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ORIGINAL RESEARCH

THE INFLUENCE OF CITRIC ACID ON THE MICRO-HARDNESS AND SURFACE ROUGHNESS OF CONTEMPORARY CAD/CAM INDIRECT RESTORATIVE MATERIALS: AN IN VITRO ANALYSIS

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ABSTRACT

Background: Despite their clinical advantages, restorative materials remain susceptible to degradation under intraoral conditions, particularly in acidic environments resulting from dietary habits . Citric acid, commonly present in citrus fruits and beverages, is a prevalent erosive agent with the potential to compromise the long-term integrity of dental restorations[7] This in vitro study aimed to assess the effect of citric acid on the micro-hardness and surface roughness of three novel CAD/CAM indirect restorative materials: lithium disilicate (IPS e.max CAD), zirconia-reinforced lithium silicate (ZLS), and resin nano-ceramic (Cerasmart).

Materials and Methods: Sixty standardized rectangular specimens (8 × 5 × 2 mm) were prepared (n = 20/material). Each group was subdivided into control (immersed in artificial saliva) and experimental (immersed in 2% citric acid, pH = 3.2) subgroups. A digital Vickers hardness tester was used to detect micro-hardness and atomic force microscopy (AFM) was used to assess surface roughness following a 7-day aging regimen at 37°C.Non-parametric statistical analysis (Mann-Whitney, Kruskal-Wallis) was conducted at a significance level $p \leq 0.01$.

Results: Citric acid significantly reduced micro-hardness and increased surface roughness in all materials tested ($p \leq 0.01$). Among the groups, zirconia-reinforced lithium silicate exhibited higher resistance, while resin nano-ceramic demonstrated the most susceptibility to acidic degradation.

Conclusions: Exposure to citric acid led to a significant deterioration in both surface and mechanical integrity, with material-dependent variations. The findings emphasize the need for cautious material selection in patients with high dietary acid exposure.

Keywords: Citric acid, CAD/CAM, Micro-hardness, Surface roughness, Emax CAD, Zirconia-reinforced lithium silicate, Resin nano-ceramic, AFM, Vickers hardness

INTRODUCTION

Indirect restorative dentistry makes extensive use of dental ceramics because of their excellent mechanical and cosmetic qualities. With the introduction of computer-aided design/computer-aided manufacture (CAD/CAM) technology, ceramic repair fabrication has become much more accurate, repeatable, and efficient[1,2]. Contemporary CAD/CAM ceramics encompass a variety of compositions, including lithium disilicate, zirconia-reinforced silicates, and resin-based nano-ceramics, each exhibiting distinct physicochemical characteristics [3,4].

Despite their clinical advantages, restorative materials remain susceptible to degradation under intraoral conditions, particularly in acidic environments resulting from dietary habits [5,6]. Citric acid, commonly present in citrus fruits and beverages, is a prevalent erosive agent with the potential to compromise the long-term integrity of dental restorations[7].

Previous studies have demonstrated that exposure to acidic media adversely affects the surface topography, gloss retention, and micro-mechanical properties of ceramics [4,8].

The present investigation aims to provide a systematic evaluation of the influence of citric acid on surface roughness and micro-hardness of three state-of-the-art CAD/CAM restorative materials, thereby contributing to the optimization of material selection and longevity in clinical scenarios characterized by acidic challenges.

MATERIALS AND METHODS

Study Design and Sample Distribution: This in vitro experimental study included three different CAD/CAM restorative materials: lithium disilicate glass ceramic (IPS e.max CAD, Ivoclar Vivadent), zirconia-reinforced lithium silicate ceramic (Vita Suprinity, Vita Zahnfabrik), and resin nano-ceramic (Cerasmart, GC Dental). With defined dimensions of 8 mm × 5 mm × 2 mm, 60 rectangular specimens ($n = 20$ per group) were created using a water-cooled low-speed diamond saw (Isomet 1000, Buehler, IL, USA). Every material has an A2 shade.

Each group of 20 samples was subdivided equally into two main subgroups:

- **Control group ($n = 10$):** Storage in synthetic saliva at pH 7.
- **Experimental group ($n = 10$):** Storage in 2% citric acid solution at pH 3.2.

Each of these subgroups was further divided:

- **Five specimens** from each were tested for **micro-hardness**.
- **Five specimens** were tested for **surface roughness**.

This yielded six subgroups (3 materials × 2 media) and ensured all combinations were represented for each outcome measure.

