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Effect of Habitual intakes on Hardness and Color of Resin Composites

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Abstract

Background: Both surface hardness and color stability are among the most important physical factors that govern the success of different composite restoratives.

Aim: This in vitro study aimed to evaluate both surface hardness and color change in different resin composites following their immersion in different media of some habitual intakes.

Methods: One hundred disc specimens were constructed in 2 groups out of methacrylate (Tetric N-Ceram Bulk Fill, Ivoclar Vivadent) and silorane-based (Filtek P90, 3M ESPE) composites (n=50 each). Specimens of each group were subjected to an interrupted immersion in 5 different media (subgroups 1-5, n = 10), water (control), natural saliva, alcohol, lime juice and khat extract for 4 weeks. Vickers hardness of all specimens was assessed on a micro-hardness tester under a load of 50g for 10s. The color parameters (L*, a* and b*) of each specimen were measured before and after immersion. The difference in specimens' color (ΔE) was calculated according to CIE formula. The collected data for all subgroups were statistically analyzed using 1-Way ANOVA and Tukeys' comparisons at $\alpha = 0.05$ to stand on the significance of differences detected between the tested variables.

Results: No difference in hardness value was recorded between composite groups ($P > 0.05$). In comparison to the control subgroup, no significant differences were noticed in the hardness values of composites in subgroups 2-4 ($P > 0.05$). A significant reduction in hardness values were recorded for composite specimens stored in khat extract ($P < 0.05$). All composite specimens showed different degrees of color change with no significant differences between groups ($P > 0.05$). Specimens in subgroups 1-4 had similar visually perceptible color change ($1 < \Delta E < 3.3$), while those in Subgroup 5 showed higher clinically unacceptable color change ($\Delta E > 3.3$) in comparison to other subgroups ($P < 0.05$).

Conclusion: Silorane-based resin composite present comparable behavior to that of Bulk-fill methacrylate-based composite in different media. All the tested intakes have no adversity on composites' hardness and color, however Khat extract seems harmful to composites' surfaces.

Keywords: Alcohol; Color; Composite; Hardness; Khat; Silorane

Introduction

Facial beauty depends to a great extent on the actual teeth esthetics. Accordingly, esthetic restoration of mutilated, discolored, and in some instances, mal-aligned teeth became frequent in everyday dental practice [1]. Current esthetic restorative usually provides ease of application and durable

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bonding to hard tooth tissues; however, the clinical longevity of dental restorations usually depends not only on the intrinsic characteristics of the restoratives used but also on their survival in the different oral environment, they are exposed to [1-5]. Surface hardness is one of the most important physical factors that govern the success of different composite restoratives [1,3,4]. Hardness often indicates about materials' ability to resist abrasion or wear. Composite surface hardness is usually referred to material's composition and degree of resin polymerization [2,6]. Methacrylate-based composites used to exhibit acceptable clinical durability; however, their wearing behavior was a point of query, especially in patients with extraordinary occlusal habits [7,8].

Dental manufacturer has recently introduced composite restoratives with different filler and resin phases. Their primary aim was to offset the recognized material's shrinkage at the time of light curing. Incorporation of nano-sized fillers into resin composite formulations not only helped optimize materials' optical characteristics but also improved its wear resistance [8]. In comparisons to methacrylate-based composites, employing silorane-based resin offered composite material with comparable properties and lower polymerization shrinkage [9]. Some researchers [9,10] confirmed higher or even comparable wear behavior of this material in oral environment [10]. On the other hand, the oral cavity is a complex, aqueous environment where composite restorations are continuously exposed to different chemicals found in saliva, acidic foods, beverages, and alcohol-containing mouth rinses [7-10]. Consequently, in the short or long term of service, composite hardness could be affected as a result of influencing both physical and chemical nature of the resin polymer networ [9-11]. One study evaluated the effect of solutions with different pH on both solubility and wear rates of methacrylate and silorane-based resin composites [7]. The results revealed increased water solubility and wearing in response to the increased acidity of the storage solution. In addition, the adverse effect of acidic media was more prominent in methacrylate than silorane-based composite. Although the data of another study [10] revealed the minimal effect of toothbrushing on the surface roughness of silorane-based composite in comparison to methacrylate-based ones, the same material showed increased wearing particularly after pH cycling. The durability of composite color in oral environment is dependent on both material's composition and surface characteristics [11]. Some studies reported that composites' color stability is usually affected by resin matrix composition, polymerization associated additives, degree of polymerization, and fillers' characteristics [12-16]. However, other studies [17,18] indicated the possible influence of the immersion media on the change in composites' color. The silorane-based composite, on the other hand, has been proved to show higher degree of polymerization and acceptable surfaces characteristics in comparison to methacrylate-based composites [10,19]. Accordingly, many in vitro studies revealed their lower susceptibility to staining or discoloration by common beverages and food intakes [20].

