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Degradation of the Mechanical Properties of CAD/CAM Ceramics upon Immersion in Various Beverages

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ABSTRACT

Background and Objective: The durability of restorative materials is crucial for their success, as their physical degradation is affected by the oral environment, such as water sorption and crushing stress. The adaptation of CAD/CAM ceramics to enamel and composite is also key to their success. The aim of this study is to evaluate the effect of different solutions on the mechanical properties (flexural strength and diagonal tensile strength [DTS]) and water sorption of CAD/CAM ceramics when immersed in artificial saliva, ethanol/water, mouthwash and cola.

Methods: In this in vitro study, four different solutions of Artificial Saliva (AS), Ethanol/water (EW), Mouth rinses (MW), and Cola (PI) and a total of 170 standardized specimens were prepared according to ISO 6872:2009 (85 for flexural strength and 85 for DTS), with an additional 20 specimens for water sorption. The specimens were then immersed in the four solutions at 37ºC. Then, measurements of flexural strength and DTS were done on days 1, 15, 30, 45, and 60, and water sorption and solubility were evaluated for 180 days.

Findings: Immersion in saliva and mouthwash solutions after 60 days had little effect on the flexural strength of lithium silicate (L-SiC) and Zirconium (Y-TZ). Flexural strength values decreased significantly in ethanol/water (EW), and cola (PI) solutions and were 35.12% and 33.42% for L-SiC, respectively. For Y-TZP, the decrease was small and were 13.92% and 13.09% for EW and PI, respectively. As for DTS, the deterioration of immersion in solution was clear, ranging from 4% to 12% for the L-SiC and from 3% to 10% for the Y-TZP. However, there was no significant difference in water sorption and solubility, which ranged from 18.7 to 13.98 µg/mm3 and 0.63 to 4.47 µg/mm3. The flexural strength and DTS of CAD/CAM blocks showed slight variation after immersion in artificial saliva and mouthwash but decreased significantly when immersed in Ethanol/water and cola. Additionally, the mass of specimens immersed in cola decreased after 40 days.

Conclusion: The results showed that the highest degradation of CAD/CAM ceramics was associated with cola.

Keywords: *CAD/CAM Ceramic, Water Sorption, Flexural Strength, DTS, Physical Properties*.

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Introduction

Over the past ten years, CAD/CAM technologies (COMPUTER-AIDED DESIGN/COMPUTER-AIDED MANUFACTURING) and materials have become increasingly important for indirect prosthetic restorations (1). Compared to direct restorative composite resin, the material characteristics have been significantly enhanced. Ceramics is by far the most common material used to fabricate CAD/CAM blocks (2). The durability of restorative materials is crucial for their success, as their physical degradation is affected by the oral environment, such as water sorption and crushing stress (3). The adaptation of CAD/CAM ceramics to enamel and composite is also key to their success. A wide range of CAD/CAM blocks, particularly aesthetic restorative materials, have been developed (4). These materials are more tooth-friendly, less brittle, and easier to machine compared to traditional materials (5). CAD/CAM interim materials are prefabricated from industrially polymerized blocks to prevent polymerization heat and shrinkage (6, 7). CAD/CAM materials can also be used for implant restorations. Various methods have been utilized to investigate the mechanical properties of materials utilized in fabricating these CAD/CAM restorations, and most studies have processed blocks in cylinders or cuboids to understand the material's mechanical properties (8, 9). Standardizing teeth into the same shape for a specimen is required to gather systematic information, requiring complex processes. Computer-assisted design/computer-assisted manufacture (CAD/CAM) technologies have been introduced to fabricate interim restorations. Polymers with various cross-linking densities are employed to fabricate CAD/CAM provisional restorations (10, 11).

Ceramics have good mechanical properties with flexural resistance (>80 MPa) and high elastic modulus (2800 MPa), making them a long-term provisional material (12). CAD-CAM ceramics are widely used due to their chemical stability, excellent biocompatibility, and good optical and mechanical properties. Nonetheless, repairs are often problematic if required once placed in the mouth. Conversely, while composites are easier to operate and repair, their biocompatibility, mechanical properties, and wear are less than ceramics (13). The most common cause of clinical failure may be the failure of restorations caused by bulk breakdown (14). Ceramics are brittle materials by nature, and therefore they are usually subject to catastrophic breakage caused by inadvertent bending forces. For intra-oral application, restorations should have a level of strength sufficient to allow them to bear the crush numerous times.

