

COMPARATIVE EVALUATION OF NOVEL DENTAL MATERIALS UNDER CORROSIVE ATTACK BY ARTIFICIAL SALIVA IMMERSION

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ABSTRACT

Aim of the study: This study evaluates the behavior of novel dental materials under corrosive attack caused by artificial saliva immersion at varying pH levels.

Material and methods: Three materials were tested: zirconium dioxide (ZrO₂), lithium disilicate-based ceramic (IPS e.Max CAD), and a hybrid ceramic composite (VarseoSmile Crownplus). The samples underwent immersion in artificial saliva at pH values of 3 and 5.5 for periods of 3, 7, and 14 days. Surface morphology and mass variations were assessed using Scanning Electron Microscopy (SEM) and analytical precision weighing.

Results: Zirconium dioxide exhibited the highest chemical stability under all tested conditions, with minimal mass variations. Lithium disilicate ceramics showed moderate stability, while the hybrid ceramic composite demonstrated the greatest mass loss, indicating susceptibility to acidic environments.

Conclusions: Zirconium dioxide proves to be the most reliable material for corrosive resistance in dental applications, followed by lithium disilicate ceramics. Hybrid ceramic composites require further development to enhance stability in acidic conditions.

Key words: zirconium dioxide, lithium disilicate, hybrid ceramic composite, artificial saliva, corrosion resistance

INTRODUCTION

Despite the wide availability of dental biomaterials, no material possesses ideal properties [1]. The selection of biocompatible materials depends on several factors, such as their mechanical and biological characteristics, stability in the oral environment (including corrosion in saliva), functionality, final cost, and aesthetics [2, 3].

Moreover, biomaterials are expected to be multifunctional, biologically active, and to exhibit combined features, as exemplified by composite dental fillings that provide antibacterial and remineralization effects [4, 5]. Over the past decade, dental biomaterials

have evolved extensively, and their functionality has been progressively improved [1].

Corrosion is defined as the loss of material from the surface as a result of chemical attack. Corrosion byproducts dissolve into oral fluids, causing changes in the material's mass. This process leads to a rougher surface, which collects food debris and continues to corrode over time [6].

Manufacturers of dental materials often classify and promote their products based on their resistance to chemical degradation in the oral environment and their mechanical properties, including flexural strength, fracture toughness, critical flaw size, and fatigue

resistance. Using CAD/CAM technology, the fabrication of fixed zirconia prosthetic restorations has become a rapid and efficient process, enabling the production of high-quality restorations within a relatively short time frame [7].

Additionally, IPS™ e.Max has gained increasing popularity since its introduction due to its combination of excellent mechanical properties and acceptable aesthetics [8]. Furthermore, three-dimensional (3D) printing processes are widely utilized in various fields of dental technology and dentistry [9]. An essential advantage of these processes is the manufacturing efficiency, which reduces material waste compared to subtractive milling techniques [10].

In February 2020, a material known as VarseoSmile Crown plus (BEGO, Bremen, Germany) was introduced for the additive manufacturing of definitive tooth-colored dental restorations [11]. It is approved for single-tooth restorations such as crowns, inlays, onlays, or veneers and was the first 3D-printable material approved for definitive dental restorations [12]. This material is a hybrid ceramic composite infiltrated with ceramic, consisting of a methacrylic ester matrix with ceramic fillers, and is classified as a “resin-matrix ceramic” (RMC) [13].

The aim of this study was to evaluate the comparative behavior of dental materials subjected to corrosive attack through immersion in artificial saliva.

MATERIAL AND METHODS

The three materials studied comparatively were zirconium dioxide (ZrO_2) produced by ERNST HINRICHS Dental GmbH, Germany, coded as C1; E-max ceramic (lithium disilicate - $Li_2Si_2O_5$) produced by Ivoclar Vivadent AG, Liechtenstein, coded as C2; and a composite material (ceramic resin) produced by BEGO Bremer Goldschlägerei Wilh.

Herbst GmbH & Co. KG, coded as C3.