On the other hand, habitual chewing of khat is a common habit among the population in Yemen, and some African and European countries in addition to the Southern part of Saudi Arabi [15]. Throughout the chewing cycle that extend to 3 or more h, the saliva/leaf slurry is normally be in contact with teeth and restorative materials [21]. Many studies did confirm the adverse effects of khat chewing on both gingival tissues and tooth substrates [22-24]. However, there is an obvious shortage in data about its effect on both color and surface hardness of the restorative materials. Therefore, the objective of this in vitro study was to evaluate the effect of different immersion media on surface microhardness and color stability of microhybrid methacrylate and silorane-based composite materials. The immersion media used were saliva, alcohol, lime juice, and khat.

Materials and Methods

A total of 100 disc specimens, 7 mm in diameter and 2 mm high, were fabricated in two groups out of methacrylate (MC, Group 1, n=50) (Tetric N-Ceram Bulk Fill, Ivoclar-Vivadent, Schaan, Liechtenstein) and silorane-based (SC, Group 2, n = 50) (Filtek P90, 3M ESPE, St. Paul, MN) hybrid composite restoratives [Table 1]. All specimens were fabricated with flat top and bottom surfaces in silicone molds (Express STD, 3M ESPE, St. Paul, MN) and between 2 plastic strips supported with microscope glass plates. Each specimen was built in layers 1 mm thick, each subjected to LED curing light at wavelength 430–480 nm, and intensity of 1200 mW/cm^2 (Elipar S10, 3M ESPE AG, Seefeld, Germany) for 20 s. To help have a standard condensation and flattening of specimens' top surfaces, the outermost composite layer was packed under a standardized weight of 400 g. The weight was applied constantly against the top glass plate and both removed immediately before conducting the curing process [27]. After releasing of the cured specimens out of the constructing molds, both top and bottom surfaces were subjected to an extra shot of the curing light to ensure their harmonized curing. The bottom surface of each cured specimen was also marked up with sharp hand instrument to help distinguish between the top and the bottom surfaces. All specimens were then stored in drinking water at 37°C ± 1°C in light-proof containers for 24 h to ensure optimum polymerization.

All specimens in each group were then subjected to 4 weeks long of intermittent immersion (15 min/3 times) daily) in 4 different media (subgroups 1, 3–5, n = 10 each) representing some of the usual daily intakes in different regions of the world, these are bottled water (negative control) (Aquafina, Pepsi, Abha, KSA), lime juice (Orient Provision and Trading Co.,

Abdelaziz KM and Saleh AA., Dent Res Oral Health 2024
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Ltd., Jeddah, KSA), ethyl alcohol (Scharlab SL, Barcelona Spain), and aqueous extract of natural khat plant. In addition, another 10 specimens (subgroup 2) served as positive control following their immersion in natural human saliva. To mimic the normal consumption regime, the immersion periods were interrupted with equal lags of immersion in water. Khat extract was prepared at Physiology Department, College of Medicine, King Khalid University using the technique used by Al-Hashem et al. [26] and described in table 1. Natural saliva was collected by a physiologist from medically free volunteer individuals [25] following the approval of King Khalid University, College of Dentistry Ethical Committee.

