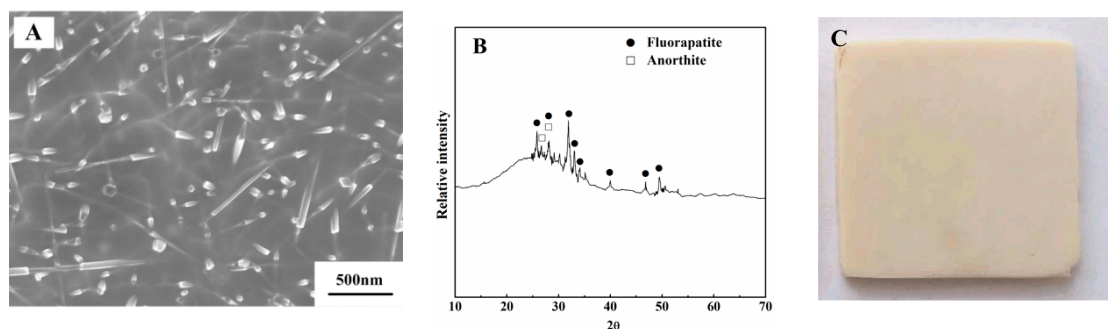


Figure 1. (A) Microstructure, (B) XRD patterns, and (C) the appearance of the fluorapatite glass-ceramic. This figure has been modified from previous publications with permission from the publishers [24].

The bending strength, elasticity modulus, and hardness of the fluorapatite and feldspathic glass-ceramic are listed in Table 1. An increase in all of the three mechanical properties from feldspathic glass-ceramic to fluorapatite glass-ceramic was observed.

Table 1. Mechanical properties of the fluorapatite and feldspathic glass-ceramic (figures in brackets represent standard deviations).

Material	Bending Strength (MPa)	Elasticity Modulus (GPa)	Hardness (HV)
Self-made fluorapatite glass-ceramic	160 (± 15)	87 (± 10)	637 (± 36)
Vita VM9 feldspathic glass-ceramic	98 (± 9)	75 (± 8)	539 (± 34)

3.2. Friction Behavior

Figure 2 presents average friction coefficient of each group at the steady stage. Two-way ANOVA revealed that friction coefficient was significantly affected by friction environment and type of glass-ceramic ($p < 0.05$). Friction coefficients of fluorapatite glass-ceramic under dry and slurry conditions were smaller than that under water and saliva environments. For feldspathic glass-ceramic, the slurry group had the smallest friction coefficient compared to the other three groups. Comparing the two ceramics, friction coefficient of fluorapatite glass-ceramic was significantly lower than that of feldspathic glass-ceramic under the dry condition, while under water, saliva, and slurry environments, the fluorapatite glass-ceramic has no significant difference with feldspathic glass-ceramics.

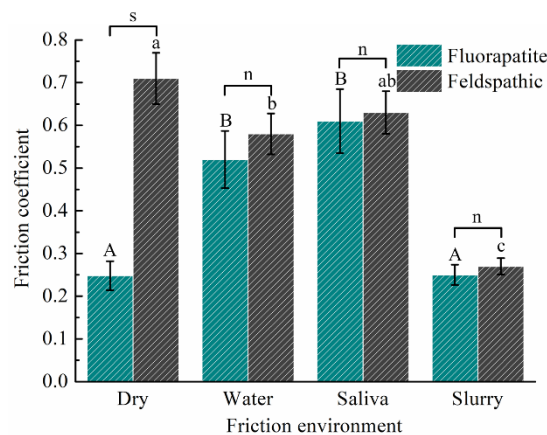


Figure 2. Average coefficients of the two ceramics in dry friction, saliva, water, and slurry conditions. s (Significant) and n (not significant) refers to the Tukey's HSD test results between the two types of specimens in the same environment ($\alpha = 0.05$). Same uppercase letters (fluorapatite) or lowercase (feldspathic) letters represent no significant differences among values of the same material in different environments ($p > 0.05$).

