



Research Paper / Makale

Investigation of Mechanical and Wear Properties of Five (5) Different CAD/CAM Restorative Materials

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Abstract: In this study it was investigated mechanical and wear properties of CAD/CAM restorative materials including feldspathic ceramic, leucite-reinforced glass ceramic, lithium disilicate glass ceramic, and resin-matrix ceramics. The hardness values of materials were measured with micro hardness tester. Wear properties were evaluated with pin on disk tribometer. Wear volume were measured with optical profilometer. Worn surfaces were examined with Scanning Electron Microscopy. The lowest friction coefficient was obtained from Lava Ultimate. It was observed that the highest wear rate was obtained from the LAVA sample was showing the lowest micro-hardness. The lowest wear rate was obtained from e-max sample which was showing the highest micro-hardness. The glass ceramic materials showed better wear resistance than resin-matrix ceramic materials.

Keywords: CAD/CAM restorative materials, Wear, Micro Hardness

Beş (5) Farklı CAD / CAM Restoratif Malzemelerin Mekanik ve Aşınma Özelliklerinin İncelenmesi

Öz: Bu çalışmada, CAD / CAM restoratif malzemelerinden olan feldspatik seramik, lösit takviyeli cam seramik, lityum disilikat cam seramiği ve reçine-matriks seramiklerinin mekanik ve aşınma özellikleri araştırılmıştır. Malzemelerin sertlik değerleri mikro sertlik test cihazı ile ölçülmüştür. Aşınma özellikleri disk üzeri pin tribometresi ile incelenmiştir. Optik profilometre ile aşınma hacmi ölçülmüştür. Aşınma izleri Taramalı Elektron Mikroskobu ile incelenmiştir. En düşük sürtünme katsayısı Lava Ultimate'den elde edilmiştir. En yüksek aşınma oranının, en düşük mikro sertliği gösteren LAVA örneğinden elde edildiği gözlenmiştir. En düşük aşınma oranı, en yüksek mikro sertliği gösteren e-max numunesinden elde edilmiştir. Cam seramik malzemeler reçine matriks seramik malzemelere göre daha iyi aşınma direncine sahip olduğu tespit edilmiştir.

Anahtar kelimeler: CAD/CAM restoratif malzemeler, Aşınma, Mikro sertlik

1. Introduction

The growing demand for biocompatible and esthetic restorations has led to the generation of numerous ceramic systems, including CAD/CAM (computer-aided design/computer-aided manufacturing), which has emerged as a novel system in the industry and is now used as an alternative to the conventional ceramic systems used in laboratories. Although first met with some hesitancy, CAD/CAM has become more widespread in recent years owing to improved technology, which has allowed for faster and more exact restorations. Now, during a single visit, dental restorations are able to be milled (CAM-computer-aided manufacturing) in a highly precise manner via a digital camera-generated optical impression and 3D restoration design created with software (CAD-computer aided design) [1].

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Tooth-colored restorations can be modeled and milled from ceramic and composite materials using dental CAD/CAM systems. The key advantage of these indirect restorations is that they prevent polymerization shrinkage and decrease micro leakage, both of which serve to improve the longevity of the restoration. Moreover, with CAD/CAM system, chairside restorations are able to be performed, eliminating the long laboratory procedures involved in conventional ceramic systems [2,3].

Glass-based ceramics are widely used in dental applications due to their unique mechanical, chemical and optical properties, such as superior wear resistance, hardness and strong resistance to oxidation, and remarkable biocompatibility [4]. One disadvantage, however, is their brittle nature, which limits their usage. Many attempts have been made from manufacturers to overcome this limitation by reinforcing ceramics with different oxides.

Gracis et al. classified ceramics according to their chemical composition: (1) glass-matrix ceramics; (2) polycrystalline ceramics; and (3) resin-matrix ceramics [5]. Leucite reinforced glass ceramics and lithium disilicate ceramics have been introduced to the market as glass-matrix ceramics. The main difference distinguishing these glass-matrix ceramics from conventional feldspathic ceramics is the addition of leucite or lithium disilicate phases to enhance the mechanical properties of the material. These phases are said to prevent crack propagation in the ceramic structure [6]. Another new trend to have emerged is the production of ceramics with composite content (resin-matrix ceramics) to achieve more wear resistant restorations. The aim here is to take advantage of the stress distributor feature of the composite in the material structure. In other words, less stress in the material provides more durable restorations under chewing forces [7,8].

