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Research Article

Surface gloss, gloss retention, and color stability of 2 nano-filled universal resin composites

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Conflict of Interest
No potential conflict of interest relevant to this article was reported.

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ABSTRACT

Objectives: This study compared the surface gloss (SG), gloss retention (GR), and color stability (CS) of 2 universal resin composites after chemical (CA) and mechanical (MA) aging.
Materials and Methods: Twenty disc-shaped samples of G-ænial A´Chord (GC-Europe) and Filtek Universal (3M-ESPE) were polished with sequential abrasive papers. For CA, specimens were stored in 1 mL of 75% ethanol for 15 days at 37°C, and readings (SG, GR, and CS) were obtained at baseline and 5, 10, and 15 days. For MA, specimens were subjected to 10,750 simulated brushing cycles. SG and CS were evaluated after every 3,583 cycles. SG was measured with a glossmeter (geometrical configuration: 60°), and values were expressed in gloss units. Color was measured with a spectrophotometer using the CIE-L*a*b* color system. The Student's *t*-test, 1-way analysis of variance, and Scheffé test were used for statistical analysis ($\alpha = 0.05$).
Results: G-ænial presented significantly higher SG values than Filtek ($p = 0.02$), with GR reductions of 5.2% (CA) and 5.3% (MA) for G-ænial and 7.6% (CA) and 7.2% (MA) for Filtek. The aging protocol had no statistically significant effect on SG or GR ($p = 0.25$) from baseline to the final readings. G-ænial–MA presented the lowest color difference ($\Delta E = 1.8$), and G-ænial–CA and Filtek–CA had the largest changes ($\Delta E = 8.6$ and $\Delta E = 11.8$, respectively).
Conclusion: G-ænial presented higher SG values and better CS. Both restorative materials demonstrated acceptable GR and CS. Aging protocols impacted these properties negatively.

Keywords: Optical properties; Color stability; Surface gloss; Gloss retention; Composite resins

INTRODUCTION

Light-cured resin composites are currently the first choice among clinicians for direct restorations in anterior and posterior teeth. Not only the need for anatomic and functional rehabilitation, but also the esthetic requirements for such restorations, have pushed manufacturers to continuously enhance the mechanical and optical properties of these materials to simulate characteristics of natural teeth and to achieve long-lasting clinical results [1].

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Surface gloss, gloss retention, and color stability are important features that reflect the optical properties of resin composites, which may be affected by degradation in the oral environment, leading to the need for repolishing, repairing, or restoration replacement [2-4].

With the advent of nanotechnology in materials science, the quality of resin composite restorations has improved. The initial surface gloss has been enhanced, mainly due to the polishable particle size and higher percentage per volume of nanofillers in the resin matrix. However, gloss retention and discoloration remain a matter of concern in long-term clinical studies [2-5]. Filler particle-related features, such as the concentration, size of the filler reinforcement, and resin formulation are known factors that may affect the surface of composites, thus eventually leading to staining of the material [5].

The physical properties of universal resin composites for anterior/posterior use must be balanced to fulfill both mechanical and aesthetic requirements, in order to simplify the selection of restorative materials that serve various purposes in a dental office [1]. Two recent resin composites, manufactured by leading companies, were selected for comparison regarding color and surface gloss variation. These restorative materials were launched with the claim that they have improved esthetic properties for anterior-posterior universal restorations.

The present study aimed to evaluate the effect of artificial aging in a laboratory setting on the above-mentioned surface optical properties. For that purpose, 2 universal resin composites enhanced with nanotechnology were subjected to chemical and mechanical aging to simulate clinical service.

Two null hypotheses were proposed for this study: 1) neither type of artificial aging (chemical or mechanical) will affect the optical properties of the selected universal resin composites; and 2) there will be no differences in color stability and gloss retention between the 2 universal resin composites evaluated.