The experimental samples evaluated in this study were prepared through milling (for the zirconium dioxide and E-max ceramic samples) and 3D printing (for the ceramic resin material) in the form of discs measuring 5 mm in height and 10 mm in diameter.

The comparative evaluation of the biodegradation of the three materials was conducted by immersing them in artificial saliva and monitoring their mass at three time intervals (3, 7, and 14 days), at a constant temperature of 37 ± 0.1 °C and two pH levels (3 and 5.5). Initially, the artificial saliva had a pH of 7.6, which was adjusted to the desired values using a 1M-HCl solution.

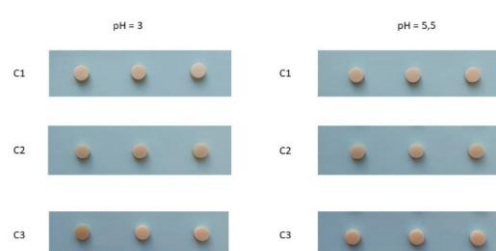


Figure 1. Images of the tested samples

During the tests, the samples were immersed in the two media and maintained at 37 ± 0.1 °C using a Memmert IF55 incubator. Before immersion, both the tested samples and the containers in which they were placed were sterilized using a UV-C lamp (280–100 nm) to prevent biofilm formation on their surfaces during the tests.

Three samples of each material were used to observe their mass changes in the two media at the predetermined time intervals of 3, 7, and 14 days. A Kern ALT 100-5AM analytical balance with a precision of 0.01 mg was employed for mass measurements.

During the tests, the immersion media were replaced periodically (every 3 days) to maintain a consistent ionic concentration in

the solutions and to prevent biofilm formation. Both the samples and containers were sterilized with UV-C irradiation after each replacement.

The samples were removed from the testing media at the predetermined time intervals, rinsed with distilled water, dried with a stream of hot air for 5 hours at a temperature of 80°C in a Memmert UF55 drying oven, and subsequently stored in a desiccator prior to weighing. Mass measurements were repeated five times for each sample to ensure the highest possible accuracy of the results.

The mass difference of the samples was calculated using the following formula:

$$\Delta m = m_f - m_i, \text{ where:}$$

Δm represents the mass difference,
 m_f is the final mass of the sample after immersion,

m_i is the initial mass of the sample before immersion.

RESULTS

The evolution of the sample masses over the specified time intervals is presented in Table 1 for the samples tested in artificial saliva with a pH of 3 and in Table 2 for those tested in artificial saliva with a pH of 5.5.

Table 1. Masses of the samples tested in artificial saliva at pH 3

Mass (mg)							
day		pH = 3					
		C1		C2		C3	
		m_i	m_f	m_i	m_f	m_i	m_f
3		2248,61± 0,00	2248,6± 0,00	962,77± 0,00	962,77± 0,00	466,8± 0,01	465,51± 0,00
	Δm	-0,01		0,00		-1,29	
7		2250,37± 0,01	2250,35± 0,01	966,06± 0,01	966,06± 0,00	525,89± 0,02	525,59± 0,00
	Δm	-0,02		0,00		-0,30	
14		2250,43± 0,00	2250,45± 0,00	965,01± 0,02	964,99± 0,00	539,47± 0,00	539,33± 0,00
	Δm	0,02		-0,02		-0,14	

Table 2. Masses of the samples tested in artificial saliva at pH 5.5

Mass (mg)							
day		pH = 5,5					
		C1		C2		C3	
		m _i	m _f	m _i	m _f	m _i	m _f
3		2244,97± 0,01	2244,98± 0,00	973,77± 0,02	962,77± 0,00	466,8± 0,01	465,51± 0,00
	Δm	0,01		0,00		-0,40	
7		2251,16± 0,00	2251,17± 0,00	958,83± 0,02	966,06± 0,00	525,89± 0,02	525,59± 0,00
	Δm	0,01		0,02		-0,29	
14		2250,33± 0, 01	2250,30± 0, 01	961,8± 0,01	964,99± 0,00	539,47± 0,00	539,33± 0,00
	Δm	-0,03		0,04		-0, 13	

Based on the immersion tests conducted for the investigated samples and the mass values from Tables 1 and 2, it can be observed that the material C3 (the hybrid ceramic) exhibited the greatest mass loss regardless of pH value. After 3 days of immersion, it undergoes significant mass loss, followed by a slight increase in mass by day 14.

