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EFFECT OF DIFFERENT SHADES ON THE SURFACE ROUGHNESS OF RESIN NANO-CERAMIC: IN VITRO STUDY

Eman Ezzat Y. Hassanien *

ABSTRACT

Objective*:* Clinicians use different shades of restorative materials to mimic the natural teeth color. However, the effect of these shades on the restoration properties was unclear. Thus, the aim of the present study was to assess the effect of different shades of resin nano-ceramic on surface roughness before and after aging.

Materials and methods*:* Twenty rectangular specimens of 1±0.05 mm thickness were obtained from Lava Ultimate resin nano-ceramic blocks with two different shades (B1 and C2); n = 10. All specimens were polished and subjected to accelerated aging using autoclave for 6 hours. The surface roughness was assessed using digital microscope before and after aging. Surface morphology analysis was performed using a scanning electron microscope (SEM). After testing for normality, the collected data revealed parametric distribution and were expressed as mean and standard deviation (SD). Data were statistically analyzed using independent t-test and Paired t-test at a level of significance ($P \leq 0.05$).

Results: Specimens with C2 shade showed statistically insignificant higher surface roughness compared to B1 shade before and after aging. Aging caused significant changes in the surface roughness in both groups, with changes in the surface microstructure.

Conclusions: The shade of resin nano-ceramic does not affect the surface roughness. Aging represents an important factor that can affect surface roughness.

KEYWORDS: Aging, Autoclave, CAD/CAM, scanning electron microscope (SEM)

* Lecturer, Department of Fixed Prosthodontics, Faculty of Dentistry, Cairo University, Cairo, Egypt

INTRODUCTION

The increased esthetic demand together with the advancements in digital dentistry led to the introduction of many CAD/CAM materials with variety of properties to satisfy each clinical situation. One of the recent machinable materials introduced was Lava Ultimate; a resin nano-ceramic (RNC) that combined the advantages of dental ceramics and composite resins [1–3]. The manufacturer of this material aimed to achieve mechanical properties similar to those of natural teeth [4], which made it suitable for minimally invasive restorations; such as inlays, onlays, laminate veneers and recently occlusal veneers [5,6]. Hence, Lava Ultimate (LU) was believed to satisfy the biomimetic principles of maximum tissue preservation while providing optimal form, occlusion, and function [4,6].

LU possessed a unique composition containing up to 80 wt% nano-ceramic fillers embedded in polymer matrix [7–9]. The polymer matrix (20 wt%) comprised Bis-GMA (bisphenol A glycol dimethacrylate), UDMA (Urethane dimethacrylate), Bis-EMA (Ethoxylated bisphenol A dimethacrylate) and TEGDMA (Triethylene glycol dimethacrylate) [5,7,10,11]. However, the fillers consisted of 25 wt% nano-sized zirconia (4 - 11 nm diameter) and 55 wt% nano-sized silica (20 nm diameter) in the form of nanoparticles and nanoclusters [9]. The combination of silica and zirconia nanoparticles with zirconia-silica nano-agglomerates reduced the space between the particles [7,8] resulting in better physical properties compared to resin-based materials containing hybrid fillers and micro-fillers [9].

Compared to conventional ceramics, LU offered reduced brittleness, ease of intraoral repairability, better machinability with shorter milling time [10,12,13] and lower wear to the antagonist teeth [10,12] with wear properties comparable to that of enamel [9]. Furthermore, it didn't require post-milling firing, hence restorations can be completed in a single appointment with manual polishing [1]. It is supplied in many shades with two translucencies to accommodate to different clinical situations.

In the oral cavity, dental restorations are subjected to many factors [10]. Their effect is usually simulated in-vitro by various aging procedures; such as accelerated hydrothermal aging using an autoclave [13]. It was reported that 1 hour of autoclave treatment at 134°C can simulate 3–4 years of exposure to clinical environment [8,13]. Although autoclave treatment is commonly performed for aging zirconia rather than resins, it was employed for RNC by many studies [8,13], due to the fact that LU contained zirconia particles, hence, hydrothermal aging was expected to affect its mechanical properties along with its surface properties [8,13]. In fact some studies reported an improvement in the physical properties of LU after autoclaving [13].