The key factor for ensuring functional oral tissues is that the dental restorative materials have excellent mechanical properties. Moreover, in addition to the mechanical properties, biocompatibility and avoidance of damage to surrounding tissues is also crucial [9]. The wear characteristics of materials is another important factor in terms of sustaining chewing function [10]. Various clinical factors, such as saliva composition and its pH, temperature, gender, age, nutritional and parafunctional habits, occlusion, neuromuscular forces, enamel thickness and hardness, material and contour of antagonistic teeth, and position of restoration can affect the wear behavior of restorative materials [11,12]. It is vital that the artificial restorative material replacing the enamel shows similar wear rates to its predecessor. Although the use of composite materials results in substance loss during mastication, they nonetheless offer an advantage over ceramics insofar as they result to less wear from the antagonist enamel.

To achieve the desired clinical performance of dental materials and ensure their longer life, it is important to know their microstructural characteristics and wear performance. In dental research, the fracture and wear behavior of bioceramics has been the major focus because of the importance of the structural longevity and predictability of bioceramic prostheses and due to the observation that most dental ceramics become abrasive toward opposing dentition [13,14].

In this study, the wear performance of three glass ceramics and two resin-matrix ceramic dental CAD/CAM materials has been investigated. The data available on the wear of CAD/CAM ceramics are very limited. Therefore, the purpose of this study is to evaluate and compare the wear performance of different CAD/CAM materials under reciprocating loads. The statement, "Different compositions of CAD/CAM materials do not affect the wear values", served as the null hypothesis for the study.

2. Materials and Methods

The chemical compositions of five CAD/CAM restorative materials are given in Table 1. The five CAD/CAM restoratives tested were feldspathic ceramic (Sirona Cerec Bloc, VITA Zahnfabrik),

leucite-reinforced glass ceramic (IPS Empress CAD, Ivoclar Vivadent), lithium disilicate glass ceramic (IPS e.max CAD, Ivoclar Vivadent), and resin-matrix ceramics, which included Lava Ultimate (3M ESPE) and Enamic (VITA Zahnfabrik).

Table 1. Chemical composition of different CAD/CAM materials

Name	Composition	(% wt)
Cerec Blocs,	SiO ₂	56-64
	Al ₂ O ₃	20-23
	Na ₂ O	6-9
	K ₂ O	6-8
	CaO	0.3-0.6
	TiO ₂	0.0-0.1
IPS Empress CAD	SiO ₂	60-65
	Al ₂ O ₃	16-20
	K ₂ O	10-14
	Na ₂ O	3.5-6.5
	Other Oxides	0.5-7.0
	Pigments	0.2-1.0
IPS e.max CAD	SiO ₂	57-80
	Li ₂ O	11-19
	K ₂ O	0-13
	P ₂ O ₅	0-11
	ZrO ₂	0-8
	ZnO	0-8
	Al ₂ O ₃	0-5
	MgO	0-5
Lava Ultimate	Ceramic	(%80)
	Resin	(%20)
Enamic	Ceramic	(%86):
	SiO ₂	58-63
	Al ₂ O ₃	20-23
	Na ₂ O	6-11
	K ₂ O	4-6
	B ₂ O ₃	0.5-2
	CaO	< 1
	TiO ₂	< 1
	Polymer	(%14):PMMA

For the wear tests, one sample from each ceramic material was cut to dimensions of 8×8×2 mm using a low-speed diamond saw (Isomet 1000, Buehler). Then, IPS e.max CAD samples underwent crystallization firing (heat treatment) according to the manufacturer's recommendations (final sintering temperature of 850°C for 10 min) in the Ceramic Oven Furnace (Programat P100; Ivoclar Vivadent). All of the surfaces of the samples were polished using SiC emery paper with 1200 and 2000 mesh grit on a slow-speed electric handpiece (300rpm), under hand pressure and water cooling, respectively.

