



## Comparative Evaluation Of The Effect Of Immersion And Thermocycling In Black Coffee And Diet Coke On Surface Roughness And Microhardness Of Nanofilled And Nanohybrid Composite Resins: - An In-Vitro Study

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### Abstract

**Context:** Limited data exists on the effects of thermocycling and immersion in different beverages on composite resin material. This study seeks to provide clinicians with evidence-based insights.

**Aim:** To Evaluate and Compare effect of immersion and thermocycling in black coffee and diet coke on surface roughness and microhardness of Nanofilled and Nanohybrid composite resin.

**Settings and Design:** In-vitro study carried out in laboratory with ideal conditions

**Methods and Material:** Sixty composite discs were prepared, thirty each from Nanohybrid(Group A) and Nanofilled composite resin(Group B) which further divided into six groups(n=10,5 each from Group A and Group B). Group I samples stored dry. Group II, III and IV stored at constant temperature of 37 °C in artificial saliva, Hot Black Coffee and Diet Coke respectively. Group V and VI thermocycled in hot black coffee(between 37 to 45°C) and diet coke(between 10 and 12°C) respectively. The surface roughness was measured using surface profilometer while microhardness using Vickers indenter.

**Statistical analysis used:** Overall intergroup comparison among three groups done using One-way Anova 'F test. Intragroup comparison in each experimental group done using unpaired t test.

**Results:** Surface roughness showed no significant differences between Nanohybrid(Group A) and Nanofilled composites(Group B), except in Diet Coke(Group IV)(p = 0.047) and after thermocycling in Diet Coke(Group VI)(p = 0.015). For microhardness, significant differences(p < 0.001) were observed between Groups A and B in all immersion media.

**Conclusions:** Within the boundaries of this research, the results revealed that the Nanofilled composite resin exhibited superior surface characteristics when subjected to these beverages than the Nanohybrid composite resin.

**Keywords:** Nanohybrid, Nanofilled, thermocycling

### Introduction

Composite resin restorations are a significant part of dentist's daily practice, driven by demand for aesthetic results.<sup>1</sup> These restorations must maintain color

stability, surface smoothness<sup>2</sup>, dimensional stability, marginal integrity, and wear resistance.

Modern Nano composites, with particle sizes between 0.1 µm and 100 nm, meet aesthetic needs in the

anterior mouth zone, offering excellent gloss and retention.<sup>1</sup> Available in Nanofilled and Nanohybrid types, their properties changes in the oral environment, particularly due to temperature fluctuations from different beverages.

Frequent exposure to beverages at varying temperatures may affect the durability of dental restorations. This study examines how simulated one-year aging, including immersion and thermocycling, impacts the surface roughness and microhardness of two composite resins with different microstructures.

### Subjects and Methods:

Sixty composite discs (Fig. No. 1) were prepared from both composite resins, 30 samples from Nanohybrid composite resin (Group A-Filtek Z250XT) (Fig. No. 4) and 30 samples from Nanofilled composite resin (Group B-Filtek Z350XT) (Fig. No. 3), using a Teflon mold with dimensions of 2 mm in thickness and 10 mm in diameter. After placing the composite resins into the molds, mylar strips and microscope glass slides were pressed onto the top surface of the resins using finger pressure (Fig. No. 5). Each sample was light-cured for 20 seconds through the mylar strip and glass slide using an LED curing unit (DentMark, India, 3000mw/cm<sup>2</sup>) on both the top and bottom surfaces (Fig. No. 6). The resulting samples, featuring a flat, smooth surface, were formed under pressure from the glass slide against the Mylar strip when sofex bur (Sof-Lex™ Contouring and Polishing Discs – 3M ESPE) was used (Fig. No. 7). The Sixty prepared discs further were divided equally into the six study groups (n=10) (Fig. No. 1) which comprised five samples each from Group A and Group B as follows:

Group I (Control): - (n=10) discs of composite resin kept dry (5 each from Group A and Group B)

Group II: - (n=10) discs kept in artificial saliva for constant temperature of 37 °C for 12 days. (Fig. No. 8-9)

Group III: - (n=10) discs of composite resin kept in black coffee for constant temperature of 37 °C for 12 days (Fig. No. 8-9).