Besides mechanical properties, surface roughness is an important property of dental materials [8]. Increased surface roughness is known to adversely affect esthetic, biological and mechanical long-term clinical success of a restoration and consequently patient satisfaction [1,10]. Generally, roughness affect brightness, gloss and color stability [12,14,15]. Increased roughness can increase surface susceptibility to staining and discoloration [1,10]. It can also increase the abrasiveness of the restoration, inducing more wear to the antagonist teeth [1]. In addition, rough surface increases bacterial adhesion and plaque retention with subsequent gingivitis, secondary caries and discoloration [1,10–12,16]. Also, the surface flaws presented in rough surfaces can be the starting point of material failure [12,17].

Thus, evaluating surface topography of restorative materials is an important issue [12]. Calculating the arithmetic mean of heights (Ra) is most commonly used in literature as an indicator for surface roughness [13,14]. Many studies have tested factors affecting surface roughness. Most researches focused on comparing different materials in relation to roughness parameters. However, information about the effect of different shades on surface roughness and subsequently on their long term success is lacking. Thus, the aim of the present study was to evaluate and compare the surface roughness of different shades of LU resin nano-ceramic before and after accelerated hydrothermal aging. The first null hypothesis was that different shades would have no effect on the surface roughness of resin nano-ceramic. The second null hypothesis was that the accelerated aging would have no effect on resin nano-ceramic surface roughness.

MATERIALS AND METHODS

Specimens' preparation

A total of 20 rectangular specimens were obtained from LU resin nano-ceramic CAD/CAM blocks (3M ESPE, St Paul, MN, USA). Two shades were tested; B1-LT (Group LB) and C2-LT (Group LC); n = 10. The rectangular specimens (1±0.05 mm thickness) were sectioned using a linear precision saw [2,3,8] (Isomet 4000; BUEHLER, Lake Bluff, IL, USA) at speed of 2500 rpm using a diamond disc of 0.5mm thickness under water coolant [1], to reduce heat generation. Specimens were then checked using magnifying lens for any surface defects. A precise digital caliper (Digital Vernier Caliper IP54, USA) was then used to measure the specimens at different points to verify their thickness. All specimens were polished using two-stage Sof-Lex Spiral polishing System Kit (3M ESPE, St Paul, MN, USA) [12,15] at low-speed and low pressure as was indicated by the manufacturer. A single experienced operator performed polishing for all specimens to minimize any human variation that might affect the outcome tested. All polished specimens were cleaned in a digital ultrasonic cleaner (CODYSON, Shenzhen, China) using distilled water for 10 minutes and air dried [1,8,11,15] to ensure the removal of any residues from the previous procedures. Each specimen was then placed in a small numbered sealed plastic bag until testing, to protect the specimens from any accidental scratches that might affect the outcome. Numbering the bags helped in blinding the assessors and statistician to the tested groups and allowed proper results documentation.

Pre-aging surface roughness assessment

A Digital Microscope [18,19] (Celestron Handheld Digital Microscope Pro, Torrance, USA) was used to capture images of the tested specimens at 60x magnification. All images were imported to ImageJ software (National institute of health, USA), which was used for image analysis and surface roughness determination without applying any filters. The software expressed the roughness parameter (Ra) in gray values. Three dimensional (3D) surface plots were also obtained using the same software.

Accelerated hydrothermal aging

All specimens in both groups were subjected to artificial accelerated hydrothermal aging using autoclave device at temperature of 134^{o}C and 2 bar (0.2Mpa) pressure [8,9] for six hours to simulate intra-oral aging of 18 years then air-dried for 24 hours [20].

Post-aging Surface roughness assessment

Surface roughness was assessed after aging in the same manner previously explained.

Scanning electron microscope (SEM) analysis

To assess the surface microstructure, one specimen from each group was arbitrarily selected and scanned before and after aging using a high resolution scanning electron microscope (SEM QUANTA FEG250, FEI Company, Netherlands). Images of the specimens were captured using 20kv energy at 600x, 1200x, 2500x, 5000x, 10,000x and 20,000x magnifications.