Wear tests with a sliding distance of 20 m were performed on a Turkeyus reciprocating tester under a normal load of 5.9N for 5000 cycles. The wear tests were carried out at room temperature (20 ± 2°C) using an Al₂O₃ ball with a diameter of 6 mm in an artificial saliva solution (Figure. 1). Saliva solution prepared in lab environment. The chemical compositions of the artificial saliva solution are shown in Table 2.

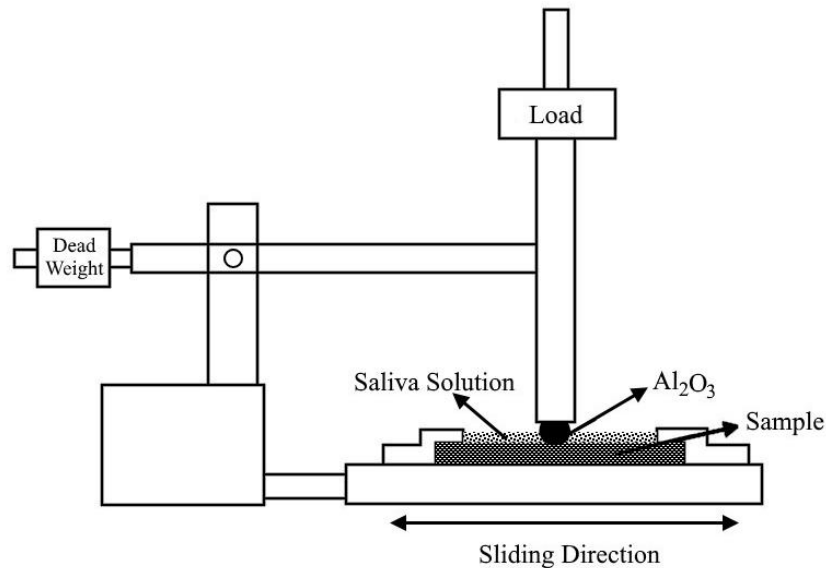


Figure 1. The test configuration

The worn surfaces of the tested restorative materials were observed with a scanning electron microscope (SEM) (FEI Quanta FEG 250). The hardness values of samples were measured by using a micro-hardness tester (FUTURE TECH FM800e) under 100 g load at 10 seconds dwell time using Vickers method.

Table 2. Chemical composition of artificial saliva solution [15].

Compound	Concentration (mg/l)
NaH ₂ PO ₃ H ₂ O	780
NaCl	500
KCl	500
CaCl ₂ H ₂ O	795
NaS ₉ H ₂ O	5
(NH ₄) ₂ SO ₄	300
Citric acid	5
NaHCO ₃	100
Urea	1000

In order to calculate the wear rate and the wear profiles, first the values were recorded using BRUKER CONTOUR GT 3D optical profilometer and then, the wear rates were computed using the equation $V=(W/ws)$ [mm³/N.m]; where V is wear rate, W is wear volume, ‘w’ is the normal load, and ‘s’ is the sliding distance.

3. Results

Scanning electron microscopy was used to analyze the surface morphology of the materials, shown in Figure 2, where the interconnected ceramic network structure is clearly evident for the Enamic sample. The same structure was also observed in another study [16].

The mean hardness values of the samples were measured with a micro-hardness tester, the results of which are given in Table 3.

