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Solubility of Zirconia-Based Ceramic Composite Resin After Immersion in Lemon Juice (*Citrus limon* (L.) Burm.)

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ABSTRACT

Zirconia-based ceramic composite resin is a restorative material with high mechanical strength and aesthetic properties, but its solubility in acidic environments remains a concern. Acidic beverages, such as lemon juice (*Citrus limon* (L.) Burm.), contained citric and ascorbic acids, which could compromise stability in the oral cavity. Therefore, this study aimed to evaluate solubility of zirconia-based ceramic composite resin after immersion in lemon juice. This laboratory experimental study used a post-test only control group design with 24 zirconia-based ceramic composite resin discs (2 mm thick, 8 mm diameter). Samples were divided into three groups, namely control (immersed in distilled water for 24 hours), as well as immersion in lemon juice for 12 hours and 24 hours. Solubility was measured by weight difference before and after immersion using an analytical balance and calculated with Oysaed & Ruyter's formula. The collected data were analyzed using the Shapiro–Wilk and Levene tests, followed by Kruskal–Wallis and Mann–Whitney tests. The mean solubility was 0 µg/mm³ in the control group, 0.00018 µg/mm³ in the 12-hour lemon juice group, and 0.00052 µg/mm³ in the 24-hour lemon juice group. Statistical analysis showed significant differences among groups, as evidenced by $p < 0.05$. In conclusion, immersion in lemon juice increased solubility of zirconia-based ceramic composite resin. Solubility values were higher with longer immersion time.

Keywords: Solubility of Material, Zirconia-Based Composite Resin, Lemon Juice, *Citrus Limon*, Acidic Beverages

ABSTRAK

Resin komposit ceramic berbasis zirkonia memiliki kekuatan mekanis tinggi dan sifat estetik baik, namun rentan larut pada lingkungan asam, termasuk jus jeruk lemon (Citrus limon (L.) Burm.) yang mengandung asam sitrat dan askorbat. Penelitian ini bertujuan mengukur kelarutan resin komposit ceramic berbasis zirkonia setelah perendaman dalam jus jeruk lemon.Metode: Studi eksperimental laboratoris post-test only control group menggunakan 24 spesimen cakram resin komposit ceramic berbasis zirkonia. Spesimen dibagi menjadi tiga kelompok: kontrol (akuades 24 jam), jus lemon 12 jam, dan jus lemon 24 jam. Kelarutan dihitung dari selisih berat sebelum–sesudah perendaman dan dianalisis dengan uji Kruskal–Wallis dan Mann–Whitney.Hasil: Rata-rata kelarutan 0 µg/mm³ (kontrol), 0,00018 µg/mm³ (12 jam), dan 0,00052 µg/mm³ (24 jam), dengan perbedaan signifikan antar kelompok ($p < 0,05$). Simpulan: Perendaman jus jeruk lemon meningkatkan kelarutan resin komposit ceramic berbasis zirkonia, seiring lamanya waktu perendaman.

Kata kunci: Kelarutan, Resin Komposit Berbasis Zirkonia, Jus Jeruk Lemon, *Citrus Limon*, Minuman Asam

1. Introduction

Dental restorative materials are evolving rapidly in response to the high demands for aesthetics, mechanical performance, and biocompatibility. Conventional resin composites are widely used due to the natural tooth-like appearance and conservative preparation. Recent advances have introduced zirconia-reinforced or zirconia-based ceramic composite resins, where zirconia nanoparticles or zirconia–silica hybrid fillers are incorporated into resin matrix to enhance mechanical and optical performance. The results of recent studies have shown that the addition of zirconia-based nanofillers significantly improves flexural strength, fracture toughness, wear resistance, color stability, and long-term aging behavior, compared with traditional nano-hybrid composites [1-3]. These enhancements are attributed to the high modulus and crack-blunting effect of zirconia particles, which increase resistance to crack propagation and mechanical fatigue. However, zirconia-reinforced composites still share the inherent limitations of resin-matrix systems, particularly water sorption and solubility. This limitation comprises long-term stability in fluctuating pH and moisture of the oral environment [4-5].

