DIFFERENCES IN MICROHARDNESS OF MICROHYBRID COMPOSITE RESIN AFTER EXPOSURE TO ALCOHOL-BASED, ALCOHOL-FREE, AND HERBAL MOUTHWASHES: AN IN VITRO STUDY

(PERBEDAAN TINGKAT KEKERASAN MIKRO RESIN KOMPOSIT MIKROHIBRID PADA PENGGUNAAN OBAT KUMUR DENGAN DAN TANPA KANDUNGAN ALKOHOL DAN OBAT KUMUR HERBAL SECARA IN VITRO)

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ABSTRACT

Composite resin is one of the most widely used restorative materials in dentistry due to its aesthetic properties, mechanical strength, and ease of manipulation. However, the long-term mechanical stability of composite resins can be affected by chemical exposure, such as from mouthwash solutions used in daily oral hygiene routines. This study aimed to determine the difference in microhardness of microhybrid composite resin after immersion in mouthwash containing alcohol, alcohol-free mouthwash, and herbal mouthwash. This was an experimental laboratory study with a pre-test and post-test group design. A total of 30 samples of microhybrid composite resin (Solare X) were divided into three groups, each immersed for 48 hours at 36°C in Listerine Multi Protect Zero (alcohol-containing), MeToo Mouthwash Probiotic (alcohol-free), and Amodent Gargle with Cardamom Essential Oil (herbal). The microhardness values were measured using a Vickers Hardness Tester, and the data were analyzed using One Way ANOVA followed by Post Hoc Bonferroni tests. The results showed a significant increase in surface hardness after immersion in all groups, with the highest mean value in the alcohol-containing mouthwash group (32.85 VHN), followed by the herbal mouthwash (29.23 VHN), and the alcohol-free mouthwash (24.05 VHN) ($p < 0.05$). It can be concluded that all types of mouthwash have a significant effect on the surface hardness of microhybrid composite resin. The increase in hardness is likely due to the high filler content, strong silane coupling, and post-cure polymerization process occurring during immersion.

Keywords: microhybrid composite resin; microhardness; mouthwash; Vickers Hardness Tester

ABSTRAK

Resin komposit merupakan bahan restorasi yang banyak digunakan dalam kedokteran gigi karena memiliki sifat estetik, kekuatan mekanik yang baik, serta mudah diaplikasikan. Salah satu faktor yang dapat memengaruhi kekuatan mekanik resin komposit adalah paparan bahan kimia dari obat kumur yang digunakan secara rutin dalam menjaga kebersihan rongga mulut. Penelitian ini bertujuan untuk mengetahui perbedaan tingkat kekerasan mikro resin komposit mikrohibrid setelah dilakukan perendaman dalam obat kumur dengan kandungan alkohol, tanpa kandungan alkohol, dan obat kumur herbal. Penelitian ini menggunakan metode eksperimental laboratoris dengan rancangan pre-test dan post-test group design. Sebanyak 30 sampel resin komposit mikrohibrid (Solare X) dibagi menjadi tiga kelompok, masing-masing direndam selama 48 jam pada suhu 36°C dalam larutan Listerine Multi Protect Zero (alkohol), MeToo Mouthwash Probiotic (tanpa alkohol), dan Amodent Gargle Minyak Atsiri Buah Kapulaga (herbal). Nilai kekerasan mikro diukur menggunakan Vickers Hardness Tester, kemudian dianalisis dengan uji One Way ANOVA dan Post Hoc Bonferroni. Hasil penelitian menunjukkan adanya peningkatan nilai kekerasan yang signifikan pada seluruh kelompok perlakuan setelah perendaman, dengan nilai rata-rata tertinggi pada kelompok obat kumur kandungan alkohol (32,85 VHN), diikuti oleh obat kumur herbal (29,23 VHN), dan tanpa alkohol (24,05 VHN) ($p < 0,05$). Dapat disimpulkan bahwa ketiga jenis obat kumur memengaruhi kekerasan resin komposit mikrohibrid secara bermakna. Peningkatan kekerasan ini diduga disebabkan oleh kandungan filler tinggi, ikatan silane yang kuat, serta proses post-cure polymerization yang terjadi selama perendaman.