Statistical analysis

Statistical analysis was performed by an expert statistician, who was blinded to the tested groups, using statistical software (IBM SPSS Statistics, v24.0, IBM Corp, USA). Kolmogorov–Smirnov test was used to test for normality (P ≤ 0.05). All data were normally distributed and were expressed as mean and standard deviation (SD). Independent t-test was used to compare the two groups, whereas, Paired t-test was used to compare dependent values within each group before and after aging. The statistically significance value was set at P ≤ 0.05.

RESULTS

The mean and standard deviation (SD) values of surface roughness parameter (Ra); presented in **Table 1**, revealed that Group LB had statistically insignificant lower mean roughness value compared to Group LC. However, aging caused statistically significant decrease in the mean surface roughness in both groups. The 3D surface plots; presented in **Figure 1**, revealed that the surface roughness pattern of specimens before aging showed relatively increased peak heights than after aging in both groups.

SEM analysis confirmed the results of the roughness test (**Figure 2**). At low magnifications (600x, 1200x and 2500x), both groups showed more homogenous surface after aging compared to before aging, with the LB Group showing the most homogenous surface. Higher magnifications (5000x, 10,000x and 20,000x) showed a slight change in the grain size after aging especially in BL Group. Upon comparing the two shades, CL Group showed slight difference in the microstructure with some evident large particles (white patches seen at 600x, 1200x and 2500x magnifications), which were decreased in prevalence after aging.

TABLE (1) Surface roughness (Ra) values

Groups	Surface roughness values					
	Before aging		**After aging**		P-value	Mean difference
	Mean	SD	Mean	SD		
LB	1.23560	0.202917	0.95480	0.132992	0.002*	0.280800
LC	1.34870	0.087823	1.02380	0.149479	0.0004*	0.324900
P-value	0.123		0.290		---------	---------
Mean difference	-0.113100		-0.069000		---------	---------

*: indicates statistically significant differences (P ≤ 0.05)