The oral cavity is frequently exposed to various dietary substances, including acidic beverages, such as fruit juices, carbonated, and energy drinks. In particular, lemon juice (*Citrus limon* (L.) Burm.) contains high levels of citric and ascorbic acids, which alters chemical and physical stability of dental materials [6]. The erosive potential of these acids compromises the structural integrity of resin composites by enhancing water sorption and increasing solubility. Several studies have shown that exposure to acidic solutions increases hydrolytic degradation of resin matrices and disrupts filler–matrix bonding, thereby reducing the longevity in the oral cavity [7]. Understanding the interaction between restorative materials and dietary acids is crucial in predicting clinical performance and guiding material selection in daily dental practice.

Solubility is a key parameter that reflects chemical stability of dental restorative materials. According to ISO 4049 standards, low solubility values are essential to ensure that restorative materials remain dimensionally stable and resistant to degradation over time [8]. Excessive solubility compromises the mechanical strength of the material and also releases unreacted monomers and filler components, which could irritate pulpal or gingival tissues. A previous study reported that acidic environments increased solubility of various composite resins, but data on zirconia-based ceramic composites remained limited [9]. Therefore, there is a need to examine the behavior in acidic conditions, such as immersion in lemon juice, to assess long-term durability.

Citrus-based beverages, such as lemon juice, are among the widely consumed fruit juices worldwide and have been recognized for both nutritional significance and potential erosive properties [10]. The erosive potential has been studied primarily in relation to dental hard tissues, where it was shown to demineralize enamel and dentin [11]. However, only a few studies have explored its effects on restorative materials, particularly advanced composites, such as zirconia-based ceramic resins. Considering the high clinical use of zirconia-based composites, there is a need to evaluate whether solubility is influenced by lemon juice exposure and the contribution of immersion time to this effect. Based on the considerations, this study was designed to measure solubility of zirconia-based ceramic composite resin after immersion in lemon juice for different periods of time. The results are expected to provide valuable insight into the stability of this material under acidic conditions and support evidence-based recommendations for restorative material selection, particularly in patients with high consumption of acidic beverages.

2. Materials and Methods

This study was a genuine experimental laboratory investigation with a post-test only control group design. A total of 24 disc-shaped specimens of zirconia-based ceramic composite resin were prepared as the samples. Each specimen was standardized to 2 mm and 8mm in thickness and diameter, respectively. A round and hollow standless steel form was used for manufacturing, then light-cured for 40 seconds on each surface, following ISO 4049 standards for resin-based materials [8]. After curing, the specimens were carefully removed, finished with fine abrasives for uniformity, and placed in a desiccator for 24 hours before testing to ensure complete polymerization.

All samples were weighed using an expository adjust with 0.0001 g precision to obtain the starting dry weight. The volume of each sample was calculated using the equation $V = \pi r^2 h$, and solubility values were normalized to specimen volume. Handling was carried out with gloves and tweezers to minimize contamination. The samples were haphazardly doled out into three bunches (n = 8 each). Group I served as the

negative control and was submerged in 20 mL of refined water for 24 hours. Group II was immersed in 20 mL of freshly prepared lemon juice for 12 hours, while III was immersed in 20 mL of lemon juice for 24 hours. Lemon juice was obtained by manually squeezing fresh ripe lemons, filtered through a fine mesh sieve to ensure the removal of pulp and seeds. The juice was used immediately without dilution to preserve its natural acidity, consistent with previous studies examining citrus juice as an erosive medium [11,12]. All immersions were carried out in sealed glass containers at room temperature, ensuring complete submersion of specimens.

After drying, the samples were washed with refined water, blotted with absorbent paper, and placed in a desiccator for 24 hours to obtain a constant mass. Final weights were recorded with the same analytical balance. Solubility was calculated as the difference between initial and final weight, normalized to specimen volume, and expressed in μg/mm³ using the Oysaed and Ruyter formula [13].

Data analysis was carried out using IBM SPSS version 25. Normality was assessed using the Shapiro–Wilk test, and homogeneity of variance was evaluated with Levene's test. Non-parametric tests were conducted for data that violated both normality and homogeneity assumptions. The Kruskal–Wallis test was used to examine the differences among groups, followed by Mann–Whitney pairwise comparisons, and statistical significance was set at $p < 0.05$.