Group IV: - (n=10) discs of composite resin kept in diet coke for constant temperature of 37 °C for 12 days (Fig. No. 8-9).

Group V: - (n=10) discs of composite resin thermocycled for 10,000 cycles (between 37 to 45°C) black coffee (Fig. No. 10).

Group VI: - (n=10) discs of composite resin thermocycled for 10,000 cycles (between 10 to 12°C) diet coke (Fig. No. 10).

Samples in Group III and Group IV were washed with distilled water, and the solutions were refreshed every 3 days to prevent bacterial or fungal contamination.<sup>3,4</sup> Samples in Group V and Group VI went through thermocycling for 10,000 cycles, with a dwell time of 60 seconds and a transfer time of 10 seconds, within a temperature range reflective of typical beverage consumption: 37 to 45 °C for black coffee and 10 to 12 °C for diet coke.

After immersion, the samples were re-tested for surface roughness and microhardness. The average surface roughness of the specimens was determined using a contact-mode surface profilometer (TR 220 Surface Roughness Tester, TIME Group, Pittsburgh, PA, USA) with a cut-off value of 0.8 mm and a range of 40 µm (Fig. No. 12). Three to four measurements were taken for each specimen, and the average surface roughness was calculated. The microhardness of each specimen was assessed using a diamond Vickers indenter with a 50 g load applied for 15 seconds (Fig. No. 11). Three indentations were made at different points on each specimen spaced 1 mm apart, and these measurements were averaged to produce a single Vickers Hardness Number (VHN).



## Results:

As shown in table 1. , Among all the experimental groups, no significant differences in surface roughness were observed between the Nanohybrid composite resin (Group A) and the Nanofilled composite resin (Group B) across various storage media, except for Group IV (storage in Diet Coke) ( $p = 0.047$ ) and Group VI (thermocycled in Diet Coke) ( $p = 0.015$ ). The samples in Group VI, thermocycled in Diet Coke, showed the highest surface roughness [0.170 (0.013)]. Samples stored in artificial saliva (Group II) ( $p = 0.651$ ) and black coffee (Group III) ( $p = 0.336$ ), as well as those thermocycled in black coffee (Group V) ( $p = 0.145$ ), did not exhibit significant differences within the groups. However, the most noticeable decline in surface roughness was observed in the Nanohybrid composites (Group A) compared to the Nanofilled composites (Group B).

For Group A, the values were Group VI (0.170 Ra)>Group IV (0.157 Ra)>Group V (0.149 Ra)>Group III (0.141 Ra)>Group II (0.132 Ra)>Group I (0.124 Ra).

For Group B, the values were as Group VI (0.148 Ra)>Group IV (0.138 Ra)>Group V (0.135 Ra)>Group III (0.132 Ra)>Group II (0.127 Ra)>Group I (0.121 Ra).

As shown in table 2., Amongst all experimental groups, the Microhardness values of Nanohybrid composite (Group A) and Nanofilled composite (Group B) had statistically significant differences ( $p < 0.001$ ) in all the immersion media. After immersion in beverages, the artificial saliva group (Group II) showed microhardness values higher than those of the other groups (Group III, IV, V and VI).

For Group A, the values were as Group I (56.16 VHN)>Group II (55.06 VHN)>Group III (53.84 VHN)>Group IV (51.08 VHN)>Group V (48.34 VHN)>Group VI (44.18 VHN).

For Group B the values were as Group I (66.58 VHN)>Group II (65.8 VHN)>Group III (64.97 VHN)>Group IV (64.22 VHN)>Group V (58.96 VHN)>Group VI (57.02 VHN).

## Discussion:

The durability of dental composite restorations largely depends on their resistance to degradation caused by exposure to food, plaque acids, and enzymes, which can soften the composite material.

