

## COMPARATIVE EVALUATION OF CITRIC ACID IMPACT ON RETENTION STRENGTH OF CAD/CAM INDIRECT RESTORATIVE MATERIALS: A CRITICAL IN VITRO INVESTIGATION

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### ABSTRACT

**Background:** The ongoing advancements in all-ceramic and hybrid restorative materials have fundamentally transformed aesthetic dentistry, yet their long-term clinical success remains intricately linked to their chemical resilience under challenging intraoral conditions. Among these, frequent exposure to acidic dietary components such as citric acid poses a significant threat to the integrity and retention of indirect restorations. **Objective:** This study aimed to critically evaluate the effect of citric acid immersion on the retention strength of three recently introduced CAD/CAM indirect restorative materials: lithium disilicate (IPS E.max CAD), zirconia-reinforced lithium silicate (VITA Suprinity), and resin nano-ceramic (Cerasmart). **Materials and Methods:** Thirty sound maxillary first premolars were extracted and randomly allocated into three equal groups ( $n = 10$ ), each corresponding to one of the tested materials. Each group was further subdivided, with half immersed in artificial saliva (control) and the other half in a 2% citric acid solution (pH 3.20) at 37 °C for seven days. Following thermocycling, crowns were adhesively cemented using dual-cure resin cement, and retention strength was evaluated via pull-off testing. Failure modes were assessed microscopically, while statistical significance was determined using Mann–Whitney and Kruskal–Wallis tests ( $p \leq 0.01$ ). **Results:** All groups exhibited a significant increase in retention strength following citric acid immersion ( $p \leq 0.01$ ). The resin nano-ceramic group demonstrated the highest retention mean value ( $149 \pm 5.96$  N), followed by zirconia-reinforced lithium silicate ( $88.56 \pm 7.48$  N), and lithium disilicate ( $81 \pm 4.95$  N). Moreover, failure mode analysis revealed that the resin group primarily exhibited mixed failures, which underscores its superior adhesive properties and cohesive integrity, compared to the lithium disilicate group that showed higher occurrences of adhesive failures, indicative of potential surface deterioration and compromised bonding effectiveness. In summary; The results emphasize the impact of acid, on how well CAD/CAM materials hold up over time; emphasizing the strong chemical durability and adhesive strength of resin nano ceramics. The observations support the idea of choosing materials based on patient needs and dietary considerations to improve the long term success of restorations.

**KEYWORDS:** CAD/CAM ceramics, citric acid, retention strength, resin nano-ceramic, adhesive failure, dental materials.

### INTRODUCTION

The ongoing development of all materials in the field of restorative dentistry has greatly impacted how dentists work due, to the notable progress made in their durability and aesthetics alongside their biocompatibility (Huang et al. 2023).

In response to increasing expectations for better aesthetic results in dental treatments over time. This has led to a rise in the need for metal dental restorations. Ceramics have become choices for visible areas, in the mouth according to Surana and Mahajan (2020).

The durability of these materials, over time doesn't just rely on their strength. Is greatly affected by how stable they remain in the complex conditions of the mouth.

Exposure to substances in the environment poses a significant threat to the durability of ceramic dental restorations; among these acids primarily sourced from what we consume daily plays a crucial role. Citric acid found abundantly in fruits like lemons and oranges along with beverages and processed food products has been proven to decrease oral pH levels as, per research by Yokel in 2025.

Repetitive exposure to acidic environments can lead to the deterioration of surfaces and the weakening of mechanical characteristics while also causing negative impacts on the bonding, between different materials used for restoration (referencing Amin *et al.*, 2022).

Furthermore, the effects of exposures are influenced by the unique makeup and microscopic structure of various types of ceramics. For example, lithium disilicate ceramics with a high glass content may be more prone to surface etching caused by acids as compared to zirconia reinforced lithium silicate and resin nano ceramics. These alternate ceramics are specifically engineered to provide stability in terms of crystalline structure and resistance, to chemicals (Banh *et al.*, 2021; Alshali *et al.*, 2022).

Hybrid resin nano ceramic materials combine fillers with a flexible resin matrix. This combination could provide adaptability and increased resistance to acidic degradation. As a result of this enhancement in durability and flexibility under changing conditions, in the mouth (as noted by Cui in 2023) these materials are able to maintain their properties effectively.

The way you decide to stick things and prepare the surface plays a big part in how well indirect restorative materials stay in place for a long time. Studies before have highlighted how important resin cements and silane coupling agents are, in making sure the bond lasts well against heat and other stresses (Tam *et al.*, 2024).