The physical deterioration observed in dental materials can occur through processes such as sorption and dissolution, leading to either loss or absorption of material. Water sorption refers to the capacity of dental materials to take in liquids, resulting in changes to their volume and weight. This phenomenon involves both physical and chemical processes. On the other hand, water solubility indicates the quantity of a chemical substance that can dissolve in water at a given temperature, typically expressed as the number of grams of solute present in one litter of a saturated solution (15). The continuity and clinical durability of dental composites may be affected by alterations in their mechanical properties when exposed to different immersion media (16).

Studies have mostly been focused on determining the water sorption characteristics of previous research has explored the colour transformation of CAD/CAM when submerged in various liquids like coffee, tea, Coca-Cola, wine, and different staining solutions (17-22). Additionally, studies have examined the impact of acid on the physical properties and the effect of salivary pH on the flexural strength.

So far, there have been no studies published that compare and evaluate the mechanical properties and kinetic sorption of CAD/CAM ceramics after being immersed in different media. Therefore, the aim of this study was to investigate the impact of immersion in various solution media (pH:2.8-7.4) on the evaluation of Sorption Kinetics, as well as the mechanical properties (Flexural strength and DTS) of CAD-CAM ceramics for 1, 15, 30, 45, 60 days, water sorption and solubility were evaluated for 180 days.

Methods

Preparation of solutions and specimens: This in vitro study selected two commercially available CAD/CAM ceramics: lithium disilicate (L-SiC) and zirconium (Y-TZP) (Table 1). Four different solutions were used as immersion media: artificial saliva (AS), ethanol/water (EW), mouthwash (MW), and cola (PI). The pH of each solution was measured using a pH meter (ISOLAB Laborgeräte GmbH, Germany).

The specimens were immersed in the four solutions at 37ºC. Flexural strength and diametral tensile strength (DTS) were measured at 1, 15, 30, 45, and 60 days, whereas water sorption and solubility were assessed over a 180-day period.

Table 2 shows the composition and pH value of each solution. Eighty-five rectangular bending specimens (14×4×3 mm, ISO 6872:2009) were prepared, along with the same number of disc specimens for DTS (Ø 6×3 mm) and twenty-disc specimens (Ø 14×1 mm) for sorption kinetics, for each tested CAD/CAM ceramic material. The specimens were cut from both commercial CAD/CAM ceramics using a cutting machine (Cori TEC 350i, imes-icore, Germany). They were then crystallized in a ceramic furnace (Ht_S, imes-icore, Germany) at 1700 °C. Then, the specimens were polished with abrasive discs of SiC paper (500 and 1200). A forced convection incubator (JISICO, Seoul, Korea) at 37°C stored the samples. For sorption kinetics, the specimens from both groups were subdivided into five specimens per each of the four immersion media used: artificial saliva (AS) as control, ethanol/water (EW), mouth rinse (MW), and cola (PI). Additionally, twenty specimens from each group were allocated for flexural strength (FS) and diametral tensile strength (DTS) testing (Figure 1).

Figure 1. Shows storage of samples in solution at 37°C

Table 1. Product information of the materials used in this study

Material		lot	EXP.	Chemical composition	Country
Lithium Disilicate ($Li_2Si_2O_5$)	L-SiC	L2220111006-038	10/1/2027	Lithium disilicate is a glass-ceramic material composed primarily of lithium, silicon dioxide, and small amounts of other oxides.	USA
Zirconium (ZrO_2)	Y-TZP	DMS-903j50	20/11/2034	Zirconia used in dentistry is typically stabilized with yttria (Yttrium oxide, Y_2O_3) to form yttria-stabilized tetragonal zirconia polycrystal (Y-TZP).	Korea