The Vickers' hardness of MC and SC was assessed on the bottom surfaces of the constructed specimens (n = 50 out of each group/n = 10 for each subgroup) using ZHV Hardness Tester, (Zwick/Roell Indentec, Ulm, Germany) having diamond indenter with equal diagonals and under 50 g weight with 15 s dwell time [28]. Each specimen was subjected to 5 successive indents distributed all over the surface. The Vickers' hardness number for each indent was automatically calculated by the aid of machine's built-in computer, and the mean number for each specimen was then determined.

The color parameter CIE L*a*b* of the top surfaces of the same 100 specimens (n = 50 out of each group/n = 10 for each subgroup) was measured against white background using Handy Digital Spectrophotometer (Easyshade Advance, Model # DEASYCHP, Serial No. H25058, VITA Zahnfabrik H. Rauter GmbH and Co., Bad Sackingen, Germany), where L* refers to the lightness value that ranges from 0 (black) to 100 (white), a* refers to red-green parameters, where red is the positive values, and green is the negative values, and b* refers to yellow-blueparameters where yellow is the positive values, and blue is the negative values [27]. The reference shades for all specimens assigned for color change assessment were measured on the top surface of each before their immersion in any of the immersion media. Shades of the same specimens' surfaces were measured following the immersion in both control and test drinks. The differences between colors (ΔE) determined before and after immersion were then calculated according to the following equation:

$$\Delta E = [(L^*_2 - L^*_1)^2 + (a^*_2 - a^*_1)^2 + (b^*_2 - b^*_1)^2]^{1/2}$$

Where, L^*_1, a^*_1, and b^*_1 are the baseline color parameters, and L^*_2, a^*_2, and b^*_2 are the color parameters measured after immersion.

Following the calculation process, the mean color changes in all subgroups were classified as (1) visually nonperceptible, when ΔE values were <1; (2) visuall perceptible (clinically

Table 1: Materials used

Restorative resin composites			
Material	**Description**	**Composition**	**Manufacturer**
Tetric N-Ceram Bulk Fill (MC)	Hybrid, methacrylate-based resin composite	Dimethacrylate monomers (Bis-GMA, Bis-EMA, and UDMA), total content of inorganic fillers 75-77 wt% (barium glass, ytterbium trifluoride, mixed oxide) with average size of 0.04-3.0 µm, additives, catalyst, stablizers, and pigments	Ivoclar-Vivadent, Schaan, Lichtenstein
Filtek P90 (SC)	Hybrid, silorane-based resin composite	3,4 epoxycyclohexylethylcyclopolymethylsiloxane, Bis 3,4-epoxycyclohexylethyl-phenylmethylsilane, 76 wt% silanized quartz, yttrium fluoride with average particle size of 0.1-2.0 µm	3M ESPE, St. Paul, MN
Immersion media			
Name	**Description/preparation**		**Manufacturer/ source**
Aquafina	Fluoridated water with 110 ppm total dissolved minerals at pH=7.0		Pepsi, Abha, Saudi Arabia
Saliva	Whole saliva samples collected from medically free individuals using passive drooling technique[25]		Natural
Alcohol	99.9% ethanol absolute		Scharlab SL, Barcelona, Spain
Freshly	100% lime juice from concentrate		Orient Provision and Trading
	2 full teaspoons mixed with 200 ml of drinking water		Co, Ltd, Jeddah, KSA
Khat extract	Two hundred grams of fresh stem and leaves of Khat shrubs (*Catha edulis*) were washed, dried, and extracted with 500 mL of water-ethanol mixture (70:30 volume%) at room temperature and then filtered. The filtrate was evaporated in a vacuum at 40°C to remove all traces of ethanol. The resulting ethanol-free extract was dissolved in freshly prepared normal saline to a final concentration of 200 mg/ml[26]		Natural

Bis-GMA=Bisphenol A-glycidyl methacrylate, Bis-EMA=Bisphenol A dimethacrylate, MC=Methacrylate-based composite, SC=Silorane-based composite, UDMA=Urethane dimethacrylate

Abdelaziz KM and Saleh AA., Dent Res Oral Health 2024
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acceptable), when E values were ranged from 1 to 3.3; and (3) clinically unacceptable, when ΔE values were >3.3 (i.e., dramatic color change) [29-31]. The mean and standard deviation of ΔE in each class were tabulated to help compare the results [Table 2]. The collected hardness and color change data were then statistically analyzed using one-way ANOVA and Tukey's pairwise comparisons at $\alpha = 0.05$ to determine the source of differences detected (if any) between subgroups.