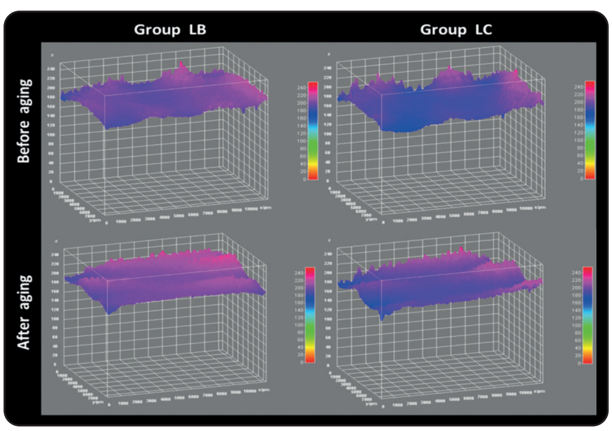

Fig. (1) 3D surface plot of the tested groups before and after aging

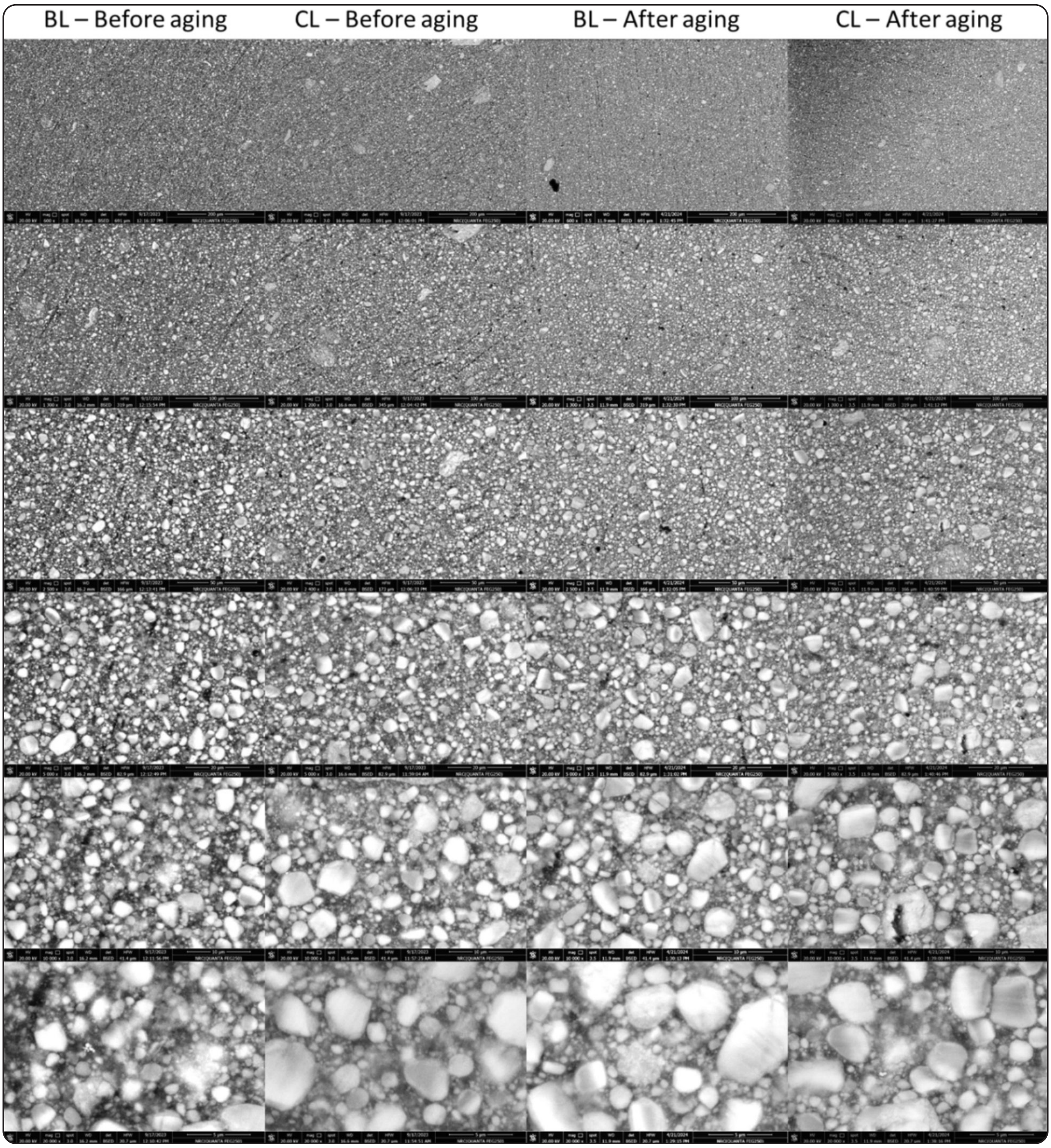

Fig. (2) SEM images of the tested groups before and after aging at 600x, 1200x and 2500x, 5000x, 10,000x and 20,000x magnifications

DISCUSSION

The first null hypothesis was accepted, where the tested shades did not significantly affect surface roughness. However, the results rejected the second null hypothesis, where aging procedure was found to significantly affect the surface roughness in both groups.

Many resin-matrix ceramics possess some inorganic pigments such as zirconium oxide, ferrous oxide and titanium dioxide in their composition that dictate their shade and translucency [21] to meet the increasing aesthetic demands [22]. Most commonly, researches test a single shade and apply the results to other shades across the brand without questioning a possible variation [22]. However, some variations are to be expected, especially with modern CAD/CAM materials.

To the author best knowledge, very little studies assessed the effect of different shades within the same material on its properties. Egilmez et al., 2018 [23] tested the effect of shade and thickness on surface roughness, color, translucency and surface gloss of resin nano-ceramic (LU), high-density composite resin and a polymer infiltrated hybrid ceramics; whereas, Li et al., 2022 [14] tested the shade and surface treatment effect on surface roughness, color and translucency of high-translucent self-glazed zirconia. However, Pop-Ciutrila et al., 2021 [3] tested the shade and thickness effect on the translucency of leucite-reinforced glass ceramic, zirconia-reinforced lithium silicate glass ceramic, conventional feldspathic ceramic and hybrid ceramic; while, Ilie, 2023 [22] tested its effect on elastoplastic and viscoelastic behavior of hybrid CAD/CAM resin-based composites. Thus, the present study aimed to assess the effect of shade on surface roughness of LU resin nano-ceramic material.

Shade B1 and C2 were tested to establish a clear assessment of the effect of light and dark shades of the material. The translucency in specimens of both groups was similar to eliminate its effect on the outcome measured. All specimens were polished only, as was recommended by the manufacturer. These recommendations might be due to the presence of high resin content in LU that might be damaged by sintering or glazing temperatures [17]. In addition, the zirconia particles exited in LU blocks are fully sintered [17].