Specimen Processing: For lithium disilicate and zirconia-reinforced specimens, glazing and crystallization were performed using a ceramic furnace (Zetain Sintering Furnace) as per manufacturer instructions. Cerasmart specimens required no thermal treatment. All specimens were polished using felt discs with diamond suspension (15 µm and 0.6 µm grit; Extec Corp, Enfield, CT, USA) at 5000 rpm under continuous water cooling for 60 seconds per specimen to simulate clinical surface finish.

Aging Procedure: Artificial saliva was prepared according to a standard laboratory protocol and maintained at 37°C in an incubator. Citric acid solution (2% w/v) was freshly prepared using analytical-grade citric acid powder dissolved in distilled water, adjusted to pH 3.2. Specimens were immersed for 7 consecutive days, and the solutions were replaced every two days to avoid contamination. Prior to testing, all specimens were dried and rinsed with distilled water after age.

Micro-Hardness Testing: Vickers micro-hardness was assessed use a digital hardness tester with a 300 g load applied for 10 seconds. Five indentations were made randomly on each sample's surface. Using the conventional procedure, the average of the five indentations was used to determine the Vickers Hardness Number (VHN). According to ASTM rules, indentations with chipped edges or unusual shapes were not included.

Surface Roughness Testing: Surface roughness was assessed use atomic force microscopy (AFM, NaioAFM, Nanosurf AG) in non-contact mode. Scanning was performed at a fixed area of 20 × 20 µm with a scanning speed of 0.8 Hz. Arithmetic mean surface height (Sa) and root mean square roughness (Sq) were measured. For statistical analysis, the average of three scans per specimen was employed.

Statistical Analysis: Non-parametric tests were used because of the non-normal data distribution, which was verified by the Shapiro-Wilk test. Within the same material, the control and acid-exposed groups were compared using the Mann-Whitney U test. To compare various materials, the Kruskal-Wallis H test and Dunn's post hoc test were used. All tests were conducted with a significance level of $p < 0.01$.

RESULTS

Micro-Hardness:

Descriptive statistics for micro-hardness (mean ± standard deviation, minimum and maximum values) of all groups are summarized in Table 1. All three materials showed a statistically substantial reducing in hardness values after immersion in citric acid.

Table 1. Descriptive statistics for Vickers micro-hardness values (VHN)

Material	Condition	N	Mean ± SD	Min	Max
IPS e.max CAD	Control	5	547.88 ± 16.83	529.70	570.10
	Acid	5	464.56 ± 7.66	457.70	475.40
ZLS	Control	5	642.66 ± 26.89	625.50	689.90
	Acid	5	492.00 ± 7.70	480.20	498.80
Cerasmart	Control	5	92.98 ± 4.00	88.90	98.90
	Acid	5	82.82 ± 1.92	80.40	85.30

The Mann-Whitney U test indicated a highly significant reduction ($p \leq 0.01$) in micro-hardness values post-treatment for each material group, as shown in Table 2.

Table 2. Mann-Whitney test comparing micro-hardness before and after acid exposure

Material	p-value
E.max	0.008
ZLS	0.008
Cerasmart	0.008

Additionally, substantial differences between materials under acidic and control conditions were established by the Kruskal-Wallis test ($p < 0.01$).Post-hoc comparisons revealed the following order of hardness resistance: ZLS > E.max > Cerasmart.

Surface Roughness

Surface roughness values also showed significant increases after exposure to citric acid. Descriptive statistics are presented in Table 3.

Table 3. Descriptive statistics for surface roughness values (Ra, nm)

Material	Condition	N	Mean ± SD	Min	Max
IPS e.max CAD	Control	5	7.36 ± 0.66	6.70	8.30
	Acid	5	15.31 ± 0.84	14.08	16.16
ZLS	Control	5	39.63 ± 5.97	32.98	44.53
	Acid	5	59.42 ± 6.97	52.00	67.92
Cerasmart	Control	5	110.37 ± 1.83	107.80	112.20
	Acid	5	141.24 ± 8.96	132.00	150.40

According to Table 4, the results of the Mann-Whitney test revealed statistically significant variations in surface roughness between all three materials before and after acid immersion ($p<0.01$).

Table 4. Comparing surface roughness before and after exposure to acid using the Mann-Whitney test:

Material	p-value
E.max	0.0093
ZLS	0.0091
Cerasmart	0.0094

Kruskal-Wallis analysis revealed that material type significantly influenced surface roughness in both control and acid conditions ($p \leq 0.01$). The order of resistance to roughening was: E.max > ZLS > Cerasmart.