MATERIALS AND METHODS

Preparation of the samples

The 2 selected composites were G-ænial A´Chord (GC Europe, Leuven, Belgium), a nanohybrid resin composite, and Filtek Universal (3M-ESPE, St. Paul, MN, USA), a nano-filled resin composite. **Table 1** provides a description of their compositions. Twenty samples of each composite were produced for each test (gloss retention and color stability) and for each aging group (chemical and mechanical), using a stainless-steel mold 6 mm in diameter × 1 mm in thickness. The specimens were light-cured for 40 seconds using a light-emitting diode device with an output of 1,400 mW/cm^2 (Silverlight, GC America, Alsip, IL, USA) and polished with

Table 1. Description of the 2 resin composites tested

Material	Shade	Batch #	Composition			Manufacturer
			Matrix	Filler	Initiator	
G-ænial A´Chord	A2	2007301	UDMA, Bis-MEPP, TEGDMA	Silicon dioxide, strontium glass, pigment	Additives, stabilizers, catalysts	GC Europe, Leuven, Belgium
Filtek Universal	A2	NC31404	UDMA, Bis-GMA, PEGDMA, Bis-EMA, TEGDMA	Silica, zirconia		3M-ESPE, St. Paul, MN, USA

UDMA, urethane dimethacrylate; Bis-MEPP, bismethacrylic acid isopropylidenebis(p-phenyleneoxyethylene) ester; TEGDMA, triethylene glycol dimethacrylate; Bis-GMA, bisphenol A glycol dimethacrylate; PEGDMA, polyethyleneglycol dimethacrylate; Bis-EMA, ethoxylated bisphenol A-dimethacrylate.

sequential abrasive papers (600 to 1,200 grit – MP-2 Grinder Polisher; ABO Lab, Facultad de Odontología - UNC, Córdoba, Argentina).

Four subgroups were established: G-ænial A´Chord, chemical aging (A-CA), G-ænial A´Chord, mechanical aging (A-MA), Filtek Universal, chemical aging (B-CA), and Filtek Universal, mechanical aging (B-MA). The baseline color and surface gloss readings (R0) were performed after the specimens' fabrication and polishing. Two independent readings were conducted in intermediate phases during chemical and mechanical aging (R1 and R2), and the final reading (R3) was performed at the end of each aging protocol.

Aging of the samples

1. Chemical aging (CA)

Specimens were stored for 24 hours in distilled water in a dark, light-proof container at room temperature and then immersed separately in 1 mL of a 75% ethanol solution for 15 days. Samples were removed every 5 days, thoroughly rinsed under tap water, and subjected to 10 strokes of brushing using a soft-grade toothbrush, and then dried gently with extra-smooth paper napkins. The ethanol solution was changed after each reading. Surface gloss and color stability readings were performed at baseline and at 5, 10, and 15 days.

2. Mechanical aging (MA)

Specimens were subjected to simulated brushing using specific equipment (wear simulation by sequential toothbrushing; INTI, Córdoba, Argentina). A toothpaste suspension was prepared by mixing 6 mL of distilled water with 6 g of toothpaste (Colgate Total 12, RDA 70 µm; Colgate-Palmolive, Sao Paulo, Brazil), and a soft toothbrush was used during the experiments (Colgate Slim-soft; Colgate-Palmolive). Brushing cycles consisted of a 3.8 cm amplitude with a 200 g weight, totaling 10,750 cycles. The aging was carried out at a controlled temperature of 37°C. After every 3,583 cycles, the surface gloss and color stability were assessed. The toothbrush and toothpaste suspension were changed after each reading period (R1, R2, and R3).

Assessment methods

1. Color stability

Color measurements were obtained using a spectrophotometer (CM-600D; Konica Minolta Sensing Inc., Osaka, Japan) and all measurements were replicated 3 times. The mean value of the recordings was considered the final value for each specimen. Before color testing, the spectrophotometer was calibrated with the specified calibration plate. The CIE-L*a*b* color system, which is defined as a 3-dimensional measurement system, was applied to interpret the readings: "L" indicates the brightness, "a" the red-green proportion, and "b" the yellow-blue proportion of color. Specimens were aged chemically and mechanically, as described above.