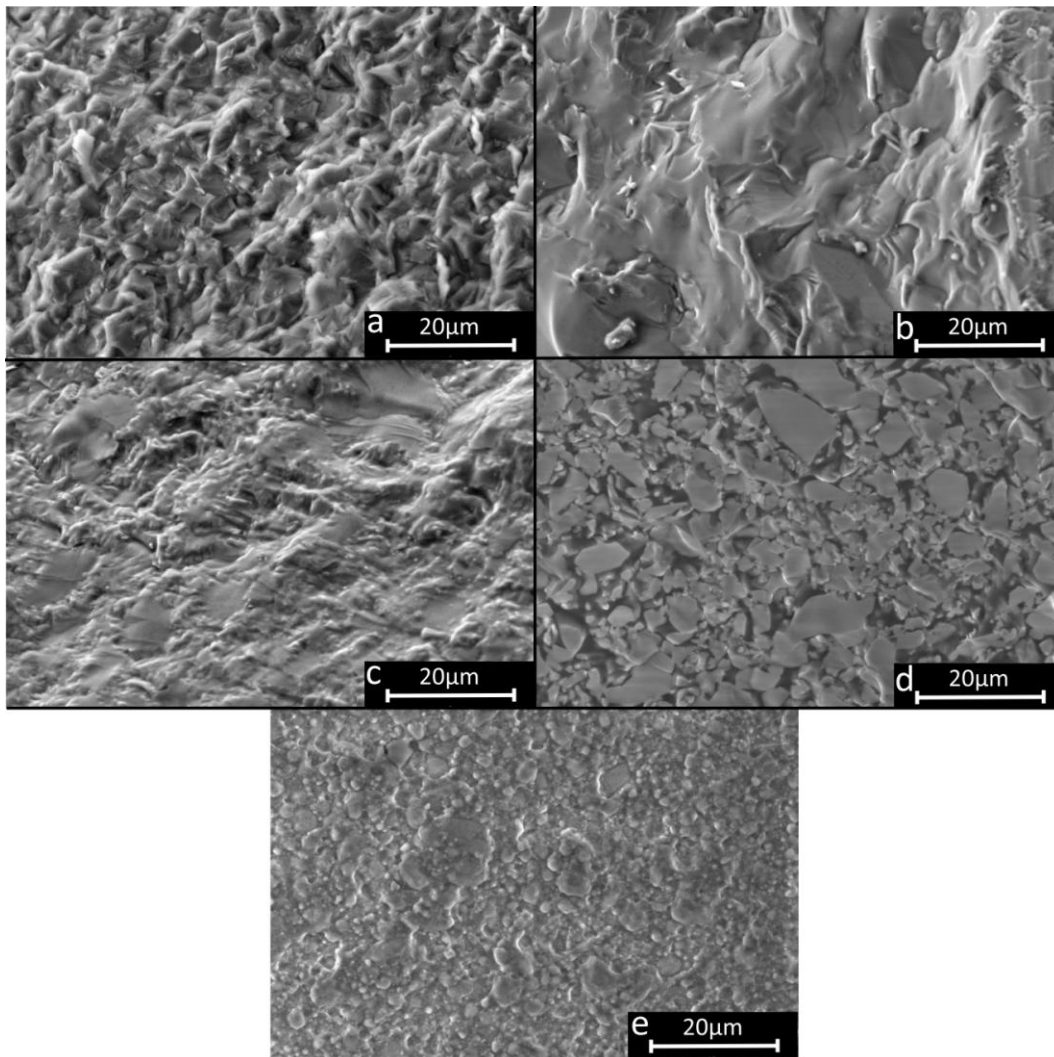


Figure 2. Surface SEM image of samples a) E-max, b) Cerec, c) Empress, d) Enamic and e) Lava Ultimate

A graph of the friction coefficients during sliding against Al_2O_3 ball for glass ceramic and resin-matrix ceramics is presented in Figure 3. The friction coefficients of the samples show the steady-state regime after a 1500 second running in period. The fluctuation of the friction coefficients of the samples is related to the third body effect of worn particles.

Table 3. Mean hardness value of CAD/CAM materials.