***Kata kunci**: resin komposit mikrohibrid; kekerasan mikro; obat kumur; Vickers Hardness Tester*

INTRODUCTION

Dental caries is a progressive oral disease characterized by enamel demineralization that extends into dentin, resulting from continuous interaction among host factors, microorganisms, substrate, and time[1]. Its development is influenced by poor dietary habits, especially the frequent consumption of sticky sugary foods, inadequate toothbrushing techniques, and irregular dental visits[2]. The presence of sucrose on tooth surfaces facilitates bacterial fermentation into lactic acid, lowering the pH to a critical threshold and initiating enamel demineralization[3]. Given the increasing prevalence of caries in Indonesia—reported at 45.3% with a national DMF-T index of 7.1—effective preventive and restorative strategies remain essential[4].

Restorative treatment is often required to prevent further progression of caries and to re-establish masticatory and esthetic functions[5]. Among the available restorative materials, composite resin is widely selected due to its esthetics, minimal invasiveness, and ability to bond to tooth structure[6]. Hybrid composite resins, including microhybrid formulations, are frequently used because their mixed filler compositions provide enhanced strength, low polymerization shrinkage, good polishability, and resistance to abrasion[7,8].

Alongside routine oral hygiene practices such as brushing, flossing, and proper diet, the use of mouthwash has increased substantially in both professional and community settings[9]. Mouthrinses vary in formulation—alcohol-based, alcohol-free, and herbal—and contain antiseptic agents such as alcohol, essential oils, phenols, and astringents[10,11]. Although beneficial for reducing microbial load, the chemical components of mouthwash may interact with restorative materials and influence their physical properties. Prior studies have demonstrated that alcohol-containing mouthwash can reduce the microhardness of nanohybrid and nanofiller composites more significantly than alcohol-free formulations[12,13]. These findings suggest that the response of composite resins may depend on filler structure and matrix composition, highlighting the importance of evaluating microhybrid composites separately.

Despite the widespread use of microhybrid composite resin in clinical practice, evidence regarding its microhardness stability following exposure to various mouthwash formulations remains limited. Understanding these interactions is crucial for minimizing restoration failure and guiding clinicians in selecting appropriate adjunctive oral hygiene products.

Therefore, this in vitro study aims to compare the microhardness of microhybrid composite resin after immersion in alcohol-based, alcohol-free, and herbal mouthwashes. The hypothesis tested is that there are significant differences in microhardness among the three treatment groups.

METHOD

This laboratory-based experimental study employed a pre-test and post-test group design to evaluate changes in the microhardness of microhybrid resin composite after immersion in three different types of mouthwash. The research was conducted at the Laboratorium Teknik Mesin Universitas Sumatera Utara, between April and July 2025. The samples consisted of commercially available microhybrid resin composite (Solare X), prepared in cylindrical molds measuring 8 mm in diameter and 2 mm in thickness. Sample size was calculated using the Federer formula, resulting in 10 samples per group and a total of 30 samples. The samples were allocated into three treatment groups: immersion in alcohol-containing mouthwash (Listerine Multi Protect Zero), alcohol-free mouthwash (MeToo Mouthwash Probiotic), and herbal mouthwash (Amodent Gargle Minyak Atsiri Buah Kapulaga). Inclusion criteria consisted of new, uncontaminated microhybrid resin composite and commercially available mouthwashes with official distribution approval, along with eligibility for microhardness measurement using a Vickers Hardness Tester (VHT). Exclusion criteria included resin composites other than microhybrid type, previously contaminated samples, mouthwashes without distribution approval, and measurements performed with uncalibrated instruments.

Sample preparation followed the technique described by Permatasari[1] with relevant modifications. Acrylic molds were placed on a glass slab lined with a celluloid strip, and the internal mold surface was coated with vaseline. The resin composite was inserted into the mold using a plastic filling instrument, covered again with a celluloid strip, and compressed with a glass slab to obtain a flat surface. After lifting the upper slab, specimens were light-cured for 20 seconds at approximately 2 mm distance in a perpendicular orientation, followed by curing on the opposite surface using the same protocol. Baseline microhardness values were obtained using a Vickers Hardness Tester with a 100-gram load applied for 20 seconds, and the average of three indentations was recorded for each specimen. Thereafter, samples were immersed in their respective mouthwash solutions for 48 hours at room temperature (approximately 36°C). Upon completion of immersion, samples were rinsed with distilled water, dried, and re-evaluated for post-treatment microhardness using the same Vickers testing procedure.

Statistical analysis began with assessment of data normality using the Shapiro–Wilk test, followed by a homogeneity of variance test. Differences among the three treatment groups were analyzed using one-way ANOVA. When statistically significant differences were detected ($p < 0.05$), post hoc Bonferroni testing was performed to identify specific intergroup differences. All statistical analyses were conducted using IBM SPSS software.