While the mouth is the most accurate testing environment due to the complex and varied conditions within it, in-vitro models are vital for understanding the underlying mechanisms of biodegradation. It is well known that during eating and drinking, food and

beverages only make brief contact with tooth surfaces and restorations before being rinsed away by saliva. This study aimed to assess the surface roughness and microhardness changes of restorative materials, by stimulating an oral behaviour in an in-vitro situation. The effects of attrition from chewing habits however weren't assessed in the current study, the oral cavity being a complex environment.

The specimens were created with dimensions of 10 mm in diameter and 2 mm in thickness, as this is the most frequently used size according to Ergücü and Türkün.<sup>5</sup> Soflex is recognized for delivering the smoothest surface finish for composites.<sup>6</sup> Therefore, in this study, the resin composite specimens were finished and polished using coarse, medium, fine, and superfine disks, following the manufacturer's recommendations.

The diet coke was chosen as an erosive inductor because of its low PH, low calcium and fluoride concentrations and relatively lowers calorie count which is the reason it is highly preferred by youngsters nowadays. Based on data showing that the pH of oral fluids returned to neutral 1-3 min after one single sip of an acidic beverage<sup>7</sup>, a 5 min immersion in each cycle was thus chosen. If pH plummets to a value below the critical pH, salivary rate heightens, and the saliva dilutes the beverage.<sup>8</sup>

In recent times, there has been a significant spike in the consumption of black coffee by young adults who are becoming increasingly health-conscious, as it is low in calories and devoid of sugar and dairy, which is ideal for their dietary requirements. Black coffee also helps in increasing the metabolism and burning fat, thus, is the preferred choice for those that need to monitor or lose weight. The caffeine level also raises the level of adrenaline, making the body ready for intense exercise, hence many use it before exercise.

This research modelled one year of clinical service, with 12 days of soaking in the staining solution equalling one year of drink usage. Likewise, in vitro thermocycling for 10,000 cycles equals one year of clinical exposure.<sup>9,10</sup>

Thermocycling enhanced water sorption, which adversely influenced the mechanical behaviour of composites by promoting hydrolytic degradation and interfacial failure.<sup>11,12</sup> Thermocycling promoted the surface degradation of resin composites by creating

surface stresses and microcracks.<sup>13</sup> Therefore several such parameters, the organic matrix structure, the inorganic filler content and particle size, and the characterization tools utilized, influenced the surface roughness and microhardness.<sup>14</sup>

Bis-GMA took up less water than the resin made by triethylene glycol dimethacrylate (Soderholm et al., 1984).<sup>15</sup> Nanohybrid resin composite (Group A) comprised TEGDMA, while the leading Nanofilled resin composite (Group B) did not.<sup>16</sup> There is important role of water absorption<sup>17</sup> as the organic matrix of the hybrid composites is more hydrophilic, leading to increased hydrolysis and eventually morphological changes such as craters, cracks, and enhanced surface roughness.<sup>18</sup>

Furthermore, the size of the filler particles has also been correlated with the enhanced physical characteristics. Large filler particles have rougher surfaces in comparison to a smaller filler particle.<sup>19</sup> The resin composites used in this study were Filtek Z350 XT (Group B) which have a filler particle of an average size 0.005-0.02 microns, which is smaller compared to Filtek Z250 XT (Group A).

For this research, surface roughness was assessed with the profilometer, and no significant differences ( $P>0.05$ ) was seen within each storage medium except that of storage in diet coke and samples thermocycled in diet coke (Group IV and Group VI). The pH value range for both Diet Coke and black coffee, however, was quite close, only Diet Coke seemed to affect the roughness of the tested composite resin. Maybe it is because of the acids and sugars present that caused erosion of surface of composite resin.<sup>16</sup> The result of the present study is comparable to that recorded by Kitchens and Owens, who noted that the surface roughness of enamel was not increased after being immersed in coffee but increased after immersion in cola.<sup>20</sup> Results are also in conjunction with that noted by Guler et al. that acidic beverages significantly increase the surface roughness of resin composites.<sup>21</sup>