Materials	Hardness (HV)
E-Max	660
Empress	570
Cerec	560
Enamic	255
Lava Ultimate	139

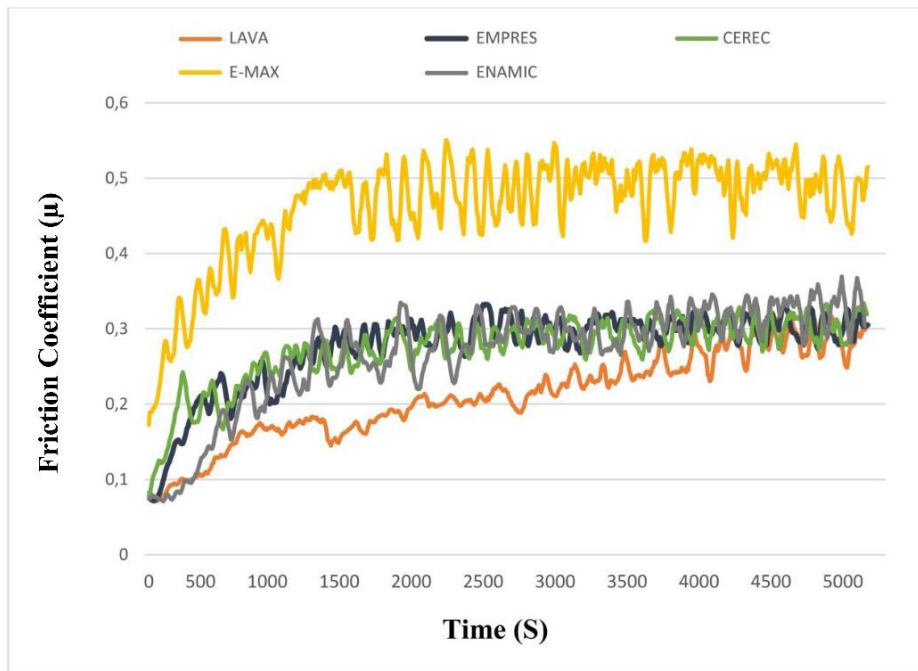


Figure 3. The friction coefficient of materials tested with Al₂O₃ antagonists (counter material).

As seen in Figure 3, five samples showed different frictional behavior under the wear testing. Furthermore, it was found that the friction coefficients increased correspondingly with the increase of hardness. The lowest friction coefficient was obtained from Lava Ultimate, which had the lowest surface micro-hardness. The micro hardness versus wear rate graph is depicted in Figure 4.

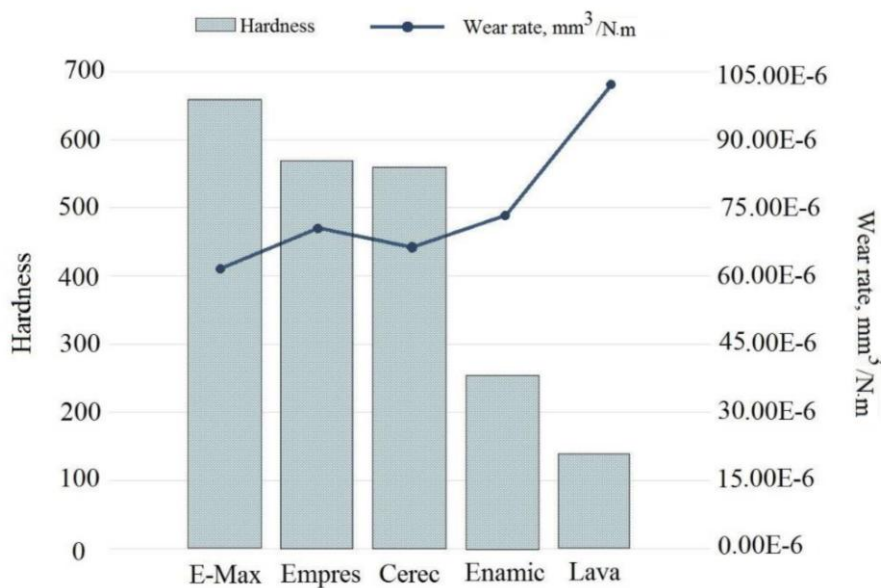


Figure 4. The wear and micro-hardness graphic of samples

When the wear rates were investigated, it was observed that the maximum wear rate was obtained from the LAVA sample, which had the lowest value of micro-hardness. On the other hand, the lowest wear rate was obtained from the e.max sample, which had the highest value of micro-hardness. As is known, hardness is the measure of the resistance to plastic deformation. Therefore, materials with high hardness value exhibit better resistance to plastic deformation caused by wear on the surface. The wear rate was calculated with 3D profilometer after scanning. The profilometer images and SEM images obtained after the wear tests are shown in Figure 5 and Figure 6.