RESULT

The baseline microhardness values showed slight variability among groups, which may be attributed to minor differences during sample fabrication that affect initial surface uniformity. Following 48 hours of immersion, all groups exhibited an increase in microhardness, although the magnitude of change differed according to the type of mouthwash. These variations reflect the distinct chemical interactions between each mouthwash formulation and the composite resin matrix.

The alcohol-containing mouthwash produced the greatest increase in microhardness, followed by the herbal mouthwash, whereas the alcohol-free formulation resulted in the smallest increase. This trend suggests that the chemical composition and solvent characteristics of each mouthwash contribute differently to the modification of the composite resin surface. The combined pre- and post-immersion microhardness values are summarized in Table 1.

Table 1. Composite Resin Hardness Test Results Using Vickers Hardness Tester

No		Before	After
1	Alcohol-based	19.34	32.85
2	Alcohol-free	19.16	24.05
3	Herbal	21.48	29.23

After identifying the data to be analyzed, the Shapiro-Wilk test was performed to test for normality. The analysis showed that all significance values for each group, both before and after immersion, exceeded the threshold of 0.05. This indicates that the data were normally distributed across the alcohol-based, alcohol-free, and herbal mouthwash groups.

Accordingly, the dataset met the statistical assumptions required to proceed with subsequent parametric analyses. These findings also suggest that the different immersion solutions did not produce extreme distributional variations that could interfere with inferential testing in the following stages. These findings are presented in detail in Table 2, which summarizes the Shapiro–Wilk normality test results for all treatment groups.

Table 2. Shapiro-Wilk Test Results

No		Before	After
1	Alcohol-based	0.945	0.764
2	Alcohol-free	0.071	0.480
3	Herbal	0.306	0.619

Before conducting the One-Way ANOVA, Levene's test was used to determine the homogeneity of variances across groups. The analysis indicated that both pre-immersion and post-immersion measurements exhibited non-significant Levene's values ($p > 0.05$), confirming that the variances among the three treatment groups were homogeneous. Combined with the previously established normality, the data met the assumptions required for parametric testing.

Subsequent One-Way ANOVA revealed statistically significant differences in the mean microhardness values among the three mouthwash groups, both before and after immersion. The pre-immersion analysis demonstrated a significant overall difference ($p < 0.05$), indicating baseline variations in microhardness among the groups. Following immersion, the significant difference became more pronounced, suggesting that the type of mouthwash solution—alcohol-based, alcohol-free, or herbal—had a meaningful effect on the microhardness of the microhybrid resin composite.

These findings highlight that immersion in different mouthwash formulations influences the mechanical behavior of the resin composite, supporting the hypothesis that the chemical composition of the solution contributes to changes in material microhardness.

The One-Way ANOVA indicated significant baseline differences in microhardness among the treatment groups, prompting a Bonferroni Post Hoc analysis. The pairwise comparisons demonstrated that only specific groups differed significantly, suggesting that each mouthwash formulation influenced the initial microhardness distinctly. Detailed results are presented in **Table 3**.

Table 3. Bonferroni Post Hoc Test Results Before Treatment

Mouthwash	Comparison	P-value
Alcohol-based	Alcohol-free	1.000
	Herbal	0.041
Alcohol-free	Alcohol-based	1.000
	Herbal	0.009
Herbal	Alcohol-based	0.041
	Alcohol-free	0.009

Based on the post hoc analysis, the alcohol-containing and alcohol-free mouthwash groups showed no significant difference (p = 1.000), indicating comparable baseline microhardness before immersion. In contrast, comparisons involving the herbal mouthwash group revealed significant differences, with the alcohol-containing vs. herbal comparison (p = 0.041) and the alcohol-free vs. herbal comparison (p = 0.009) both demonstrating statistically meaningful disparities. These variations may be attributed to minor inconsistencies during sample preparation, such as differences in molding pressure, material homogeneity, or variations in polymerization intensity.

Post-immersion analysis revealed clear statistical differences in microhardness among all treatment groups. The Bonferroni Post Hoc test indicated that each pairwise comparison demonstrated significant variation, confirming that the chemical characteristics of the alcohol-containing, alcohol-free, and herbal mouthwashes produced distinct effects on the microhardness of the microhybrid resin composite. These findings indicate that immersion in different mouthwash formulations alters the material's surface properties in a significantly different manner. Detailed pairwise outcomes are presented in **Table 4**.