Exploring these factors is essential to understand how citric acid impacts the bond strength of modern CAD/CAM indirect dental materials recently brought into use, for restorations. This research seeks to compare how lithium disilicate performs against zirconia reinforced lithium silicate and resin nano materials in an acidic setting that mimics regular dietary contact. Through this exploration, the research endeavors to provide clinically relevant insights to inform material selection and optimize restorative longevity in patients predisposed to acidic oral conditions.

## MATERIALS AND METHODS

In pursuit of elucidating the influence of citric acid on the retention strength of contemporary CAD/CAM indirect restorative materials, this meticulously designed in vitro investigation utilized thirty sound maxillary first premolars extracted for orthodontic purposes from patients aged 18 to 24 years, following ethical approval granted by the Research Ethics Committee at the Faculty of Dentistry, University of Mosul (REC reference no: Uom.Dent.24/1009). Were selected based on their crown measurements of approximately 9 to 9.6 mm in length of bucco-lingual 7 to 7.4 mm in width from mesio-distal and between 7.7 to 8.8 mm in cervico-occlusal height, from gum line to top all measured using a digital caliper (Wafaie *et al.*, 2018).

Each tooth was carefully examined under dental loupe at a magnification of 3.5x., to confirm the absence of fractures, caries, or resorptive defects, thereby ensuring uniformity and methodological rigor.

The extracted teeth were subjected to thorough debridement using ultrasonic scaling and polished meticulously to remove soft tissue remnants and calculus, after which they were stored in distilled water at ambient temperature to preserve their structural integrity until subsequent processing. The specimens were then embedded in autopolymerizing acrylic resin within polyvinyl rings, maintaining a standardized exposure  $2 \pm 0.5$  mm below to the cemento-enamel junction, thereby simulating the biologic width and ensuring vertical stability during mechanical testing.

Impressions will be made of the tooth using silicone putty before preparing the cavity to ensure standardization and reduction. Each premolar was individually impressed to create a silicon index. The impression was then divided vertically into two halves to capture a side view of the preparation. To achieve standardized preparation All group's teeth were prepared uniformly under a water spray high-speed handpiece (NSK, Japan) that was attached to the surveyor's vertical arm as, occlusal reduction was conducted to a depth of 1.5–2 mm using a round-ended tapered diamond bur, which was replaced after every four preparations to maintain cutting efficiency. A Shoulder finish line was meticulously crafted with a 0.01 mm shoulder bur, and convergence angles of 3° and 6° were achieved using two distinct tapered burs to optimize axial wall geometry. For the tooth with modified vertical preparation, a diamond fissure bur (Komet, Germany) All line angles were rounded to mitigate stress concentration and promote uniform stress distribution during load application. The specimens were examined using a putty index by using a periodontal probe after all sharp edges were rounded in the final phase.

Digital impressions were acquired utilizing a laboratory scanner (D2000, 3Shape, Copenhagen, Denmark), followed by virtual design in exocad software to create crowns with mesial and distal projections, thus facilitating controlled tensile force application during pull-off testing. The lithium disilicate (IPS E.max CAD) and zirconia-reinforced lithium silicate (VITA Suprinity) samples underwent crystallization in a programmable furnace (Vita Vacumat 6000MP), adhering to manufacturer-recommended thermal protocols to ensure optimal microstructural integrity. Meanwhile, resin nano-ceramic (Cerasmart) samples were fabricated through wet milling, leveraging their hybrid composition to obviate post-milling sintering.

Prior to cementation, intaglio surfaces were conditioned via air-abrasion and silanized with Monobond Plus, while tooth surfaces were etched with 35% phosphoric acid for 30 seconds, rinsed, and air-dried. Glass or

ceramics are etched with hydrofluoric acid (Ultraetch; Ultradent, Utah, USA) for 30 seconds, followed by a 30-second water spray. A universal adhesive was applied to both surfaces and intaglio surface, followed by dual-cure resin cementation (Variolink Esthetic DC, Ivoclar Vivadent), ensuring precise seating under controlled pressure. Excess cement was carefully removed, and polymerization was finalized through light curing.