Table 2. Immersion media used in the study

Immersion media	Chemical Composition	pH
Artificial Saliva (AS)	carboxymethyl cellulose, Sodium chloride NaCl 125.64, Potassium chloride KCl 963.9, Potassium thiocyanate KSCN 189.2, Potassium Dihydrogen orthophosphate KH_2PO_4 654.5, Urea $CO(NH_2)_2$ 200, Calcium chloride dehydrate $CaCl_2$. $2H_2O$ 227.8, Sodium sulphate $Na_2SO_4.10H_2O$ 763.2, Sodium Hydrogen Carbonate $NaHCO_3$ 630.8 and Ammonium chloride NH_4Cl 178 mg/L	6.92
Ethanol/water (EW)	40%Vol Ethanol and 60%Vol water	7.99
Mouth wishes (MW)	Potassium Sorbate, Chlorhexidine (0.12%), Zinc Sulphate, Sodium Fluoride (0.05%) and Ascorbic Acid	5.25
Cloa (PI)	phosphoric acid, carbonated water, sugar, caramel color, natural flavors, and sodium	2.8

Mechanical testing: Flexural strength (FS) is one of the important and required measurements within the ISO 6872:2015 for CAD\CAM ceramics (23) to measure flexural strength using a 3-point bending testing device in a universal testing machine (Zwick/Roell BT1-FR2.5TN Germany) at a crosshead speed of 0.5 mm/min. The flexural strength was obtained by using the formula (24):

$$FS\ (MPa) = \frac{3PL}{2bd^2} \qquad (1)$$

Where P is the load at fracture (N), L is the distance between supports (14 mm), and b and d represent the specimen's width and thickness (mm), respectively. Diametral tensile strength (DTS) was examined according to the ANSI/ADA- Specification #27. The disk specimens were loaded until fracture at a 1mm/min testing speed on a universal testing machine (Zwick/Roell BT1-FR2.5TN Germany). The formula used for diametral tensile strength was (24):

$$DTS\ (MPa) = \frac{2P}{\pi DT} \qquad (2)$$

Where P is load at fracture (N), D is diameter (mm), and T is thickness (mm) of specimens.

Sorption kinetics: Water absorption test was also performed. First, all specimens were placed in a desiccator for one week. After this period, the initial mass (m_0) of each specimen was measured using an electronic scale (KERN 770, Germany), and the dimensions were measured with a digital caliper (INSIZE, China). Then, specimens were divided into four groups (n=4) in accordance with the immersion medium and stored for 180 days at 37℃. The mass was weighed daily until no significant change in weight was observed (m_1), indicating that equilibrium had been attained. This took about 30 days for all immersion media. After drying, the mass of the specimens' absorbed water was measured. The calculations were completed using the equations described below (25):

$$\text{Water sorption } \left({}^{\mu g}/_{mm^3}\right) = \frac{m_0 - m_2}{V} \qquad (3)$$

$$\text{Water solubility } \left({}^{\mu g}/_{mm^3}\right) = \frac{m_1 - m_2}{V} \qquad (4)$$

Results

The results were expressed as means and standard deviations (SDs) for each immersion solution group and for each material (L-SiC and Y-TZP). Mechanical testing (flexural strength and Diametral tensile strength) is summarized in Table 3. Notably, the mechanical properties of both materials decreased overall. The magnitude of this decrease depended on two factors: the degree to which each material was affected by the immersion medium and the composition of the material itself. After 60 days of immersion, a substantial decrease in flexural strength was observed for L-SiC CAD/CAM samples immersed in cola (PI) and ethanol/water (EW), with values falling from 289.34±16.14 MPa to 225.78±19.38 MPa and 222.89±17.16 MPa, respectively. For Y-TZP, the flexural strength decreased from 597.97±9.93 MPa to 570.47±14.29 MPa (EW) and 573.65±13.40 MPa (PI). On the other hand, both materials exhibited only a slight decrease in flexural strength when immersed in artificial saliva (AS) and mouthwash (MW). Figure 2 shows the behaviour of the samples of CAD/CAM through the immersion period. The same behavior was observed for CAD/CAM materials in the DTS test. A significant decrease in values was noted for samples immersed in cola (PI) and ethanol/water (EW) for 60 days. For L-SiC, the DTS decreased from 174.25±8.04 MPa to 155.01±9.53 MPa (EW) and 52.95±3.74 MPa (PI). For Y-TZP, the DTS decreased from 196.61±6.19 MPa to 175.71±11.32 MPa (EW) and 181.96±9.51 MPa (PI). Figure 3 illustrates the DTS behavior of both CAD/CAM samples over the 60-day immersion period.