3. Results

The mean solubility values of zirconia-based ceramic composite resin after immersion in different solutions are shown in Table 1. The control group, which was immersed in distilled water for 24 hours, showed no measurable solubility (0 μg/mm³). This result was consistent with the expectation that distilled water, as a neutral medium, had minimal erosive effect on restorative materials. However, both experimental groups immersed in lemon juice exhibited measurable solubility. After 12 and 24 hours of immersion, the mean solubility was 0.00018 μg/mm³ and 0.00052 μg/mm³, respectively. These results showed that immersion in lemon juice led to measurable dissolution of zirconia-based ceramic composite resin, with solubility increasing alongside exposure time.

Shapiro–Wilk testing showed non-normal data distribution, and Levene's test suggested heterogeneity of variance, necessitating non-parametric methods. The Kruskal–Wallis analysis showed statistically significant differences among the groups ($p < 0.05$), suggesting that both immersion medium and exposure duration exerted a significant effect on solubility of zirconia-based ceramic composite resin. Further pairwise analysis using the Mann–Whitney test showed significant differences between distilled water and 12-hour lemon juice immersion. There was also a significant difference between distilled water and 24-hour immersion, as evidenced by $p < 0.05$. A significant difference was also observed between the 12-hour and 24-hour immersion groups, confirming that prolonged exposure increased solubility. These results were supported by the progressive numerical increase in solubility values. Although solubility after 12 hours of immersion was relatively low, the value was approximately tripled after 24 hours. This trend suggested a time-dependent dissolution process, in which prolonged exposure to citric and ascorbic acids in lemon juice increased degradation of resin matrix and filler components. Despite the small magnitude of the absolute solubility values, even minor solubility over time could compromise the dimensional stability and mechanical strength of restorations in clinical conditions.

In general, the result showed the vulnerability of zirconia-based ceramic composite resin to acidic challenges in the oral environment. Furthermore, there was a significant increase in solubility following lemon juice immersion, and a time-dependent pattern in which longer exposure increased the degradation process. These results showed the clinical importance of dietary acids, particularly the frequent consumption of citrus beverages rich in citric and ascorbic acids, in influencing the longevity of resin-based restorative materials.

Table 1. Mean solubility of zirconia-based ceramic composite resin

Group	Immersion Medium	Duration	Mean Solubility (μg/mm³)	*p*-value
Control	Distilled water	24 h	0.00000	< 0.05
Test 1	Lemon juice (*C. limon*)	12 h	0.00018	< 0.05
Test 2	Lemon juice (*C. limon*)	24 h	0.00052	< 0.05

4. Discussion

The results of this study show that immersion of zirconia-based ceramic composite resin in lemon juice significantly increased its solubility compared to specimens immersed in distilled water. The control group, immersed in distilled water for 24 hours, had no measurable solubility. Meanwhile, samples immersed in lemon juice for 12 and 24 hours showed progressively higher solubility values. These results suggest that the acidic components of lemon juice, mainly citric and ascorbic acids, play a significant role in degrading chemical stability of resin composites. Although the numerical values appear small, repeated or prolonged exposure in the oral cavity may cumulatively weaken restorative materials and shorten the clinical longevity [9].

The significant differences observed in both the Kruskal–Wallis and Mann–Whitney tests confirmed that immersion medium and immersion time are critical factors affecting solubility. Acidic solutions have been shown to promote hydrolytic degradation of resin matrix, leading to polymer chain scission and leaching of unreacted monomers [14,15]. This process explains the increased solubility observed after longer immersion periods. Previous studies reported similar results in composite resins exposed to cola drinks, fruit juices, and other acidic beverages, where prolonged exposure led to greater weight loss and compromised surface hardness [9,7]. The results of this study add to the body of evidence by confirming that lemon juice, as a naturally acidic beverage, exerts comparable effects on advanced restorative materials.

The mechanism of citric and ascorbic acids in lemon juice is highly relevant. Citric acid acts as a chelating agent, binding to metal ions in the filler particles, thereby weakening the filler–matrix interface and facilitating particle loss [16]. Both citric and ascorbic acids reduce the environmental PH, enhancing resin matrix water sorption and hydrolysis. This procedure leads to softening and dissolution of the polymer network [17]. In natural teeth, these acids demineralize hydroxyapatite crystals in enamel and dentin, causing erosion and loss of mineral content [18]. Therefore, the same acidic challenge affects the restorative material and compromises the surrounding tooth structure, leading to marginal breakdown and reduced clinical performance of restorations.