All specimens underwent thermocycling for 5000 cycles between 5 °C and 55 °C, simulating approximately one year of oral thermal fluctuation. Subsequently, specimens were immersed for seven days at 37 °C in either artificial saliva or 2% citric acid solution (pH 3.20), the latter prepared by dissolving citric acid powder in distilled water per standardized laboratory protocols, thereby replicating intraoral acidic challenges common to modern dietary habits. The artificial saliva was meticulously formulated to include methyl paraben, potassium chloride, magnesium chloride, dextrose, sodium fluoride, and albumin, following validated recipes to simulate salivary biochemical milieu.

The retention strength was quantitatively assessed using a universal testing machine (Model 3345, Instron, USA), where tensile force was applied at a crosshead speed of 0.5 mm/min until debonding occurred. The forces required for dislodgment of the crowns were recorded in

N. Post-testing failure modes were critically evaluated under a digital microscope, categorizing failures as adhesive, cohesive, or mixed, thus providing insights into the nature of bond integrity.

Finally, statistical analysis was performed employing the Shapiro–Wilk test to examine normality assumptions. Given non-normal data distributions, non-parametric tests (Mann–Whitney and Kruskal–Wallis) were applied to detect significant differences between groups, with a significance level set at  $p \leq 0.01$ , thereby ensuring robust inferential conclusions.

## RESULT

The comparative evaluation of retention strength, as initially presented in Table 1, underscores a distinct and statistically significant enhancement across all material groups following citric acid exposure ( $p \leq 0.01$ ). Specifically, the resin nano-ceramic group demonstrated the highest mean retention value after acid immersion ( $149 \pm 5.96$ ), notably exceeding zirconia-reinforced lithium silicate ( $88.56 \pm 7.48$ ) and lithium disilicate ( $81 \pm 4.95$ ).

In addition to these findings, further detailed descriptive and inferential statistics on retention values for each material group before and after acid exposure are summarized below.

**Table 1: Descriptive statistics of retention values (N) for ceramic groups.**

Restorative Material	N	M $\pm$ SD	Min	Max
Emax control	5	$69.20 \pm 3$	65.00	73.00
Emax acid	5	$81 \pm 4.95$	77.00	86.83
Zirconia control	5	$58 \pm 6.88$	50.19	67.38
Zirconia acid	5	$88.56 \pm 7.48$	80.78	98.09
Resin control	5	$110.34 \pm 6.74$	98.08	116.79
Resin acid	5	$149 \pm 5.96$	143.00	157.85

Moreover, the Mann-Whitney analysis, summarized in Table 2, further confirms significant differences ( $p \leq 0.01$ ) in retention values between control and acid-treated subgroups for each material.

**Table 2: Mann-Whitney analysis of retention value for all groups.**

Restorative material	Condition	Mean $\pm$ SD	p-value
Emax	control	$69.2 \pm 3$	0.008
Emax	acid	$81 \pm 4.95$	-
Zirconia	control	$58 \pm 6.8$	0.008
Zirconia	acid	$88.56 \pm 7.48$	-
Resin	control	$110.34 \pm 6.74$	0.008
Resin	acid	$149 \pm 5.96$	-

Further comparative analysis among the materials, detailed in Table 3, demonstrates significant differences as evaluated by Kruskal-Wallis testing ( $p \leq 0.01$ ).

**Table 3: Kruskal-Wallis analysis of the impact of material on retention value.**

Restorative Material	Condition	M $\pm$ SD	Test value	p-value
Emax	control	$69.2 \pm 3$	12	0.002
Zirconia	control	$58 \pm 6.88$		
Resin	control	$110.34 \pm 6.74$		
Emax	acid	$81 \pm 4.95$	10	0.004
Zirconia	acid	$88.56 \pm 7.48$		
Resin	acid	$149 \pm 5.96$		

These additional integrated data from the pull-off tests align with the primary MPa-based retention findings, enriching the analytical narrative and providing comprehensive support for material-dependent behavior under acidic conditions.

## DISCUSSION

The integrative analysis of this study elucidates a compelling narrative wherein citric acid exerts a profound modulatory effect on the retention strength of CAD/CAM indirect restorative materials, with statistically significant enhancements observed across all tested groups. The remarkable increase in retention strength within the resin nano-ceramic group, as evidenced by its highest mean value ( $149 \pm 5.96$  N), can be mechanistically attributed to the synergistic interactions between the resin matrix (comprising Bis-GMA, UDMA, and DMA) and citric acid's carboxylic and hydroxyl groups. Such interactions likely promote additional hydrogen bonding and micro-mechanical interlocking at the adhesive interface, consequently augmenting overall retention (Hamilton et al., 2021).