Test of microhardness is employed extensively in research as a polymerization marker. Parameters that influence the microhardness of restorative materials significantly include filler volume fraction, composition, type of resin, and the degree of polymerization.<sup>22</sup> Microhardness was determined for the materials prior to, as well as after soaking in the drinks using the Vickers hardness test. Immersion of



the composite resin specimens in all the drinks reduced the surface microhardness in the present study. The pH of the solutions used in this study were close approximations; artificial saliva: 7, black coffee: 5 to 6, diet coke: 1 to 3. But the greatest decrease in microhardness was seen in solution with lowest pH levels. Other secondary causes of the reduction in surface microhardness, after exposure to the beverages, could be extended loss of silica after matrix degradation, cracking of the matrix–filler interface, and subsurface damage. This decrease in hardness is due to the intraparticle decomposition of silane coupling agents and siliceous filler particles.<sup>23</sup> Hydroxyl ions are accumulated in this water, which attacks the siloxane bond to convert them into silanol groups resulting in degradation of filler surface.<sup>24</sup>

The results of this experiment conform to previous studies that demonstrated some food materials are responsible for the softening and enhanced wear of resin composites. Since conditions in vivo vary from conditions in vitro due to the presence of saliva, more in vivo investigations are needed to validate the outcomes of this research.

## Conclusion

Within the boundaries of this research, the results provided insights into the surface roughness and microhardness of Nanohybrid and Nanofilled composites after exposure to commonly consumed beverages in daily life. This study revealed that Nanofilled composite resin showed superior surface characteristics when subjected to these beverages than the Nanohybrid composite resin.

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**Table 1: Comparative statistics of surface roughness between Group A (Nano hybrid composite) and Group B (Nanofilled) respectively**

	Group A (Nanohybrid) Mean (SD)	Group B (Nanofilled) Mean (SD)	Unpaired t test	P value, Significance
Group I (Control)	0.126 (0.019)	0.13 (0.007)	t = -0.431	p =0.678 (NS)
Group II (Artificial Saliva)	0.124 (0.03)	0.19 (0.042)	t = -2.825	P =0.022*
Group III (Black Coffee – 37 <sup>0</sup> c)	0.136 (0.023)	0.114 (0.049)	t =0.897	P =0.396 (NS)
Group IV (Diet Coke-37 <sup>0</sup> c)	0.154 (0.04)	0.17 (0.057)	t =-0.512	P = 0.622 (NS)
Group V (Thermocycling- Black Coffee)	0.13 (0.025)	0.178 (0.054)	t = -1.796	P =0.110 (NS)
Group VI (Thermocycling- Diet Coke)	0.152 (0.028)	0.172 (0.062)	t = -0.650	P =0.534 (NS)

p &gt;0.05 – no significant difference

\*p&lt;0.05 – significant difference

**Table 2: Comparative statistics of microhardness between Group A (Nano hybrid composite) and Group B (Nanofilled) respectively**

	Group A (Nanohybrid) Mean (SD)	Group B (Nanofilled) Mean (SD)	Unpaired t test	P value, Significance
Group I (Control)	58.86 (6.32)	65.3 (2.98)	t = -2.058	p =0.074 (NS)
Group II (Artificial Saliva)	59.78 (8.14)	63.98 (2.01)	t = -1.118	P =0.296 (NS)
Group III (Black Coffee – 37 <sup>0</sup> c)	57.32 (4.58)	60.13 (2.16)	t = -1.241	P =0.250 (NS)
Group IV (Diet Coke-37 <sup>0</sup> c)	55.98 (5.64)	58.62 (3.77)	t = -0.869	P = 0.410 (NS)
Group V (Thermocycling- Black Coffee)	52.9 (5.96)	56.16 (2.82)	t = -1.105	P =0.301 (NS)
Group VI (Thermocycling- Diet Coke)	51.92 (4.96)	56.22 (2.38)	t = -1.745	P =0.119 (NS)

\*\*p&lt;0.001 – highly significant difference