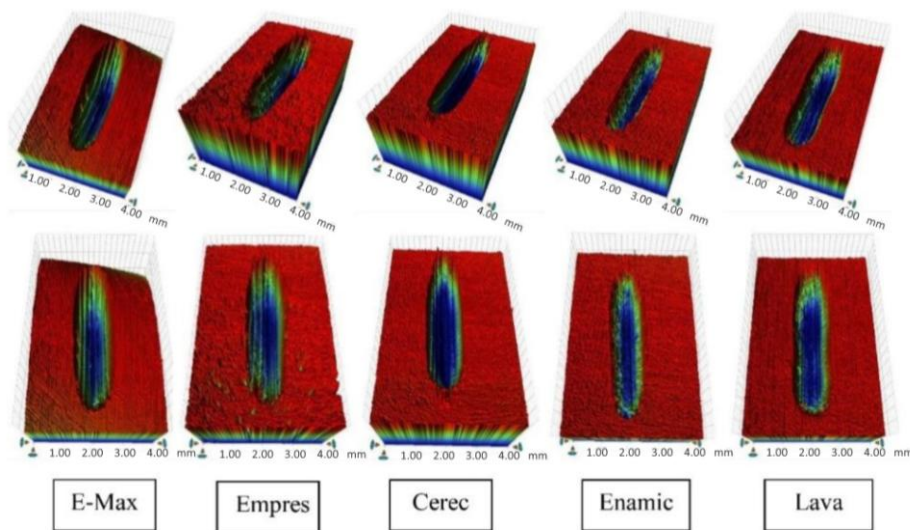


Figure 5. Optical profilometer images of samples.