The duration of acid exposure plays a significant role, as short -term exposure may cause surface softening or slight solubility. However, prolonged or repeated contact, as simulated in the 24-hour immersion group, worsens the degradation process [11,12]. Longer immersion provides acids more time to penetrate resin matrix, disrupt cross-linked polymer chains, and accelerate leaching of unreacted monomers [19]. In teeth, extended acid contact increases enamel erosion depth and dentin demineralization, which can be compounded by mechanical forces, such as toothbrushing [20]. This time-dependent effect explains why the 24-hour immersion group showed approximately three times greater solubility than the 12-hour.

A previous study showed the detrimental role of acidic beverages in compromising dental restorative materials. Aliping-McKenzie *et al.* reported that fruit juices significantly reduced the surface hardness of glass-ionomers and compomers [9]. Barac *et al*. also reported that soft drinks caused erosive effects on nanohybrid composite resins, increasing both surface roughness and weight loss [18]. According to Erdemir *et al.*, energy drinks significantly altered the color stability of nanohybrid composites, further confirming the broad impact of acidic beverages on restorative materials [19]. The results were consistent with this present study, strengthening the conclusion that dietary acids were important external factors influencing the durability of restorations.

Recent studies have shown that zirconia-reinforced or zirconia-filled composite resins are susceptible to chemical degradation when exposed to acidic environments. Acid-induced hydrolytic breakdown of resin matrix and weakening of the filler–matrix interface have been reported, accompanied by reductions in mechanical performance and structural stability [20,21]. Aging studies on nano-zirconia–containing composites have shown additional deterioration under acidic or low-pH conditions, confirming that zirconia-modified systems cannot fully resist erosive or chemical challenges [1]. In general, these results show that while zirconia-based composites may have superior physical properties under neutral conditions, the stability is still compromised in acidic environments such as lemon juice.

Based on a clinical perspective, solubility of restorative materials is directly related to long-term performance. Materials with higher solubility tend to undergo dimensional changes, marginal breakdown, and reduced mechanical strength [8, 21]. Leaching of unreacted monomers and fillers also leads to adverse biological consequences, including pulpal or gingival irritation [22]. Therefore, understanding how commonly consumed acidic beverages affect solubility is important for material selection and patient education.

Several epidemiological studies have shown the increasing consumption of acidic beverages, including soft drinks, citrus juices, and energy drinks, particularly among adolescents and young adults [23]. This lifestyle trend has been associated with extrinsic staining of restorations and also dental erosion, which affects both natural tooth structure and restorative materials [24]. Patients with high intake of acidic beverages present a significant risk factor for restoration failure due to high degradation processes. Therefore, evaluating solubility of zirconia-based composites in acidic conditions is important for predicting the long-term clinical performance.

The impact of acidic beverages extends beyond solubility. Frequent exposure to acids contributes to surface softening, microcrack formation, and increased surface roughness in both enamel and restorative materials [25]. These alterations can worsen solubility and weaken the marginal integrity of restorations. The result of this study confirms chemical vulnerability of zirconia-based ceramic composites and also shows the broader clinical implications of dietary acids. Preventive strategies in clinical practice, including patient counseling to limit acidic beverage consumption, should be prioritized to improve the longevity of resin-based restorations.

5. Conclusion

In conclusion, solubility of zirconia-based ceramic composite resin after immersion in lemon juice increases significantly compared to distilled water. Longer immersion duration also increases solubility values, confirming a time-dependent effect of acidic exposure. Citric and ascorbic acids in lemon juice are responsible for the degradation process, compromising chemical stability of zirconia-based composites.

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7. Conflict of Interest

The authors declare no conflict of interest.

References

Zheng L, Zhang Y, Bai Y, Zhang Z, Wu Q. Study on the mechanical and aging properties of an antibacterial composite resin loaded with fluoride-doped nano-zirconia fillers. Front Bioeng Biotechnol 2024; 12: 1397459.

Yu X, Yang S, Han D, Wang T, Xie Q, Xie W. A novel dental resin composite based on POSS and MDP-modified zirconia: Preparation and evaluation. J Dent 2025; 155: 105638.