On the contrary, the lithium disilicate group exhibited the lowest mean retention after acid immersion ( $81 \pm 4.95$  N), which may be explained by the susceptibility of its glassy matrix to acid-induced surface degradation. Citric acid, possessing multiple carboxylic moieties, interacts with the silanol groups on silica-rich surfaces, potentially compromising the microstructural integrity and diminishing silane-mediated bonding potential (Xu et al., 2024; Kropff, 2024).

This observation aligns with findings reported by Singh and Gulati (2022), who demonstrated decreased micro-hardness and increased surface vulnerability in lithium disilicate ceramics following acidic exposure.

Furthermore, zirconia-reinforced lithium silicate materials exhibited intermediate retention values ( $88.56 \pm 7.48$  N), suggesting a partial resistance to acidic degradation. This resilience could stem from the incorporation of zirconia, which enhances the material's crystalline stability and mitigates acid-mediated hydrolysis of silicate phases (Tardy et al., 2021; Harada et al., 2024). Silica in the material can still be affected by changes in acidity levels which may lead to adhesion after treatment due, to surface alterations that enhance mechanical bonding effectively.

The prevalence of failure patterns in lithium disilicate crowns following exposure to citric acid provides additional evidence, for the theory of weakened chemical bonding and surface deterioration (as suggested by Pilecco et al., 2023).

The resin nano ceramic group mainly showed mixed failures rather than individual breakages which suggests a strong bond at the interface that is stronger, than the material itself. This emphasizes how resin nano ceramic

systems combine elements to improve mechanical strength and adhesive properties by integrating fillers with the matrix and reducing brittleness (Liang et al., 2025).

Furthermore, the results highlight the role of taking into account the eating habits of patients when choosing materials. Regular intake of drinks and foods. Such as citrus fruits, carbonated beverages and specific vegetables. Can worsen the risk of restoration failure in materials susceptible, to acid related breakdown (Nito et al., 2023).

Resin nano ceramic materials seem to be beneficial for individuals with increased exposure to diet as they provide improved durability and biomechanical strength, over time.

The findings clearly emphasize the importance of clinicians embracing a strategy that considers the specific materials and individual needs when designing indirect dental restorations. The exceptional durability shown by resin nano crowns, under regular and acidic conditions not only confirms their inherent mechanical strengths but also supports their effectiveness in demanding oral situations.

## CONCLUSION

In summary of the research findings presented here convincingly shows that citric acid has an impact on the durability of different CAD/CAM dental materials in a way that varies with the specific material used. Resin nano ceramic displayed adhesive strength resilience compared to lithium disilicate which exhibited the highest vulnerability to acid induced deterioration. These results highlight the need for selecting materials tailored to individual patients needs especially for those with significant exposure to acidic diets. This is essential, for achieving lasting dental restoration and successful outcomes in clinical practice.

## Limitations

Despite the planning and precise execution of this study in a lab setting there are some limitations that need to be acknowledged critically here. In the controlled experimental conditions even though, meticulous care was taken to maintain them they may not fully capture the intricate and ever-changing environment inside the mouth where factors like saliva flow constantly changing enzymatic reactions variable pH levels and physical stresses all work together to impact how dental materials behave. Additionally, while having a sample size of 10 individuals, per group is statistically sound it might limit how broadly we can apply these findings to real world clinical situations. Another crucial factor to think about is the week of immersion lasting seven days which focuses mainly on mimicking short term acidic situations but may not fully reflect the long-term impacts of ongoing dietary acid exposure experienced over extended periods in real life settings. Unquestionably future research



studies with participant groups longer observation durations and real-life validation are vital to support and build upon the existing discoveries. This will help bridge the divide, between laboratory data and practical clinical applications.

### Clinical Recommendations

In light of the present study's compelling evidence demonstrating material-dependent variations in retention strength under acidic exposure, it becomes imperative for clinicians to adopt a patient-centered and diet-conscious approach when selecting indirect restorative materials. Specifically, the superior performance exhibited by resin nano-ceramic crowns in acidic conditions underscores their potential as a preferred choice for individuals with high dietary acid intake, such as those frequently consuming citrus fruits, carbonated beverages, or acidic snacks. Furthermore, clinicians should emphasize patient education regarding the detrimental effects of prolonged acidic exposures on dental restorations, thus encouraging dietary modifications and reinforcing preventive strategies. Integrating such personalized material selection with comprehensive dietary counseling not only optimizes the longevity and biomechanical integrity of restorations but also enhances overall patient satisfaction and long-term oral health outcomes.

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