The means and standard deviations of water sorption and solubility for CAD/CAM materials immersed in various solutions are presented in Figures 4 and 5. Sorption differed slightly between the two ceramic materials, with the following ranking across media: AS < MW < EW < PI. The highest sorption values were recorded in cola (PI), measuring approximately 17.69 µg/mm³ for L-SiC and 29.80 µg/mm³ for Y-TZP after 90 days of immersion. Water solubility also varied significantly depending on the immersion medium, with the ranking: PI < EW < AS < MW. Notably, a marked decrease in water solubility was observed in the PI group after 40 days of storage (Figure 6).

Table 3. Mean Values (± Standard Deviation) of Flexural Strength (MPa) and Diametral Tensile Strength (DTS, MPa) for CAD/CAM Ceramics Following Immersion in Different Media

Immersion Solution	Time (day)	CAD/CAM specimens (n=5)			
		L-SIC		Y-TZP	
		FS (MPa)	DTS (MPa)	FS (MPa)	DTS (MPa)
Artificial Saliva (AS)	1	353.84 (26.22)	174.25 (8.04)	481.17 (42.83)	196.61 (16.19)
	15	343.56 (35.72)	172.57 (4.56)	475.37 (10.95)	195.03 (13.70)
	30	333.56 (30.69)	169.30 (5.97)	467.52 (23.17)	194.20 (18.35)
	45	312.15 (20.55)	168.87 (9.18)	465.57 (37.71)	191.32 (28.97)
	60	302.68 (26.9)	167.59 (6.54)	461.47 (13.73)	190.45 (14.28)
Ethanol/water (EW)	1	353.84(26.22)	174.25 (8.04)	481.17 (42.83)	196.61 (16.19)
	15	328.63 (29.38)	164.46 (1.97)	464.36 (28.18)	193.48 (10.30)
	30	308.11 (36.76)	162.25 (11.4)	451.27 (34.44)	188.78 (18.55)
	45	283.13 (19.78)	157.48 (5.76)	437.92 (34.81)	184.79 (8.74)
	60	229.66 (27.84)	155.01 (9.53)	414.33 (61.6)	181.96 (19.51)
Mouth wishing (MW)	1	353.84(26.22)	174.25 (8.04)	481.17 (42.83)	196.61 (16.19)
	15	323.35 (55.46)	171.08 (7.97)	473.67 (26.4)	194.69 (8.90)
	30	317.86 (28.45)	170.34 (6.80)	470.54 (10.49)	191.95 (21.54)
	45	296.93 (47.68)	166.28 (2.75)	466.04 (14.9)	188.81 (18.16)
	60	291.06 (47.95)	165.46 (7.18)	457.93 (32.24)	187.01 (16.92)
Cola (PI)	1	353.84(26.22)	174.25 (8.04)	481.17 (42.83)	196.61 (16.19)
	15	324.70 (46.97)	167.62 (5.11)	465.45 (31.31)	191.80 (8.01)
	30	281.62 (62.54)	161.85 (4.19)	453.19 (27.61)	187.95 (20.27
	45	266.21 (43.43)	159.61 (3.63)	439.49 (44.62)	184.37 (26.36)
	60	235.06 (31.44)	152.95 (3.74)	418.02 (26.4)	175.71 (11.31)