3.3. Wear Behavior

The wear volume of the ceramics and teeth after 1500 revolutions are shown in Figure 3. The wear volume of fluorapatite glass-ceramic in water and saliva was significantly larger than that in dry and slurry conditions. However, in the four feldspathic glass-ceramic groups, dry friction made the largest wear volume of both glass-ceramic and teeth. In all the four groups, feldspathic glass-ceramics and their corresponding teeth showed greater mean wear volume than fluorapatite groups, especially in dry friction.

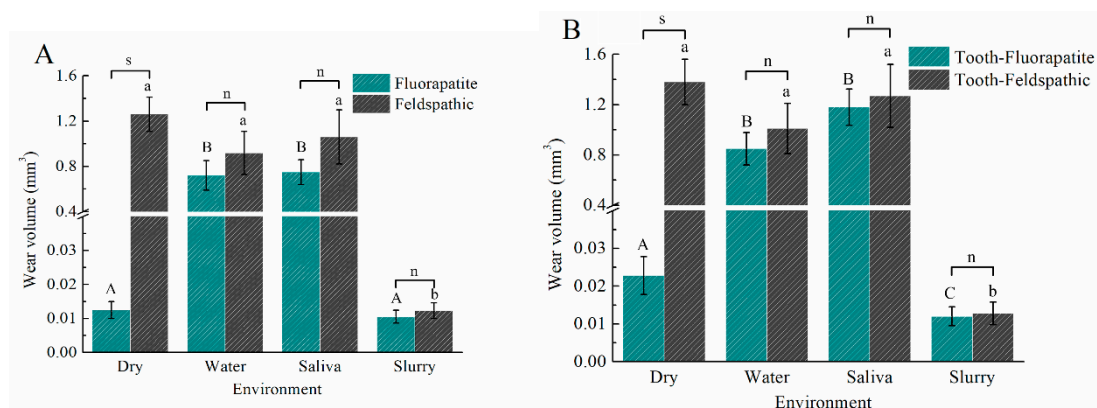


Figure 3. Wear volume of (A) fluorapatite and feldspathic glass-ceramics, (B) teeth tested with fluorapatite and feldspathic glass-ceramics. s (Significant) and n (not significant) refers to the Tukey's HSD test results between the two types of specimens in the same environment ($\alpha = 0.05$). Same uppercase letters (fluorapatite) or lowercase (feldspathic) letters represent no significant differences among values of the same type of specimens in different environments ($p > 0.05$).

Figure 4 shows the typical wear morphology of ceramics and teeth after 1500 revolutions. Different lubrication conditions led to various appearances of wear. Under dry friction, slight grinding and scratch traces were observed on both fluorapatite glass-ceramics and teeth surfaces, along with local cracks. However, feldspathic glass-ceramics and their corresponding teeth showed severe wear with cracks and chippings. In saliva and water environments, both groups showed severely worn surfaces with cracks, defects, and chippings, and extremely rough surfaces with massive chipping were observed for the opposing teeth. However, there were some differences between water and

saliva groups, in which fluorapatite glass-ceramics and teeth in saliva group showed more extensive shedding. In the slurry environment, all the ceramics and teeth showed the slightest wear among the four conditions.

