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Assessment of thermocycling effect on surface roughness and color stability in nanoceramic hybrid restorative materials

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Abstract:

Thermal stress can adversely affect the surface integrity and colour stability of dental restorative materials, compromising their long-term clinical performance. Therefore, it is of interest to evaluate the effect of 10,000 thermocycles on the surface roughness and colour stability of three nanoceramic hybrid restorative materials compared with a conventional nanohybrid composite using atomic force microscopy and CIELAB spectrophotometric analysis. After thermocycling, nanoceramic hybrids showed significantly lower surface roughness (Ra: 0.18–0.24 μm) and lower colour change (ΔE : 1.82–2.14) than the conventional nanohybrid composite (Ra: 0.41 μm ; $p < 0.001$). Nanoceramic hybrids may therefore provide improved long-term aesthetic and clinical performance in restorative dentistry. Thus, data shows the resistance of nanoceramic hybrid materials to thermally induced surface degradation and colour alteration.

Keywords: Surface roughness (Ra), colour stability, restorative materials**Background:**

The nanoceramic hybrid materials are an evolutionary step in dental restorative technology where the aesthetic qualities of the resin composites are merged with the mechanical strength and wear resistance of the ceramics [1]. These materials involve the use of nanoscale ceramic particles in a resin-based structure that forms a special microstructure that should be able to maximize functional and aesthetic results in both direct and indirect restoration work [2]. Nanoceramic technology integration is to overcome the various limitations of the traditional composites such as surface degradation, color stability and wear resistance under clinical environment [3]. The roughness of the surface and color stability is some important parameters that determine long-term effectiveness of aesthetic restorations. When the surface is rough, bacterial adhesion, biofilm growth and plaque retention become significantly easier and there is a possibility of secondary caries and periodontal inflammation [4]. Moreover, the rough surfaces have a greater susceptibility to stains and discoloration that impact negatively on aesthetic results and patient satisfaction [5]. Stability of color is especially relevant in the anterior restoration where aesthetic needs are paramount and color shifts beyond the clinical acceptability limits (> 3.3) will require replacement of the restoration [6]. Another common *in vitro* approach to modeling thermal stresses to which dental restorations are subjected in the oral cavity is thermocycling [7]. The thermal processes of expansion and contraction caused by the repeated exposure to thermo-cycles during the use of food and beverages are likely to worsen the material matrix, interfaces between filler and resin and speed up surface corrosion [8]. The international organization of standardization (ISO) suggests thermocycling guidelines to assess the material sustainability and usually, they use temperature intervals of 5°C to 55°C with different cycles to represent different clinical periods [9]. There are recent studies that explored a range of features of the nanoceramic hybrid materials such as their mechanical properties, wear behavior and optical properties [10, 11]. Experiments have also achieved positive flexural strength and fracture toughness with these materials in relation to the traditional composites [12]. Experiments comparing the

polishability of nanoceramic hybrids and their starting surface conditions have shown good results and some of the materials are nearly as smooth as feldspathic ceramics in their surfaces [13]. Nevertheless, the extent to which these materials can sustain thermal stressing conditions is poorly described. Although some studies have examined the impact of thermocycling on traditional composite resins [14, 15], few comparative studies focusing on the analysis of thermal degradation resistance of nanoceramic hybrid materials have been studied in the literature. The compositional and structural peculiarities of nanoceramic hybrids are indicative of possibly dissimilar behavior to thermal cycling than existing composites and should be systematically investigated [16]. Therefore, it is of interest to evaluate the effect of thermocycling on surface roughness and color stability of nanoceramic hybrid restorative materials in comparison with conventional nanohybrid resin composites.