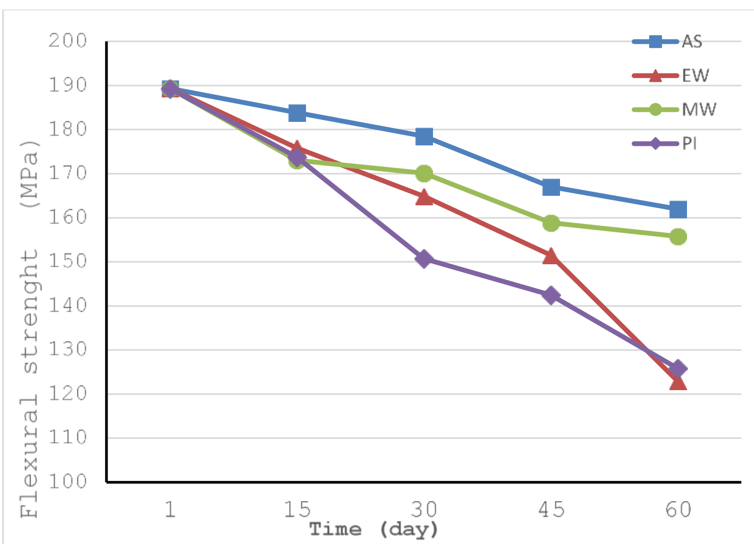

Figure 2. Mean for flexural strength of the CAD\CAM after immersion in solutions

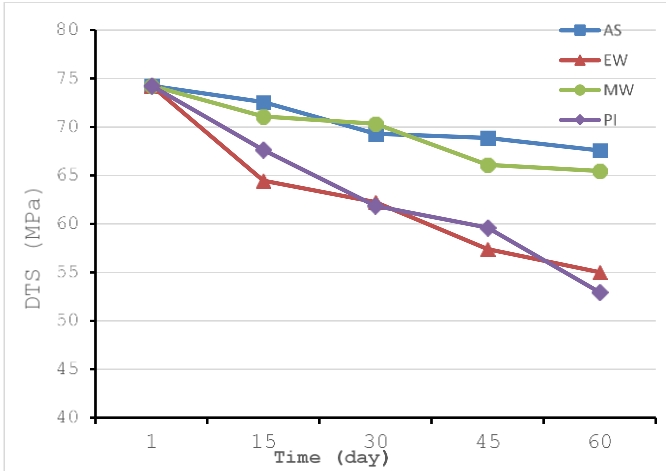

Figure 3. Mean for DTS of the CAD\CAM after immersion in solutions

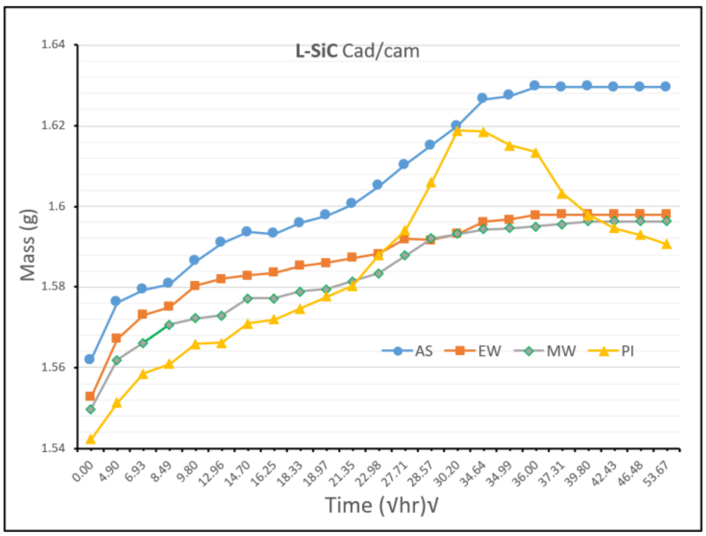

Figure 4. Plot of mass against the time for L-SIC CAD\CAM immersion 120 days

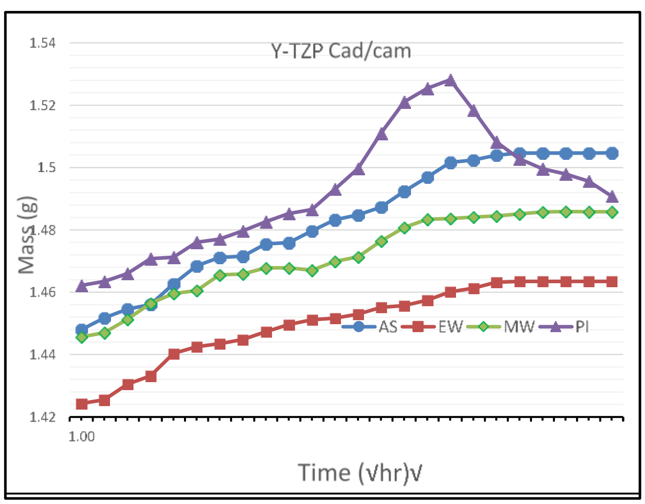

Figure 5. Plot of mass against the time for Y-TZP CAD\CAM immersion 120 days

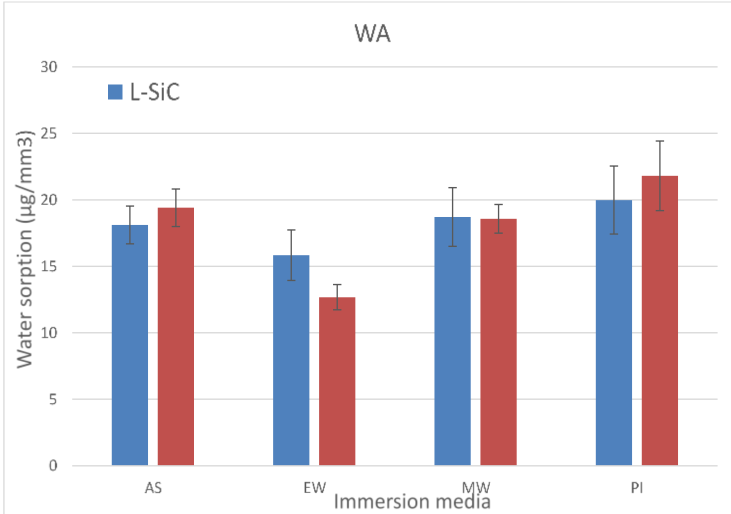

Figure 6. Mean and SD for water sorption (µg/mm^3) of the CAD\CAM after immersion in solutions

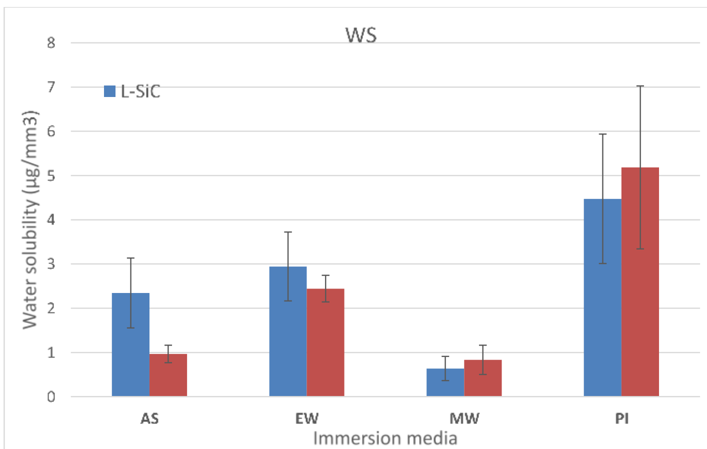

Figure 7. Mean and SD for water solubility (µg/mm^3) of the CAD\CAM after immersion in solutions

Discussion

The findings showed that flexural strength values decreased significantly when specimens were immersed in EW and PI. For L-SiC, the flexural strength decreased by approximately 35% after immersion in EW and 33% after immersion in PI. In contrast, reductions in AS and MW were about 14% and 17%, respectively. For Y-TZP, the decreases were slightly less pronounced, reaching approximately 13.1% in EW and 13.7% in PI.

Although many individuals consider the consumption of soft drinks and fruit juices to be relatively benign, this habit is not as harmless as commonly believed. Regular intake of these beverages is associated with several serious health concerns. The acid and sugar they contain exhibit acidogenic and cariogenic properties, which can lead to dental caries and enamel erosion (26). The primary objective of dentistry is to enhance an individual's quality of life. This can be achieved through disease prevention, pain alleviation, improved mastication, enhanced speech, and aesthetic augmentation. One of the most important properties determining the durability of dental materials in the oral cavity is their resistance to dissolution or disintegration, which is affected by common consumable foods and drinks (e.g., water, carbonated soft drinks, alcoholic beverages, and food derivatives) (16).