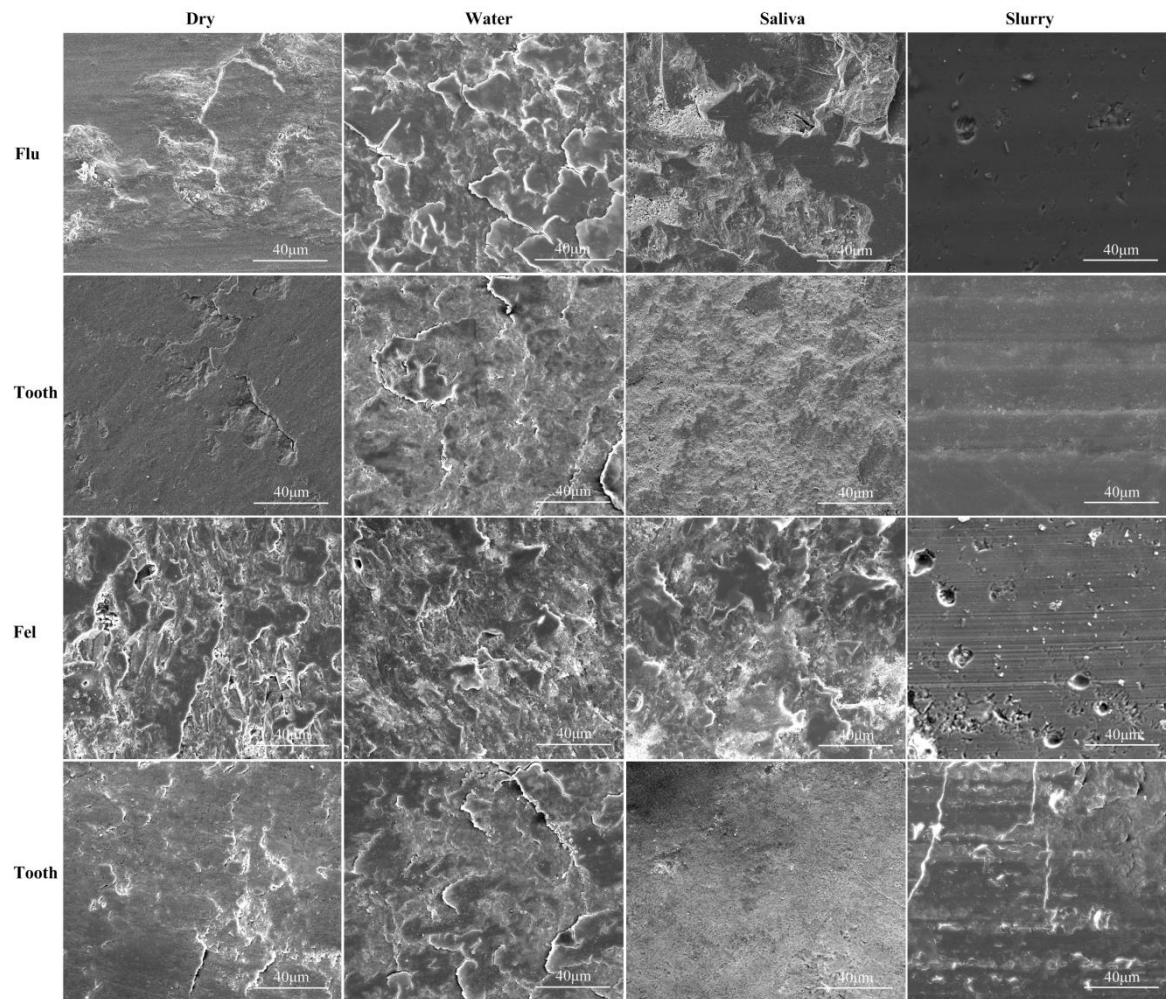


Figure 4. Wear morphology of the fluorapatite glass-ceramics (the first row) with their opposing teeth (the second row) and feldspathic glass-ceramics (the third row) with their opposing teeth in dry, water, saliva, and slurry conditions (the first column to the fourth column, respectively).

EDX analysis was performed on the worn surfaces three times, and the contents of the elements were averaged. The part of the elements that had significant changes are listed in Figure 5. A greater increase in the content of Ca and P elements was detected on the fluorapatite ceramic surfaces in water and saliva conditions than in the dry and slurry conditions. The opposing teeth had small amounts of Si and Al elements in water and saliva conditions, which were the contents of the glass-ceramic. For feldspathic groups, larger amounts of Ca and P elements were detected on the ceramic surfaces in dry, water, and saliva conditions than in slurry condition. There was Si element on the opposing teeth except in the slurry condition.