Materials and Methods:**Study design and sample preparation:**

The present *in vitro* experimental study took place in the Department of Dental Materials Science in a period between April 2024 and June 2024. Eighty of them were produced as disc-shaped specifications and placed in four groups ($n=20$ each) according to the type of material: Group A (Ceram.x SphereTEC One, Dentsply Sirona, Germany), Group B (Admira Fusion x-tra, VOCO, Germany), Group C (Vita Enamic composite, Vita Zahnfabrik, Germany) and Group D (Filtek Z350 XT nanohybrid composite, 3M ESPE, USA) (as a control). Power analysis was used to determine the sample size (use $\alpha=0.05$, $20=0.20$ and effect size=0.75, as the smallest possible sample was 18 specimens per group and 20 was taken to represent possible losses). The fabrication of the specimens was made by using standardized cylindrical Teflon molds (10 mm diameter and 2 mm thickness) mounted on the glass slides with polyester strips. The material was put in a single increment and a second polyester strip over it followed by compression of the material using a glass slide and subsequently curing using the LED curing device (Bluephase Style, Ivoclar Vivadent, Liechtenstein)

at the light intensity of 1100 mW/cm² and curing it under the specifications of the manufacturer. To fully cure it, both sides were cured (40 seconds on each side). Group C specimens were pre-polymerized vitra blocks of Vita Enamic, which were cut into 2 mm-thick discs using a precision cutter (Isomet 1000, Buehler, USA) with the water cooling the blocks before being subjected to polishing in order to match the thickness of the others. After 24-hour storage in the dark at 37°C, all the specimens were taken out of molds and voids or defects was examined under a magnifying lens (×10) and the faulty specimens were discarded and replaced.

Surface finishing and polishing guideline:

Finishing and polishing a standardized finishing and polishing of all surfaces on the specimen were done to provide baseline surface properties. The polishing was done in decreasing grit size: coarse (dark orange), medium (orange), fine (light orange) and superfine (yellow): sequential polishing was done with aluminum oxide abrasive discs (Sof-Lex Pop-On, 3M ESPE, USA). The discs were applied to each side and allowed to rotate 30 seconds at 10,000 rpm with light pressure with the water coolant in a low-speed handpiece. Last polishing used a diamond polishing paste (Dia- Polish, EVE Ernst Vetter, Germany) and 60 seconds per specimen using felt wheels. Before measuring the baseline 10 minutes of ultrasonic cleaning with distilled water, followed by air-drying and 24 hours of storage in artificial saliva with a temperature of 37°C, was carried out on the specimens.

Thermocycling protocol:

Thermocycling was done with an automated thermocycling device (THE-1100, SD Mechatronik, Germany) as per ISO 11405:2015. Specimens were made to undergo 10,000 thermal cycles between a 5°C and 1°C water bath and a 55°C, 1°C water bath with 30s dwell time per bath and 10s transfer time. This protocol is a simulation of about 1 year of clinical service using the known conversion factors. To get homogenous conditions of exposure, the specimens were randomly placed in thermocycling chambers.

Surface roughness analysis:

Atomic force microscopy (AFM, Park NX10, Park Systems, South Korea) was used to measure the surface roughness in non-contact mode. Scanning was conducted on three random spots of 50 µm × 50 µm on each specimen at 0.5 Hz and a resolution of 256 × 256 pixels. The mean roughness (Ra), root mean square roughness (Rq) and peak-to-valley height (Rz) were computed by use of XEI software (Park Systems). Measures were taken at baseline (T0), T1 (5,000 cycles) and T2 (10,000 cycles). Three measurements were taken on each score and averaged to come up with representative mean scores.

Color stability assessment:

The color measurements were carried out with a portable spectrophotometer (VITA Easyshade V, VITA Zahnfabrik, Germany) which was calibrated every time with the

manufactures specifications. The specimens were placed in a standardised white surface and measurements were made at the location of the middle of a specimen in D65 standard illuminant conditions. The coordinates of the CIELAB color space at T0, T1 and T2 were noted as L, a and b. The color difference (ΔE) was computed by a formula: $\Delta E = [(DL^* 2 + aL^* 2 + bL^* 2)0.5]$, where 9 is the difference between the baseline and the post-thermocycling values. To reduce the variation of the operator, the means of three measurements per specimen were taken.