Results

The mean hardness values and standard deviations of composite specimens in all subgroups were presented in table 2. For each group (either methacrylate or silorane-based resin composite), no difference ($P > 0.05$) was detected in the hardness values of composite specimens immersed in saliva, lime juice, and alcohol (subgroups 2–4) in comparison to the negative control (immersed in drinking water-subgroup 1). On the other hand, specimens immersed in the extract subgroup 5) showed lower hardness values than other specimens immersed in water, saliva, lime juice, and alcohol (subgroups 1–4) ($P < 0.05$) for both methacrylate and silorane-based composites.

Shifting in color parameter (ΔL*, Δa*, and Δb*) and the calculated color differences (ΔE) in all subgroups were listed in table 3. Within each group (composite type), only specimens immersed in khat extract showed a significant color change in comparison to those in other subgroups (Tukey's, $P < 0.05$). Within the same subgroups, no difference (Tukey's, $P > 0.05$) was detected between methacrylate and silorane-based composites in terms of color change following the immersion in different media. Immersion of Group 1 specimens (methacrylate-based composite) in both water and saliva recorded clinically, visually perceptible color change (1< ΔE <3.3), while immersion in lime juice, alcohol, and khat extract caused clinically unacceptable color change (ΔE ≥3.3).

In Group 2 (silorane-based composite), only those specimens immersed in lime juice and khat extract caused clinically unacceptable color change (ΔE ≥3.3), whereas the other subgroups showed visually perceptible change in color. Both methacrylate and silorane-based specimens showed brighter colors following their immersion in alcohol. On the red-green axis, silorabe-based specimens showed reddish (positive Δa*) discoloration in all subgroups, whereas methacrylate-based specimens showed greenish discoloration (−ve Δa*) in all subgroups. On the yellow-blue axis, methacrylate-based specimens showed bluish (−ve Δb*) discoloration in all subgroups, whereas silorane-based specimens showed yellowish discoloration (+ve Δb*) in subgroups 1, 2, 4, and 5. The only silorane-based specimens that showed bluish discoloration was that immersed in lime juice (subgroup 3, −ve Δb*).

Table 2: Hardness values of composite specimens following immersion in different media

	Immersion media				
Composite material	**Water (negative control)**	**Saliva (positive control)**	**Lime juice**	**Alcohol**	**Khat extract**
MC	65.75±8.07^{a}	59.33±8.43^{a}	55.66±4.52^{a}	56.83±5.15^{a}	35.25±4.18^{b}
SC	69.41±5.98^{a}	62.00±4.39^{a}	57.58±7.55^{a}	60.33±10.39^{a}	36.91±1.66^{b}

Same superscripts indicate insignificant differences between test subgroups. MC=Methacrylate-based composite, SC=Silorane-based composite

Table 3: Differences in composites' color parameters following immersion in different media

Immersion media	MC				SC			
	ΔL*	Δa*	Δb*	ΔE	ΔL*	Δa*	Δb*	ΔE
Water (negative control)	−1.22±0.79	−1.59±0.46	−1.13±0.89	3.05±0.34^{a,1}	−0.75±0.88	1.37±0.47	1.46±0.86	2.61±0.92^{a,1}
Saliva (positive control)	−1.77±0.90	−0.56±0.26	−1.02±1.79	2.74±0.89^{a,1}	−1.35±1.02	1.23±0.39	1.09±1.32	2.89±0.67^{a,1}
Lime	1.64±0.51	−0.76±0.51	−2.22±0.79	3.61±0.64^{a,1}	−1.72±1.26	1.47±0.41	−2.23±1.50	4.25±1.67^{a,1}
Alcohol	1.99±1.07	−1.23±0.30	−2.23±0.70	3.99±0.79^{a,1}	1.12±0.61	0.39±0.26	1.34±1.63	2.24±0.97^{a,1}
Khat	−0.28±1.01	−2.38±0.41	−3.88±0.43	5.68±0.54^{b,1}	−1.67±1.89	1.08±0.21	3.10±0.92	4.79±0.72^{b,1}