The obtained values were automatically stored digitally by a computer connected to the spectrophotometer. One-way analysis of variance (ANOVA) was used to assess and compare the difference in the color stability of the different composites. Specific color coordinate differences (ΔL, Δa, Δb) were calculated for the following intervals: R0 (baseline)-R1, R1-R2, R2-R3, and R0-R3.

Total color differences (ΔE) were calculated using the following formula:

$$\Delta E = \{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2\}1/2$$

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2. Gloss retention

Readings were performed at baseline and at 5, 10 and 15 days for the chemical aging group, and at baseline and every 3,583 cycles until 10,750 cycles were reached for the mechanical aging group, using a glossmeter device (ETB6-6 Glossmeter; Shanghai Total Industrial Co., Ltd., Sanghai China) with a 60° geometrical configuration (light incidence). Values were expressed in gloss units (GU). Three randomized measurements were performed for each sample during each reading, and the average of these measurements was used for statistical analysis.

Although surface roughness was not a variable considered for evaluation in the present study, illustrative images were obtained from representative samples of each subgroup at every reading period using a confocal laser scanning microscope (Olympus LEXT OLS4000, LAMARX; Facultad de Astronomía, Matemática y Física, Universidad Nacional de Córdoba, Córdoba, Argentina) at ×200 magnification to check the appearance of the surfaces after being subjected to the polishing and aging protocols (**Appendix 1**).

Statistical analysis

Data were entered for analysis using SPSS 19 software (IBM SPSS Inc, Chicago, IL, USA). The Student's *t*-test was used to determine whether significant differences between the 2 materials for each aging subgroup occurred. One-way ANOVA was used to analyze color differences, surface gloss and gloss retention among the 4 subgroups. The complementary Scheffé test was used for a deeper analysis of the statistical differences among the 4 subgroups ($\alpha = 0.05$).

RESULTS

Color stability

Table 2 summarizes the color differences (ΔE) for the 2 materials between readings (R0-R1, R1-R2, and R2-R3), and from baseline (R0) to the final reading (R3) for each aging protocol using the CIE L*a*b* assessment tool. A negative effect on color stability of artificial aging was observed, being more evident for the chemical protocol than for mechanical aging. The greatest color differences were identified between R2 and R3 in the chemical aging group, both for G-ænial A´Chord and Filtek Universal.

Between R0 and R1, color differences were restricted to values within a range of 2.8 to 4.5, with a reduction of these values for the 4 subgroups between R1 and R2. For both subgroups subjected to chemical aging, color difference values increased significantly between R2 and R3; this did not occur in the mechanical aging subgroups, which maintained a ΔE of < 2. It is important to highlight that the average human eye is not able to notice values of $\Delta E < 2.5$ [6].

Table 2. Color differences for each material and aging protocol between readings and from baseline to final reading

Subgroups	N	ΔE 0-1	ΔE 1-2	ΔE 2-3	ΔE 0-3
A-MA	20	2.8 ± 1.3	1.5 ± 0.6	2.2 ± 1.0	1.8 ± 0.4[a]
A-CA	20	3.6 ± 1.0	2.1 ± 0.8	5.6 ± 2.2	8.6 ± 2.5[b]
B-MA	20	4.5 ± 2.2	3.3 ± 2.3	2.1 ± 1.1	1.3 ± 0.6[a]
B-CA	20	3.6 ± 0.9	1.6 ± 0.7	9.3 ± 4.6	11.8 ± 4.3[c]

ΔE expressed as mean ± standard deviation for each material and aging protocol.
B-MA, Filtek Universal, mechanical aging; A-MA, G-ænial A´Chord, mechanical aging; A-CA, G-ænial A´Chord, chemical aging; B-CA, Filtek Universal, chemical aging.
Different letters express significant differences among groups (Scheffé test $p \leq 0.05$).