Statistical analysis:

The statistical analysis was done on SPSS version 28.0 (IBM Corp., USA). Kolmogorov Smirnov tests, Shapiro-Wilk tests and the use of histogram analysis were used to assess the data normality. Descriptive statistics such as means, standard deviations and 95 percent confidence interval were used. The roughness and color parameters of groups at each point were compared through one-way ANOVA with the post-hoc test of Tukey. ANOVA repeated measures were used to assess changes within the groups over time. The relationships between surface roughness and color changes were evaluated by Pearson correlation analysis. The level of statistical significance was set at $p < 0.05$ correcting through Bonferroni.

Results:

All eighty specimens completed the thermocycling protocol without visible cracks, fractures, or delamination. Data showed normal distribution ($p > 0.05$, normality tests), permitting parametric statistical analysis. **Table 1** presents comparative surface roughness parameters (Ra values) across all tested materials at different thermocycling intervals. Significant differences were observed among materials and across time points. At baseline, no significant differences existed among groups ($p = 0.127$). After 5,000 cycles, Group D exhibited significantly higher Ra values compared to Groups A, B and C ($p < 0.001$). After 10,000 cycles, conventional composite (Group D) showed the highest surface roughness ($0.41 \pm 0.07 \mu\text{m}$), significantly exceeding all nanoceramic hybrid groups ($p < 0.001$). Among nanoceramic materials, Group A showed the lowest roughness ($0.18 \pm 0.04 \mu\text{m}$), though differences among nanoceramic groups were not statistically significant ($p > 0.05$). The percentage increase in surface roughness from baseline to 10,000 cycles was: Group A (125%), Group B (133%), Group C (100%) and Group D (273%). Repeated measures ANOVA revealed significant effects of thermocycling ($F = 198.3$, $p < 0.001$), material type ($F = 87.6$, $p < 0.001$) and their interaction ($F = 23.4$, $p < 0.001$). **Table 2** summarizes color stability parameters across materials and thermocycling intervals. All materials exhibited color changes, with varying magnitudes and clinical significance. At 10,000 cycles, all nanoceramic hybrid materials maintained ΔE values below the clinical acceptability threshold ($\Delta E < 3.3$), while conventional composite exceeded this limit ($\Delta E = 3.76 \pm 0.51$). Group D showed significantly higher color changes compared to all nanoceramic groups at both measurement intervals ($p < 0.001$). Among nanoceramic materials, no statistically significant differences were detected

($p=0.284$ at 5,000 cycles; $p=0.315$ at 10,000 cycles). **Table 3** presents the detailed changes in individual CIELAB color coordinates for all materials after 10,000 thermocycles, providing insight into the nature of color alterations. All materials exhibited darkening (negative ΔL^*), slight reddening (positive Δa^*) and yellowing (positive Δb^*), with yellowing representing the predominant color change vector. Group D showed significantly greater changes in all three coordinates compared

to nanoceramic groups ($p<0.001$). The yellowing component (Δb^*) contributed most substantially to overall color change in all materials. Pearson correlation analysis revealed significant positive correlations between surface roughness (R_a) and color change (ΔE) values ($r=0.742$, $p<0.001$), suggesting that increased surface roughness facilitates color alteration. Both null hypotheses were rejected as thermocycling significantly affected both surface roughness and color stability.

Table 1: Mean surface roughness (R_a , μm) comparison across thermocycling intervals

Material Group	Baseline (T0)	5,000 Cycles (T1)	10,000 Cycles (T2)	p-value*
Group A (Ceram.x)	0.08 $\pm$ 0.02 ^a	0.14 $\pm$ 0.03 ^a	0.18 $\pm$ 0.04 ^a	<0.001
Group B (Admira Fusion)	0.09 $\pm$ 0.02 ^a	0.16 $\pm$ 0.04 ^a	0.21 $\pm$ 0.05 ^a	<0.001
Group C (Vita Enamic)	0.12 $\pm$ 0.03 ^a	0.18 $\pm$ 0.04 ^a	0.24 $\pm$ 0.05 ^a	<0.001
Group D (Filtek Z350)	0.11 $\pm$ 0.03 ^a	0.28 $\pm$ 0.06 ^b	0.41 $\pm$ 0.07 ^b	<0.001
p-value**	0.127	<0.001	<0.001	-