Although many in-vitro studies were done using short-term intra-oral simulation, the artificial aging employed in the present study was performed using accelerated aging for 6 hours in an attempt to assess the long-term performance (18 years intra-oral simulation), in accordance with Egilmez et al., 2018 [8] and Yan et al., 2020 [9], who also performed long-term artificial aging when testing resin nano-ceramics; reaching up to 12 and 20 hours accelerated aging respectively.

Traditionally, contact or non-contact profilometers were used to assess surface roughness parameter quantitatively and qualitatively with high accuracy. However, the high expense of these devices stands as an obstacle in many researches. In the present study, the author aimed to use a digital microscope and open-source user-friendly image-processing software (ImageJ) to evaluate the surface roughness. Digital microscopes were used by Giugovaz et al., 2022 [18] and Hassan et al., 2022 [19] to assess the surface roughness. However, the employed software (ImageJ) was used for determining surface roughness, but in combination with different devices; such as Kim et al., 2017 [24], who used it to interpret confocal laser scanning microscope images, Pham and Vo, 2020 [25], who used it to analyze field emission scanning electronic microscope images, and Balderrama et al., 2021 [26], who used it in combination with scanning electron microscope to determine implant surface roughness. Although the technique presented in the current study in not as accurate as other conventionally used method such as profilometers [27], the values obtained were helpful in comparing the groups and predicting the surface properties.

SEM was also used in the present study to help analyze the surface topography and justify the results obtained by surface roughness test.

The present results showed that both tested shades had insignificantly different surface roughness before or after aging. This might be because both groups have similar nature. A similar result was shown by Li et al., 2022 [14], who found that different shades (A1 and A3) did not influence the surface roughness of high-translucent zirconia when being analyzed by non-contact 3D laser scanning microscope.

Although insignificant, Group LC showed higher roughness compared to Group LB. This might be attributed to the slight difference in microstructure between the tested shades as was evident in the SEM images. A similar result was also shown by Li et al., 2022 [14], who found that more chromatic shade (A3) of polished high-translucent self-glazed zirconia showed insignificantly higher Ra value compared to shade (A1).

However, there was a significant decrease in the surface roughness after artificial aging in both groups, which was also confirmed by the SEM. This result might be attributed to the chemical composition and the resin content in LU which affected its reaction to accelerated aging [17]. Yan et al., 2020 [9], suggested that high pressure and temperature induced by accelerated aging might cause an increase in crosslinking and polymerization of the resin matrix present in LU, which might affect its properties.

Our results came in agreement with Hamza et al., 2017 [17], who found that lava ultimate discs showed significantly lower surface roughness after accelerated aging by weathering process when using contact profilometer, where they recorded 0.6219 μm ±0.28367 for aged specimens and 0.9371 μm ±0.33515 for non-aged specimens. Our results partially agreed with Egilmez et al., 2018[8], who found that the surface roughness of LU after accelerated autoclave aging (69.90 nm ±5.48) were insignificantly lower than non-aged specimens (74.17 nm ±15.44) when using coherence scanning interferometry. The difference in values significance might be due to applying more aging time (12 hours) compared to the aging time applied in the present study (6 hours). However, a study conducted by Al-Harbi et al., 2017 [28] showed that LU surface roughness insignificantly decreased after 3 and 6 months water storage and insignificantly increased after thermocycling, the difference in the results might be due to applying different aging procedures compared to the present study.

The values recorded in the present study cannot be compared to the critical threshold value for bacterial retention stated in most studies (0.2 μm) [8], because the nature of the captured images and the measuring unit in the present study were different from those researches.

Limitations of the present study included the inability to mimic the effect of saliva or different beverages, cyclic loading and testing only two shades of the material.

Further studies are recommended to investigate the effect of different shades on color stability and flexural strength of LU. Testing the effect of different shades in other ceramic materials is also recommended.

CONCLUSIONS

- Different shades of LU do not adversely affect its surface roughness before or after aging.
- Artificial accelerated aging can significantly affect the surface roughness of LU resin nano-ceramic.

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