DISCUSSION

The surface and mechanical characteristics of every ceramic material examined in this study showed statistically significant deterioration after being exposed to citric acid, highlighting the importance of understanding chemical interactions within the oral environment. The findings align with previous studies that report acidic agents can adversely affect the micro-hardness and surface morphology of indirect restorative materials [9, 10].

Citric acid, as a weak organic acid with chelating properties, facilitates ionic dissolution within the ceramic matrix. In lithium disilicate-based ceramics such as IPS e.max CAD, the glassy matrix is particularly vulnerable to acid attack. The dissolution of alkali ions, notably lithium and silica, compromises the microstructural integrity, leading to a notable decrease in hardness and an increase in surface irregularities [11]. The observed reduction in Vickers micro-hardness from 547.88 ± 16.83 to 464.56 ± 7.66 confirms the susceptibility of this material to acid-induced degradation. Furthermore, previous studies have demonstrated that the leaching of ions from the glass phase contributes to volumetric shrinkage and microcrack formation, thus exacerbating surface wear [12].

Zirconia-reinforced lithium silicate ceramics (ZLS) presented comparatively higher resistance to acid exposure, yet still showed a significant reduction in hardness and increase in roughness. This could be attributed to the partial presence of a glass phase susceptible to degradation, despite the reinforcement provided by zirconia crystals. As indicated by Elraggal et al. (2022) [13] , citric acid can form complex bonds with surface oxides of zirconia, thereby inducing surface demineralization and ion release. The interaction is believed to initiate at the glass-zirconia interface, leading to surface topography alterations without complete structural collapse.

Cerasmart, a resin nano-ceramic, demonstrated the most significant deterioration among the three materials. The decrease in micro-hardness and the rise in surface roughness following exposure to citric acid are consistent with its hybrid structure, which includes an organic matrix susceptible to hydrolytic degradation. As shown in studies by Al-Harbi et al. (2017) and Unalan Degirmenci et al. (2025) [14, 15] , the ester, amide, and urethane bonds within the polymeric matrix are prone to cleavage in low-pH conditions, resulting in filler-matrix debonding and surface porosity. Additionally, Aldamaty et al. (2020) [16] , reported that acidic exposure compromises the mechanical performance of resin-ceramics more than glass ceramics, due to the polymer matrix's affinity for water absorption and subsequent hydrolysis.Surface roughness data from AFM revealed consistent patterns with the hardness findings. Acid-induced surface topography changes increase the potential for plaque accumulation, discoloration, and wear on antagonistic dentition. Cruz et al. (2020) and Kukiattrakoon et al. (2011) [17, 18], emphasized that prolonged exposure to acidic solutions alters the ceramic surface at the nanoscale, diminishing clinical aesthetics and longevity. Even mild surface roughening, as documented in the present study, may predispose restorations to secondary complications such as bacterial adhesion and marginal breakdown.

From a clinical standpoint, the implications are substantial. Patients with diets rich in acidic beverages or systemic conditions such as GERD may experience accelerated deterioration of certain ceramic restorations. Thus, material selection must consider both chemical and mechanical resistance, particularly when restoration longevity and patient-specific risk factors are evaluated.

In summary, the findings reinforce the chemical vulnerability of all tested CAD/CAM materials under acidic challenge, albeit to varying degrees. While zirconia-reinforced ceramics offer improved resistance, they are not immune. The selection of materials for indirect restorations must balance aesthetics, strength, and environmental durability.

Limitations and Future Work:

This study, while methodologically robust, has limitation. The in vitro design lacks the complex variables of intraoral conditions, such as salivary flow, enzymatic activity, and masticatory forces. Additionally, only citric acid was evaluated; other acids (e.g., phosphoric, acetic) may yield different degradation profiles.

Future studies should incorporate dynamic aging models, broader acidic environments, and long-term cycling to simulate real-life conditions more accurately. Clinical trials are also recommended to validate these findings in vivo.

CONCLUSIONS

The mechanical strength and surface quality of CAD/CAM restorative materials are severely weakened by citric acid, with resin nano-ceramics being most affected and zirconia-reinforced ceramics demonstrating superior resistance. These findings provide a foundational basis for clinical decision-making, especially in acid-challenging oral environments.

DECLARATIONS

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Competing interests

The authors declare no competing interests.

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