Table 4. Bonferroni Post Hoc Test Results After Treatment

Mouthwash	Comparison	P-value
Alcohol-based	Alcohol-free	<0.001
	Herbal	0.002
Alcohol-free	Alcohol-based	<0.001
	Herbal	<0.001
Herbal	Alcohol-based	0.002
	Alcohol-free	<0.001

DISCUSSION

The key finding of this investigation confirms the hypothesis that all three types of commercial mouthwash—alcoholic, non-alcoholic, and herbal—

significantly influence the microhardness of the microhybrid composite resin (*Solare X*). Uniquely, the results consistently demonstrated a significant increase in the surface microhardness across all tested groups after immersion. This outcome contrasts sharply with previous literature concerning the impact of mouthwash on resin composites, particularly studies involving *nanohybrid* or *nanofiller* materials, which frequently report a decrease in hardness due-to solvent-induced degradation and subsequent *filler* washout. The novelty of this work lies in highlighting the superior stability of the microhybrid composite, thereby addressing current challenges related to material degradation in the oral environment. This increase in microhardness is primarily ascribed to the material's composition: the *Solare X* microhybrid composite possesses a high content of inorganic *filler* (60-70%), providing enhanced mechanical stability and resistance to both chemical dissolution and fluid penetration. This dense internal structure effectively limits the fluid uptake and matrix degradation that is more common in composites with a lower *filler*-to-resin ratio, such as *nanohybrids* or *nanofillers*.

Furthermore, the observed increase is attributed to post-cure polymerization, where the degree of monomer conversion continues to advance during the 48-hour immersion period at room temperature. The low concentration of alcohol in the mouthwash, for instance, may temporarily soften the surface, but this condition paradoxically facilitates the formation of new polymer *cross-links* upon drying, resulting in a net increase in matrix density and a higher microhardness value, a phenomenon further supported by the high *filler* content that restricts excessive polymer chain movement. Similarly, the hydrophobic essential oil compounds (e.g., 1,8-cineole, limonene) found in the herbal mouthwash are proposed to interact with the hydrophobic resin surface, reducing water penetration and minimizing matrix degradation, thus contributing to the sustained or increased hardness. Ultimately, the divergent findings underscore that the material response is highly dependent on the type and composition of the *filler* and the degree of polymer conversion.[12-15] These results carry crucial clinical implications, suggesting that dentists must carefully consider the specific composite system selected for restorations, as materials like microhybrid composites exhibit enhanced stability or *post-cure* effects, while others may be more prone to surface degradation when exposed to common oral hygiene agents.

CONCLUSION

Based on the analysis of the research findings, it is concluded that significant differences exist in the surface microhardness of the microhybrid composite resin (*Solare X*) following 48-hour immersion in three distinct types of mouthwash: alcoholic (*Listerine Multi Protect Zero*), non-alcoholic (*MeToo Mouthwash Probiotic*), and herbal (*Amodent Gargle* Minyak Atsiri Buah Kapulaga). The study successfully answered its primary hypothesis by demonstrating that all immersion groups exhibited an increase in surface microhardness after the treatment period. This observed increase is attributed to two key factors: first, the inherent material characteristics of the microhybrid composite, specifically its high content of inorganic *filler* and the strong bonding between the *filler* and the resin matrix via the *silane coupling agent*.

This composition collectively enhances the material's stability, providing superior resistance to fluid penetration and chemical degradation, contrasting with the degradation often reported in other composite types. Second, the phenomenon of post-cure polymerization is also suggested to play a role in the hardness enhancement, where the immersion process accelerates the reaction of residual monomers into polymers, thus increasing the matrix density. These findings imply that microhybrid composite resins demonstrate favorable mechanical stability against the chemical challenges posed by various mouthwashes, even showing an advantageous hardening effect under the tested conditions. However, the study acknowledges limitations, primarily related to the controlled *in vitro* experimental setting, which does not fully replicate the complex environment of the oral cavity, including temperature fluctuations, masticatory forces, and salivary flow. Therefore, it is recommended that future research involve more sophisticated *in vitro* or *in vivo* studies to evaluate long-term material performance, incorporating other physical and chemical properties such as water sorption and *filler* release, and utilizing simulation models that more closely mimic actual clinical conditions.

CONFLICT OF INTEREST

No potential conflict of interest was reported by the authors

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