Higher ΔE indicates higher color change (ΔE <1 indicates visually nonperceptible color change, 1< ΔE <3.3 indicates visually perceptible change, and ΔE >3.3 indicates clinically unacceptable color change). +ve ΔL*=Values indicate shifting to brighter color, −ve ΔL*=Values indicate shifting to darker color, +ve Δa*=Values indicate shifting to reddish color, −ve Δa*=Values indicate shifting to greenish color, +ve Δb*=Values indicate shifting to yellowish color, −ve Δb*=Values indicate shifting to bluish color. For each composite type, the same superscript letters in each column indicate insignificant differences from the control. The same superscript numbers in each row indicate insignificant differences between groups. MC=Methacrylate-based composite, SC=Silorane-based composite

Abdelaziz KM and Saleh AA., Dent Res Oral Health 2024
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Discussion

Longevity of resin composite restorations is normally affected by many physical factors [1,2,5]. Optimum composite hardness usually offers restorations with proper resistance to scratching and wear and those accordingly help maintain the proper restorations' texture, anatomy, and function [1]. This issue is true especially in patients with paranormal occlusal functions. Composite color stability helps maintain the desired esthetic qualities of the performed restorations throughout its service life [1,2,5]. Many factors were documented to affect both properties including the effect of habitual and daily intakes [9-11]. At the same time, some communities are known to have special habits, including the consumption of alcoholic beverages and tobacco or khat chewing, that could also harm the existing resin composite restorations [15,21-24]. Therefore, the current in vitro study aimed to evaluate the effect of immersion in different media representing habitual and 1 daily intakes on both surface hardness and color stability of commercially available, chemically-different resin composite restoratives. The intermittent immersion of specimens in the tested media was designed to mimic the usual consumption of different intakes. Food and drinks normally get a transient contact with teeth and restorations' surfaces before they are washed away [32]. The designed null hypothesis was that the tested media, water, saliva, alcohol, lime juice, and khat extract, would have no effect on composites' hardness and color in spite of their different chemistries. In agreement with Kusgoz et al. [33] who reported comparable hardness of both silorane and methacrylate-based composites, results of the current in vitro study indicated no difference between surface hardness of both types of composite restoratives tested [Table 2]. This result could be referred to the comparable filler loadings of the tested composites. Both restoratives are belonging to the hybrid type of resin composites with minimal difference in their filler loadings (75–77 and 76 wt% for MC and SC) and sizes (0.04–3.0 and 0.1–2.0 Δm for MC and SC) that probably affect the total fillers' distribution on specimens' surfaces and consequently their hardness values. This postulation could be supported by the statements of many researchers [34-36] those indicated a significant effect of fillers' size and distribution on some physical and mechanical properties, including surface hardness. On the other hand, the role of resin matrix on surface hardness was declared by some researchers [36]. Although Agrawal et al. [37] recorded higher hardness of the silorane-based composite in response to its higher degree of conversion than methacrylate-based one, others, in contrary, Guiraldo et al. [38] deduced greater degree of polymerization of the methacrylate-based composite compared to the silorane-based composite. This finding has been interpreted by the higher surface hardness of methacrylate-based composite in comparison to the silorane-based one [39]. However, the composition of the newly introduced bulk-fill, methacrylate-based resin composites usually contains flexing and plasticizing resins, such as Bis-EMA of the MC utilized in this study, those could reduce the hardness of the cured material nearly to the level achieved by the silorane-based resin composite [40].

In addition to the material type, both storage solution and the immersion time were reported to be significant factors influencing composites surface hardness [41]. Results of the current study indicated that both composite materials showed no difference in surface hardness of specimens immersed in water, saliva, lime juice, and alcohol. However, those specimens immersed in khat extract exhibited lower hardness values in comparison to the control subgroups (water and saliva). These results could be referred to the chemical nature of the immersion solutions, as water, saliva, lime juice, and alcohol are weak agents with minimal degradation effect on composite's resinous content [42]. In addition, the interrupting washing periods help wash those chemical away from specimens surface minimizing their contact time and accordingly the chance to adversely affect surfaces resin matrix of the tested composites [43]. On the other hand, khat extract was documented to have an alkaline contents that could have a more serious effect on the resin components of the tested composite [21]. Many studies proved a significant reduction in hardness and increased solubility of composites subjected to alkaline media and those findings for sure can support the results of the current study [44,45].