Additionally, it is evident that materials C1 and C2 are chemically more stable across both pH values, as they do not show significant mass variations after immersion testing.

Material C1 (zirconium dioxide) demonstrates different tendencies depending on pH value. At a neutral pH of 5.5, there is a slight mass decrease after 14 days, from a constant value maintained over the first 7 days of immersion (0.01 mg) to -0.03 mg on day 14, corresponding to a total loss of approximately 0.04 mg. At a pH of 3, the mass decreases during the first 7 days to -0.02 mg, followed by a recovery of approximately 0.04 mg on

day 14.

At pH 3, material C2 is chemically stable for the first 7 days, followed by a slight decrease of ~0.02 mg. Conversely, in artificial saliva with a pH of 5.5, material C2 exhibits a mass gain, showing a progressive increase over the 14 days of immersion, reaching a maximum of 0.04 mg.

Material C3 exhibits a sharp mass loss during the first 3 days, more pronounced at pH 3, where the mass decreases to -1.3 mg, followed by a recovery to approximately -0.02 mg. At pH 5.5, the mass of material C3 follows a similar upward trend but is less pronounced compared to the more acidic pH.

DISCUSSIONS

Microhardness, fracture toughness [14, 15], and color stability [16, 17] of materials obtained through CAD-CAM technology in low pH environments have been previously

investigated in the literature. Currently, no standardized in vitro erosion exposure protocols exist that can accurately simulate clinical conditions [18]. The ISO standard for measuring the solubility of dental ceramics involves immersing the material for 16 hours in 4% citric acid at 80°C [19].

Conversely, other studies have reported no significant effects of erosive acids on CAD-CAM materials [20, 21]. This variability in results can be attributed to the different microstructures of the materials studied, variations in the erosion simulation protocols applied, and differences in testing temperatures. Additionally, measurement methods and parameters used vary between studies, leading to differing results that preclude a clear consensus.

The performance of prosthetic restorations is influenced by both the nature and properties of the materials, as well as their behavior in the environment in which they are intended to be used. It is well known that the oral environment is biologically aggressive, due to variations in temperature and pH caused by food and beverage consumption, as well as the presence of oral bacteria.

The present study demonstrated that the analyzed samples obtained through CAD-CAM technology exhibited greater resistance

to acid attack compared to the samples obtained through 3D printing. The results of this study are supported by similar studies, which have shown that CAD-CAM materials, such as zirconia and lithium disilicate, exhibit superior chemical and mechanical stability due to their uniform microstructure and advanced sintering processes [15, 22, 23]. In contrast, 3D-printed materials, while promising due to the efficiency of the manufacturing process, are more susceptible to mass loss and surface changes in acidic environments, as reported in recent studies [9, 24].

Low pH oral environments with weak buffering capacity can cause the erosion of dental ceramics, resulting in a porous, rough surface [25]. The rough surface of ceramic restorations can act as a potential abrasive surface for opposing natural teeth [26].

CONCLUSIONS

Immersion tests in artificial saliva at two pH levels over periods of 3, 7, and 14 days revealed that the tested materials exhibited different behaviors. It was observed that material C1 is the most chemically stable in artificial saliva, regardless of the pH value, showing the smallest mass variation, followed by C2 with similar results, and C3, which exhibited the largest mass variations.

Acknowledgements

The authors would like to thank Assoc. Prof. PhD Mihai Cosmin Cotruț, Faculty of Materials Science and Engineering, National University of Science and Technology POLITEHNICA Bucharest, for the support and constructive comments.

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