A key property determining the durability of dental materials in the oral cavity is their resistance to dissolution or disintegration, as degradation results in the deterioration of their physical and mechanical properties. The extent of this deterioration is influenced by both exogenous and endogenous factors. Exogenous factors encompass patient-related behaviors, including dietary habits, tooth brushing, and the consumption of soft drinks, alcoholic beverages, and hot drinks. Endogenous factors include particle size, hardness, water sorption, hydrolysis, and surface roughness. To overcome certain limitations associated with powder-liquid and paste mixing systems, manufacturers have recently introduced CAD/CAM blocks for interim restorations (27).

The findings demonstrated that MW contains ascorbic acid, while PI contains phosphoric acid. The pH of these solutions was measured at 5.25 for MW and 2.8 for PI, compared to 6.92 for artificial saliva. The increased acidity of the MW and PI solutions likely influenced the composition of the CAD/CAM ceramics. This lowered pH may have accelerated the degradation of the restorative materials within the ceramic structures (28).

The chemical agents present in ethanol/water (EW) and cola (PI) can adversely affect CAD/CAM samples through chemical degradation. Water sorption and solubility properties are critical for the long-term success of a restorative material, as this affinity for water alters its physical properties and induces dimensional changes (29, 30).

One advantage of CAD/CAM restorations is the reduced need for intraoral repair. This is because hydrofluoric acid - traditionally required for surface treatment of glass-ceramic restorations - is no longer necessary (31). The chemical agents present in EW and PI can adversely affect CAD/CAM samples via their chemical degradation. Water sorption and solubility are critical properties for ensuring the long-term success of a restorative material, as water affinity induces dimensional changes and alters physical properties (29, 30).

The water absorption characteristics of ceramics are influenced by several factors, including the composition of the ceramics, their orientation, temperature, surface area exposure, void content, and the hydrophilicity of the individual components. The findings indicate that the number of absorbed solutions increases with prolonged immersion time for both materials. During the initial phase of the experiment, specifically from days 2 to 7, there is a significant increase in weight, indicating rapid moisture penetration into the ceramic materials. This behavior is consistent across all solutions examined in this study. The observed pattern can be attributed to the water's penetrability and capillary action, which become prominent as water infiltrates through the voids and pores present in the ceramics. However, the weight increase does not maintain a consistent relationship with immersion time. After 20 days of immersion, the rate of solution absorption begins to decelerate, ultimately reaching a saturation point after 60 days. At this point, the weight of the specimens remains almost constant, with a difference of 0.0001 g, and measurements continue for 180 days. Overall, the water absorption behavior of L-SiC and Y-TZP in the four solutions can be characterized as a Fickian process (Fick's second law). An initial increase in mass was noted, reflecting predominant water adsorption, followed by a trend toward saturation as immersion time progressed. Similar findings have been documented in previous research. Additionally, the results indicate that after 104 days of immersion, the weight gain of specimens in acidic solutions was comparable to that of specimens immersed in artificial saliva. Analysis of the solutions showed that the pH remained nearly neutral for both materials, except for specimens immersed in cola (PI), which exhibited a decrease in mass due to the corrosive effects of the low pH environment.

This in vitro study of CAD/CAM ceramics revealed that immersion in various solutions led to notable changes in the flexural strength of L-SiC and Y-TZP, particularly when exposed to alcohol (EW) and cola (PI). The average flexural strength decreased across all specimen groups. Despite this, the mean diametral tensile strength (DTS) remained within clinically acceptable limits, indicating that the mechanical properties of these materials are impacted by solution immersion. Additionally, only slight changes in water absorption and solubility were observed for most solutions, except cola, which caused a noticeable increase that remained within clinically acceptable ranges. Dentists should advise patients about the impact of certain beverages on the physical and mechanical properties of ceramic restorations.

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