Within-group comparison across time (Repeated measures ANOVA)

**Between-group comparison at each time point (One-way ANOVA)

Different superscript letters indicate significant differences between groups at each time point ($p<0.05$, Tukey's test)

Values expressed as mean $\pm$ SD

Table 2: Color change (ΔE) values after thermocycling

Material Group	ΔE at 5,000 Cycles	ΔE at 10,000 Cycles	Clinical Interpretation**	p-value*
Group A (Ceram.x)	1.04 $\pm$ 0.23 ^a	1.82 $\pm$ 0.31 ^a	Clinically acceptable	<0.001
Group B (Admira Fusion)	1.18 $\pm$ 0.28 ^a	1.96 $\pm$ 0.34 ^a	Clinically acceptable	<0.001
Group C (Vita Enamic)	1.26 $\pm$ 0.31 ^a	2.14 $\pm$ 0.38 ^a	Clinically acceptable	<0.001
Group D (Filtek Z350)	2.14 $\pm$ 0.42 ^b	3.76 $\pm$ 0.51 ^b	Clinically unacceptable	<0.001
p-value***	<0.001	<0.001	-	-

Within-group comparison between time points (Paired t-test)

**Based on ΔE threshold: $\Delta E < 3.3$ = clinically acceptable

***Between-group comparison at each time point (One-way ANOVA)

Different superscript letters indicate significant differences between groups ($p<0.05$, Tukey's test)

Values expressed as mean $\pm$ SD

Table 3: Changes in CIELAB color coordinates after 10,000 thermocycles

Material Group	ΔL^* (Lightness)	Δa^* (Red-Green)	Δb^* (Yellow-Blue)	Dominant Change
Group A (Ceram.x)	-0.82 $\pm$ 0.34 ^a	+0.34 $\pm$ 0.18 ^a	+1.56 $\pm$ 0.28 ^a	Yellowing
Group B (Admira Fusion)	-0.91 $\pm$ 0.38 ^a	+0.41 $\pm$ 0.22 ^a	+1.63 $\pm$ 0.31 ^a	Yellowing
Group C (Vita Enamic)	-1.04 $\pm$ 0.42 ^a	+0.38 $\pm$ 0.19 ^a	+1.74 $\pm$ 0.35 ^a	Yellowing
Group D (Filtek Z350)	-1.87 $\pm$ 0.46 ^b	+0.76 $\pm$ 0.28 ^b	+3.12 $\pm$ 0.48 ^b	Yellowing
p-value*	<0.001	<0.001	<0.001	-

Between-group comparison (One-way ANOVA with Tukey's post-hoc test)

Different superscript letters indicate significant differences between groups ($p<0.05$)

Values expressed as mean $\pm$ SD; Negative ΔL indicates darkening; Positive Δa^* indicates reddening; Positive Δb^* indicates

Discussion:

This paper shows that nanoceramic hybrid restorative materials have better thermal degradation resistance than the traditional nanohybrid composites, which appear as the lack of surface roughness development and stability of color after 10,000 thermal cycles. The implications of these findings are significant in the choice of materials when it comes to selecting materials in clinical cases where long-term aesthetic and functional stability are essential. The improved effect of nanoceramic hybrids is explainable by their compositional and structural peculiarities. These materials use ceramic particles at the nanoscale with a high filler loading (generally 70-80 by weight) and form a denser microstructure with less volume of resin matrix [1, 2]. Such high filler content reduces exposure of resin matrix to thermal stress thus minimizing the possibility of thermal expansion-contraction mismatch and resultant degradation of the surface. Also, the increased strength of interfacial bonds between the filler and the matrix due to the better silane technology enables the filler to avoid being lost when the thermal cycle is repeated [17]. The