Cho K. Dental resin composites: A review on materials to product realizations. Compos B Eng 2022; 230: 109495.

Mitra SB, Wu D, Holmes BN. An application of nanotechnology in advanced dental materials. J Am Dent Assoc 2003; 134(10): 1382–1390.

Ferracane JL. Resin composite—State of the art. Dent Mater 2011; 27(1): 29–38.

Zero DT, Lussi A. Erosion—chemical and biological factors of importance to the dental practitioner. Int Dent

J 2005; 55(4 Suppl 1): 285–290.
Yap AU, Wattanapayungkul P. Effects of in-office tooth whitening agents on resin composites: a review. Oper Dent 2002; 27(2): 137–141..
International Organization for Standardization. Dentistry — Polymer-based restorative materials. ISO 4049. Geneva: ISO; 2009.
Aliping-McKenzie M, Linden RW, Nicholson JW. The effect of Coca-Cola and fruit juices on the surface hardness of glass-ionomers and compomers. J Oral Rehabil 2004; 31(11): 1046–1052.
Karki N, Achhami H, Pachhai BB, Bhattarai S, Shahi DK, Bhatt LR, et al. Evaluating citrus juice: A comparative study of nutritional quality and health benefits. Foods 2024; 13(6): 11648903.
Gradinaru I, Vasiliu AL, Bargan A, Checherita LE, Ciubotaru BI, Armencia AO, et al. The influence of beverages on resin composites: an in vitro study. Biomedicines 2023; 11(9): 2571.
Yerliyurt K, Sarıkaya I. Color stability of hybrid ceramics exposed to beverages in different combinations. BMC Oral Health 2022; 22(1).
Berger SB, Palioto ARM, Cavalli V, Giannini M. Characterization of water sorption, solubility and filler particles of light-cured composite resins. Braz Dent J 2009; 20(4): 314–318.
Hampe T, Wiessner A, Frauendorf H, Alhussein M, Karlovsky P, Bürgers R, et al. Hydrolytic degradation and monomer release of dental resin composites in acidic conditions. Polymers (Basel) 2022; 14(9): 1790.
Ilie N. Degradation of dental methacrylate-based composites in simulated clinical immersion media. J Funct Biomater 2022; 13(1): 25.
Peutzfeldt A. Resin composites in dentistry: the monomer systems. Eur J Oral Sci 1997; 105(2): 97–116.
Ren YF, Malmstrom H, Eliav E. Dental erosion: etiology, diagnosis and prevention. Compend Contin Educ Dent 2011; 32(9): 46–52.
Reddy PS, Tejaswi KLS, Shetty S. Effects of Commonly Consumed Beverages on Surface Roughness and Color Stability of the Nano, Microhybrid and Hybrid Composite Resins: An in vitro Study. J Contemp Dent Pract 2013; 14(4): 718–23.
Erdemir U, Yildiz E, Eren MM, Ozel S, Topcu FT, Tokar E. Effects of energy drinks on the color stability of nanohybrid composites. Eur J Dent 2017; 11(3): 347–351.
Geha O, Inagaki LT, Favaro JC, González AHM, Guiraldo RD, et al. Effect of chemical challenges on the properties of composite resins. Int J Dent 2021; 2021(1): 4895846.
Hampe T, Wiessner A, Frauendorf H, Alhussein M, Karlovsky P, Bürgers R, et al. Monomer release from dental resins: the current status on study setup, detection and quantification for in vitro testing. Polymers (Basel) 2022; 14(9): 1790.
Schweikl H, Spagnuolo G, Schmalz G. Genetic and cellular toxicology of dental resin monomers. J Dent Res 2006; 85(10): 870–7.
Tahmassebi JF, Duggal MS, Malik-Kotru G, Curzon ME. Soft drinks and dental health: a review of the current literature. J Dent 2006; 34(1): 2–11.
Hara AT, Zero DT. The potential of saliva in protecting against dental erosion. Monogr Oral Sci 2014; 25: 197–205.
Ganss C, Schlueter N, Hardt M, von Hinckeldey J, Klimek J. Effects of toothbrushing on eroded dentine. Eur J Oral Sci 2007; 115(5): 390–6.