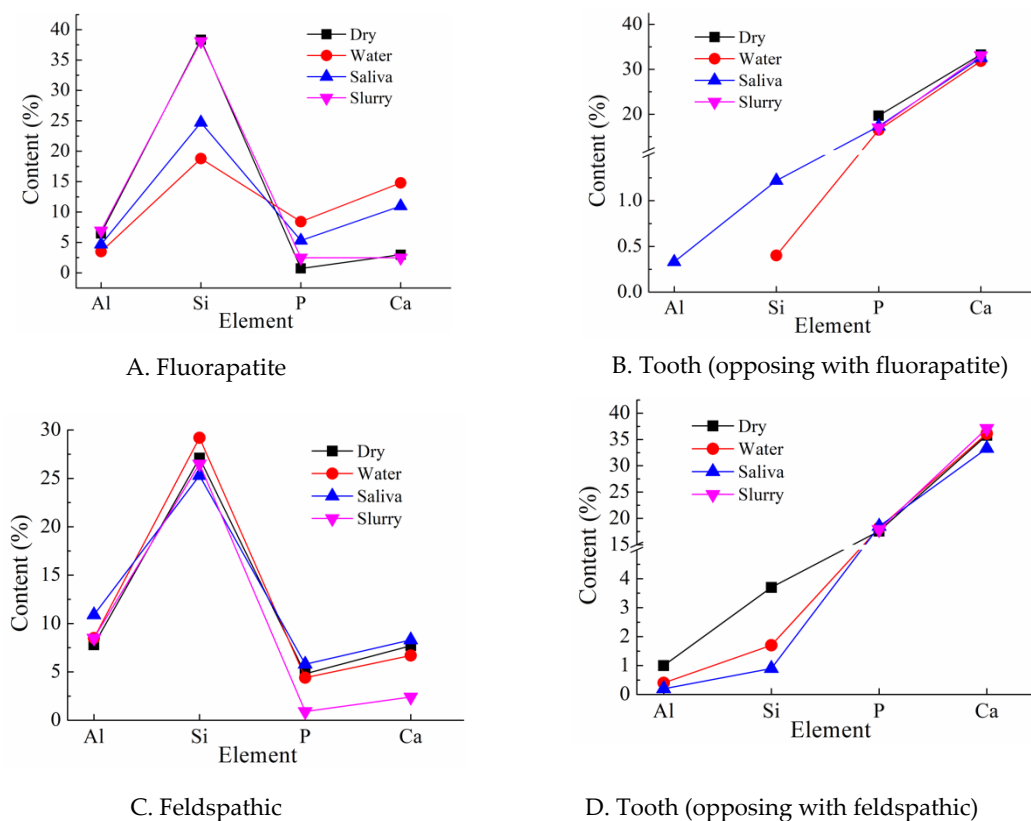


Figure 5. Contents of some elements on worn surfaces of (A) fluorapatite glass-ceramics and (B) their opposing teeth, and (C) feldspathic glass-ceramics and (D) their opposing teeth, obtained by EDX.

4. Discussion

The fluorapatite glass-ceramic made by a sintering process has higher bending strength, elastic modulus, and hardness than commercial ones, such as IPS e.max Ceram [13], which may be attributed to the microstructure. It can be seen from SEM that fluorapatite crystals with different sizes were evenly distributed in the glass matrix (Figure 1). The self-made fluorapatite glass-ceramic had more fluorapatite crystals and a larger average size than the commercial one [13]. The crystals, which had a long needle shape, exhibited a highly interlocking microstructure and play an important role in the reinforcement of the strength and hardness.

In the two-body friction modes, which were dry, water, and saliva situations, fluorapatite glass-ceramic showed better tribological performance than feldspathic glass-ceramic. Especially in dry condition, friction coefficient and wear volume of feldspathic glass-ceramics and antagonistic teeth were significantly larger. The surface of feldspathic glass-ceramic showed more severe wear, especially in the dry condition. The results were different from reference [13], in which a commercial fluorapatite glass-ceramic showed larger friction coefficient and wear rate than feldspathic glass-ceramic. This can be attributed to the microstructure and mechanical properties of the materials. According to the mechanical tests, the self-made fluorapatite glass-ceramic had larger strength, elastic modulus, and hardness than the commercial one and feldspathic glass-ceramic.