roughness advancement of the surface in this study is in agreement with the degradation processes that are known to be related to thermal cycling. Variations in temperature cause dissimilar thermal expansion of the organic resin matrix, together with inorganic filler particles and produce interfacial strains that reduce bond strength [18]. Such stresses mount up over repeated cycles, which may result in filler particle debonding, plasticization of the matrix and surface microcracking. The much smaller values of roughness in nanoceramic groupings indicate that there is resistance to these degradation routes and this may be due to a better compatibility of matrix-filler and lower coefficient of thermal expansion differentials [3, 4]. The roughness increase in the conventional composite of 273% per 10,000 cycles is significantly high as compared to the nanoceramic materials (100-133%), which goes along with the past study results of the susceptibility of traditional methacrylate-based composites to thermal stress [5]. Final R_a of conventional composite (0.41 μm) nears the threshold (0.2 μm) after which the bacterial retention can grow enormously

and, thus, increase the risk of caries and periodontal disease [6]. Conversely, Ra of all the nanoceramic materials was lower than 0.25 0mm and thus it was with clinically permissible limits of bacterial resistance. The stability of color results shows obvious benefits of nanoceramic hybrid materials and they all have a ΔE value less than the clinical acceptability threshold ($\Delta E < 3.3$) after thermal aging simulation of one year. The 3.76 value of the ΔE in the conventional composite would be noticeable to the common eyes and may require a visual fix or some sort of replacement [7, 8]. The yellowing pattern prevailing in all of the materials is well documented degradation that includes resin matrix oxidation, degradation of amine accelerator and formation of chromophores during thermal aging [9, 10]. Nanoceramic decreases the yellowing process associated with the decreased resin contents since the color changes mainly take place at the organic matrix stage. Thermocycling of water causes plasticization and chemical degradation of the resin network which are proportionally lower in highly filled material [11]. Also, there are others that use different resin chemistries, including ormocer technology in Admira Fusion, which is proven to have a higher hydrolytic stability than more standard dimethacrylate matrices [12]. The substantial positive correlation ($r=0.742$) between surface roughness and color change favors the mechanistic relationship between the two parameters. Heightened surface roughness forms micro-irregularities and enhanced surface area to absorb and entrap stains, leading to extrinsic discoloration alongside inherent colour change with matrix degradation [13, 14]. This correlation shows that it is necessary to have smooth surfaces to ensure maximum color stability which can be obtained by the choice of material and proper polishing guidelines. There are significant clinical implications of such findings. In cases where esthetics matters such as in anterior restorations, nanoceramic hybrid restorations have superior predictability in long-term color stability, which may prolong the life of a restoration and decrease the frequency of replacement. The enhanced surface stability can also be translated to a decrease in the plaque and a higher periodontal health in the restoration margins. Clinicians should, however, bear in mind that nanoceramic materials may need special finishing and polishing guidelines in order to obtain the best starting surface properties and that handling properties of materials can vary with the conventional materials that are well known [15]. A number of limitations should be mentioned. This in vitro experiment could not reproduce the complex dentin situation of masticatory wear, toothbrushing abrasive challenges, chemical exposures to food materials and enzymatic degradations by saliva. Although thermocycling is standardized to make comparisons, it is but one facet of clinical aging. The materials used in the study were tested in standardized conditions without paying attention to substrate effect and adhesive interfaces and cavity structures which affect the clinical performance. Only a single conventional composite was also used as control; the findings can be different according to various product formulations [16]. Also, Centon N like

materials showed superior colour stability compared with glass ionomer cements, suggesting that its resin-based matrix and enhanced filler composition provide greater resistance to thermally induced colour changes [17]. Further studies must integrate the application of combined aging measures such as mechanical loading, chemical testing and longer renal periods to enhance a more realistic clinical environment. Long-term follow-ups can only be achieved through clinical trials to confirm these laboratory results and come up with evidence-based guidelines on how to apply them in particular clinical situations [18].

Conclusion:

Nanoceramic hybrid restorative materials showed significantly lower surface roughness and superior colour stability after thermal cycling than conventional nanohybrid composites. These findings indicate better resistance to thermally induced surface degradation and improved aesthetic durability. Nanoceramic hybrids may therefore be preferred for long-term aesthetic restorations, particularly in the anterior region.

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