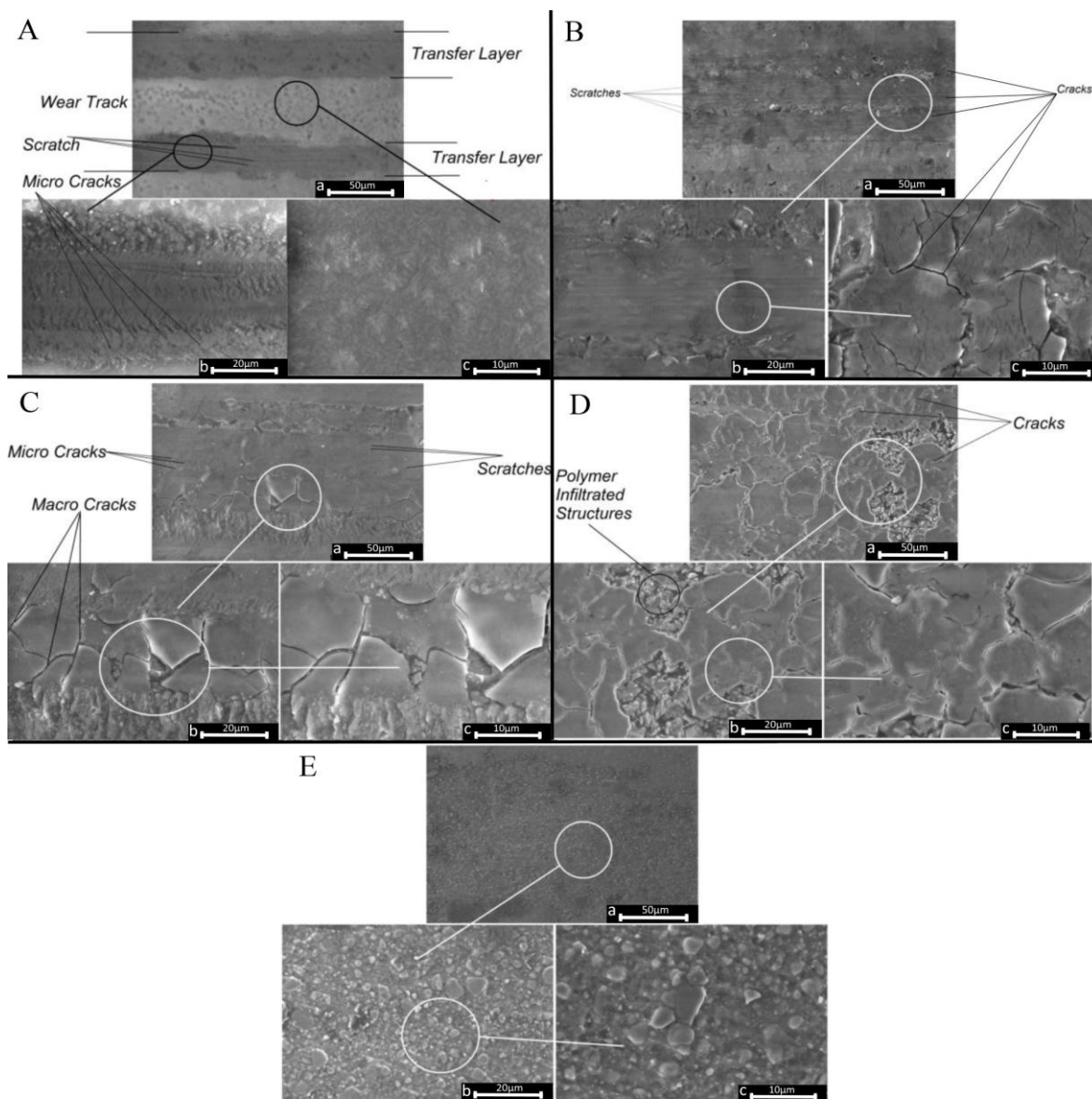


Figure 6. SEM images of worn surfaces of samples. A) E-max, B) Cerec, C) Empres, D) Enamic and E) Lava Ultimate

When the SEM images were examined, it was seen that abrasive wear was the dominant wear mechanism of Lava Ultimate, Enamic and Empress. The e.max and Cerec were only slightly worn, with the surfaces showing a mix of scratches and smoothness, as compared to other samples.

4. Discussion

In this study, an Aluminum Oxide ball was used as the counter surface. The highest friction coefficient was attained from e.max, while the lowest friction coefficient was attained from LAVA Ultimate material. The highest hardness value, which was 660 HV, was measured from the e.max sample, whereas the lowest hardness value, which was 139 HV, was measured from the Lava Ultimate sample. Evaluations of the micro-hardness values of the samples indicated that the resin-matrix ceramic materials were softer than the glass ceramic materials. The same result was also reported in a separate study [17]. Considering these findings, it can be inferred that the e.max sample's higher friction value is related to its highest hardness value, and likewise, that the Lava Ultimate sample's lowest friction value is related to its lowest hardness value. Empress and Cerec had relatively the same friction coefficient, and their hardness values were also close to each other. The wear rate results of the samples are given in Figure 4, where it can be seen that e.max showed the highest wear resistance and LAVA Ultimate the lowest wear resistance to Al_2O_3 counter surface. IPS Empress and Cerec Blocs showed almost the same wear rate. Lava Ultimate and Enamic showed relatively higher degrees of wear. In general, the e.max, Empress and Cerec ceramic materials exhibited higher wear resistance than resin-matrix ceramic, Enamic and Lava Ultimate. Similar results were also reported in other studies [18,19]. These wear resistant findings could be related to the different hardness values seen in the samples, as demonstrated by the fact that the e.max, which has high hardness, shows high wear resistance, while Lava Ultimate, which has lower hardness, shows lower wear resistance [19]. Another reason for the lower wear resistance of Lava Ultimate and Enamic may be related to the filler content proportions, the quality of the interfacial bond between the fillers and matrix, and the extent of the curing of the resin matrix, as indicated in another study [18]. In contrast to these studies, Stawarczyk et al. found that the wear resistance of Enamic was higher than that of e.max and Empress [20].