Friction coefficient and wear rate of the fluorapatite glass-ceramics were larger in saliva and water situations than in the dry friction condition, which was similar with some previous studies on glass-ceramic or teeth [17,19]. It can be seen in the SEM and EDX results that the wear mechanisms in the dry condition were a combination of abrasive and fatigue wear. While in the wet condition, severely worn surfaces with massive chippings were presented on the both ceramic and tooth surface. A greater increase in the content of Ca and P elements (rich in teeth, other than ceramics) could be detected on the fluorapatite glass-ceramic surfaces in water and saliva conditions than dry and slurry conditions, which

meant that fragments of teeth were adhered on the ceramic surfaces in water and saliva conditions. In addition, Si and Al elements, which only belong to the glass-ceramic, were detected on the teeth surfaces, indicating the adhesion of glass-ceramic on the teeth surfaces. This demonstrated that the main wear mechanism in water and saliva conditions was adhesive wear. The water and saliva softened the surfaces of glass-ceramic and enamel and enhanced the adhesion of the two surfaces. Besides, it showed larger friction coefficient and wear rate in the saliva situation compared with water situation, because saliva, which had a normal pH of 6 to 7, also induced corrosion wear due to its acid erosion effect [9,15,28]. In the acidic environment, positively charged hydrogen ions in the solution exchange with cations in the surface of glass-ceramic such as Na^+ , K^+ , and Ca^{2+} [9]. Additionally, the glass network dissolves in a wet environment through the breakdown of Si–O network. Consequently, the peeling and adhesion increased the friction coefficient and wear rate.

For feldspathic glass-ceramic, the adhesive wear occurred in dry, water, and saliva environments, which can be proven from the surface appearance and element analysis. Different from fluorapatite glass-ceramic, the feldspathic glass-ceramic showed the largest friction coefficient and wear rate in dry condition, which was identical to results of previous research [17]. Teeth surfaces in dry conditions had the most Al and Si elements, which belong to the ceramic, indicating the most severe adhesive wear occurred in the dry condition. The feldspathic glass-ceramic had lower hardness, elastic modulus, and strength than fluorapatite glass-ceramic, making it more likely to adhere to the teeth surfaces during friction.

In the three-body mode, the glass-ceramics and teeth didn't contact directly. However, feldspathic glass-ceramics also had larger wear than fluorapatite glass-ceramics, which may be because the greater hardness of fluorapatite glass-ceramic made it more abrasion resistant. SEM analysis also showed that furrows appeared on the feldspathic glass-ceramics and teeth surfaces, but hardly showed on the fluorapatite glass-ceramic surfaces. It can be seen from the results that two-body wear was greater than three-body wear, which was in accord with early research [29,30]. In the three-body condition, the materials were in fluid lubrication, or in contact with the softer food, which has lower friction coefficient and wear rate. It also can be explained that normal chewing (three-body wear) causes slight wear, but sleep bruxism (two-body wear) induces severe wear.

In this work natural tooth, natural saliva, and slurry were employed in the experiments to simulate the real friction state of the dental material in service. However, the loading regime could not accurately simulate the real chewing process. In the future, the tribological experiments using chewing simulator or in vivo tribological experiments should be performed.

5. Conclusions

In the present study, the mechanical properties of the self-made fluorapatite glass-ceramic were measured, and its two-body and three-body wear behavior were evaluated. Within the limitations of this study, the following conclusions are drawn:

- (1) Good mechanical properties of fluorapatite glass-ceramic can be achieved by the sintering process and material components. The fluorapatite glass-ceramic has greater hardness, elastic modulus, and strength than the feldspathic glass-ceramic that was tested.
- (2) In both the two-body and three-body modes, the fluorapatite glass-ceramic had better tribological performances and caused less damage than the feldspathic glass-ceramic. The fluorapatite glass-ceramic and antagonistic tooth had a larger friction coefficient and wear rate in the saliva and water conditions than in the dry and slurry conditions because water and saliva facilitate the adhesion of the two contact surfaces and change the main wear mechanism from abrasive wear and fatigue wear to adhesive wear. Meanwhile the feldspathic glass-ceramic showed adhesive wear in dry, water, and saliva environments and had the largest friction coefficient and wear rate in dry condition.

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Conflicts of Interest: The authors deny any actual or potential conflict of interest.

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