The known classification of color change [27] into nonperceptible ($\Delta E < 1$), visually perceptible or clinically acceptable (ΔE = 1–3.3), and clinically unacceptable ($\Delta E > 3.3$) was used in the current study to indicate about the clinical relevance of the noticed shift in composites' color following their exposure to the selected test media. In spite of the obvious advantages of the currently available composite restoratives, these materials are still having the ability to be stained/discolored in service when exposed to different food and fluid intakes, that in turn, could adversely affect their clinical service life [17,18,27]. The previous studies [12,32] indicated that silorane-based composites are less susceptible to staining/discoloration in different media in comparison to methacrylate-based resin composites. Authors of those studies referred their results to the hydrophobic nature and accordingly the lower fluid sorption of siloran resin matrix. However, results of the current study came in disagreement indicating no difference in the color change values (ΔE) of both types of resin composites (MC and SC). The recorded values [Table 3] usually lied within the clinically perceptible range ($1 < \Delta E < 3.3$) for specimens in contact with water, saliva, lime juice, and alcohol (subgroups 1–4). At the same time, those values were clinically unacceptable ($1 < \Delta E < 3.3E > 3.3$) in contact with khat extract in (subgroup 5) with no significant difference between both composites. These findings could be explained based on the documented

Abdelaziz KM and Saleh AA., Dent Res Oral Health 2024
DOI:10.26502/droh.0077

hydrophobicity of silorane resin [12,32,46] together with the hydraulic expansion of methacrylate-based resin matrix and silane coupling agent during the initial incubation period in water [47].

Both mechanisms, in turns, can probably minimize the sorption behavior of both composites in different media reducing their susceptibility to discoloration. Benetti et al. [48] confirmed no difference between methacrylate and silorane-based resin composites in response to storage in water and citric acid although they reported different influence of alcohol on both materials. Their results are partially agreed with the result of the current study that revealed no significant difference between the discoloration tendencies of both MC and SC in similar media (water, alcohol, and lime juice). The existing contradiction is probably the result of using the same solution throughout their experimental periods in addition to the prolonged immersion time that could reach 180 days. In comparison, the immersion of composite specimens in this in vitro study was interrupted by cyclic washing to mimic the normal consumption situation and last for only 4 weeks. [32] Many of the previous studies [27,48] indicated that storing resin composite specimens in water and alcohol affects its color parameters especially the brightness. These findings were coincide with the results of the current study that showed minimal clinically perceptible color change ($1.0 > \Delta E < 3.3$) in MC and SC specimens following their immersion in water, saliva, alcohol, and lime juice. This finding could be referred to the clear, nonstaining nature of those solutions in addition to the limited sorption of the nominated solutions onto the tested composites [49]. On the other hand, the color change of test specimens in alcohol was also accompanied with an increase in their brightness (+ve ΔL) and this could be a direct effect of the organic dissolving effect of the alcohol [5,48]. At the same time, significant darkening (−ve ΔL) and staining ($\Delta E > 3.3$) of both MC, SC in the aqueous solution of khat extract could be normal in response to the softening effect of khat extract on composites' resin matrices that, in turn, enhance the sorption process of the greenish stain of the khat onto composite specimens [21,27,42]. Because composites could exhibit different degrees of polymerization in response to different curing conditions, this variable should be considered for further evaluation following immersion in the same uptakes.

Conclusion

Silorane-based resin composite presents comparable behavior to that of bulk-fill methacrylate-based composite in different media. All the tested intakes have no adversity on composites' hardness and color; however, khat extract seems harmful to composites' surfaces.

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Conflicts of interest

There are no conflicts of interest.

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Abdelaziz KM and Saleh AA., Dent Res Oral Health 2024
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