SEM images of the wear tracks, after the wear test of both glass ceramics and resin-matrix ceramics against the Al_2O_3 counter surface, are shown in Figure 6. SEM observation of the worn surfaces of the sample revealed different wear mechanisms. For e.max, it can be seen from Figure 6A that the wear track consists of two main regions, the first being the transfer layer, which smeared on the sides of tracks, and the second being the place where materials were ruptured. In this image, many micro cracks are apparent on the surface of the transfer layer. The formation of a transfer layer decreased the wear rate of the materials. At the beginning of the experiment, there was contact between e.max and the counter body. However, after a short time, due to the transfer layer mechanism, there is contact between e-max and e-max. Because the e-max material is transferred to the counter material and adheres to the surface. This mechanism serves to explain the higher friction coefficient but lower wear rate. In the e.max wear test, the transfer layer that formed on the surface of the counter material (Al_2O_3) is shown in Figure 7.

For Cerec, with repeated loadings the materials compressed, resulting in rupture, as shown in Figure 6B. The same occurred for Empress, whose wear rates were also similar to those of Cerec (Figure 4). Cracking and spalling are the main wear mechanisms for Empress and Enamic materials. The Enamic restorative materials include 86-75 wt.% inorganic phase (ceramic) and 14 wt.% organic phase (polymer). Sliding scratches can be seen on the e.max, Cerec, and Empress. These scratches can be attributed to abrasive wear caused by the transferred material to the alumina (Al_2O_3) ball surface. The Lava samples had a granular morphology, as shown in Figure 6E.

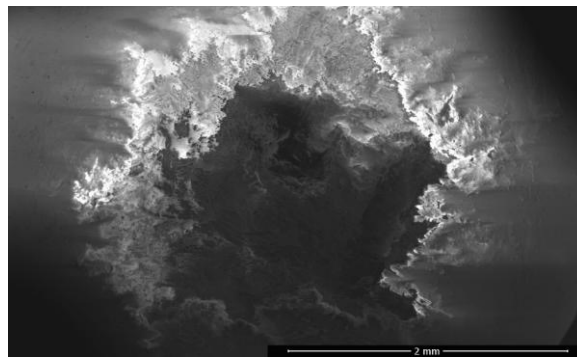


Figure 7. SEM image of antagonist Al₂O₃ ball demonstrating transfer layer on the surface.

Lava Ultimate's morphological structure (Figure 2), in particular, remained the same after worn experiments (Figure 6E). This finding suggests that there was insignificant tribological reaction and that the material was worn due to its brittle structure, resulting in it having the highest wear rate. Moreover, there was no significant transfer layer observed for this material, and it had the lowest friction coefficient, which is related to its polymeric structure. However, due to the lack of transfer layer, it did not help to reduce the wear rate. For Enamic materials, as shown in Figure 6D, the cracks occurred mainly along the polymer/ceramic interface. From Figure 6, it can be seen generally all samples had fatigue wear. The literature reports that due to the repetitive loading, the fatigue wear mechanism is a very common feature of ceramic materials [21]. In this study, during the reciprocating sliding wear experiment, tension and compression stress zones formed in the tested samples, resulting in the formation of cracks in the sub-surfaces. With time, all the cracks reached to the surface and led to fragments being lost from the materials. This fragmented wear debris also caused 3-body abrasive wear on the tested materials. The SEM images, shown in Figure 6, confirmed these results. The similar failure mode including subsurface cracks, fragmentation and consequently causing abrasive effect was also observed in other studies [4,22].

5. Conclusions

In this study, the friction and wear properties of 5 different CAD/CAM restoratives were investigated. The tested materials were chosen because of their popularity among clinicians; however, little information can be found about their wear properties. The results from this study can be summarized as follows:

1. The wear resistances of the 3 glass ceramic materials were higher than those of the 2 resin-matrix ceramic materials.
2. The highest hardness value was measured for e.max, followed by Empress, Cerec, and Enamic and Lava Ultimate, which had the lowest.
3. The hardness of the restorative material plays a relevant role in wear resistance.

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