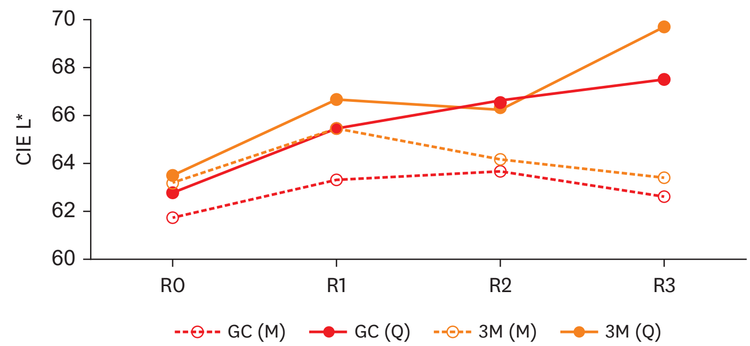

Figure 1. Changes in the L* coordinate (lightness) for each subgroup at different reading periods.
GC, G-ænial A´Chord; 3M, Filtek Universal; (M), mechanical aging; (Q), chemical aging; R, readings 0 (baseline), 1, 2 and 3.

In a general description of each group, it was possible to observe that the A-MA subgroup presented the lowest variations between readings, with a final ΔE0-3 of 1.8, whereas the greatest variations were found in the A-CA and B-CA subgroups, especially between the R2 and R3 periods. These 2 subgroups reached final mean ΔE0-3 values of 8.6 and 11.8, respectively. The B-MA subgroup showed a high variation between R0 and R1, although the final change in color was the lowest (ΔE0-3 = 1.3).

The mean differences between R0 and R3 were used to evaluate the overall ΔE for each subgroup. The Student's *t*-test showed significant differences between both materials using each aging protocol. Chemical aging showed a greater effect on the color stability (ΔE0-3) of both composites than mechanical aging.

When different parameters of CIE L*a*b* were evaluated separately, it was possible to identify the variations in each of these coordinates and how they were related to changes in the color of the resin composites. **Figure 1** shows the variations in L* (lightness), which showed a replication of the same color change patterns that were described for the ΔE results calculated for each subgroup.

Regarding the CIE a* and b* coordinates, a slight increase of yellowness (+b) at R1 in the subgroups subjected to chemical aging was noted (**Figure 2**), but for the R3 outcome, the change was quite large, shifting the shades of the composites to red/orange tones (+a/+b).

Gloss retention

Table 3 shows the mean values and respective standard deviations of the surface gloss values of each subgroup. G-ænial A´Chord showed higher mean surface gloss values than Filtek Universal, irrespective of the aging protocol, for each reading period ($p < 0.01$). In these 2 materials, there was a reduction in gloss retention from baseline (R0) to the final reading (R3), either with chemical or mechanical aging protocol.

Table 3. Surface gloss values for the 4 subgroups in each reading period

Subgroup	N	R0	R1	R2	R3
A-MA	20	97.2 ± 2.3	94.6 ± 2.3	93.6 ± 2.2	92.0 ± 2.2
A-CA	20	98.4 ± 1.8	95.7 ± 1.7	94.1 ± 1.6	93.3 ± 1.6
B-MA	20	87.4 ± 2.3	84.6 ± 2.2	83.1 ± 1.9	81.1 ± 2.6
B-CA	20	86.9 ± 2.8	84.5 ± 2.5	82.7 ± 2.5	80.3 ± 2.4

Surface gloss values expressed in gloss units (GU) as mean ± standard deviation.
B-MA, Filtek Universal, mechanical aging; A-MA, G-ænial A´Chord, mechanical aging; A-CA, G-ænial A´Chord, chemical aging; B-CA, Filtek Universal, chemical aging.

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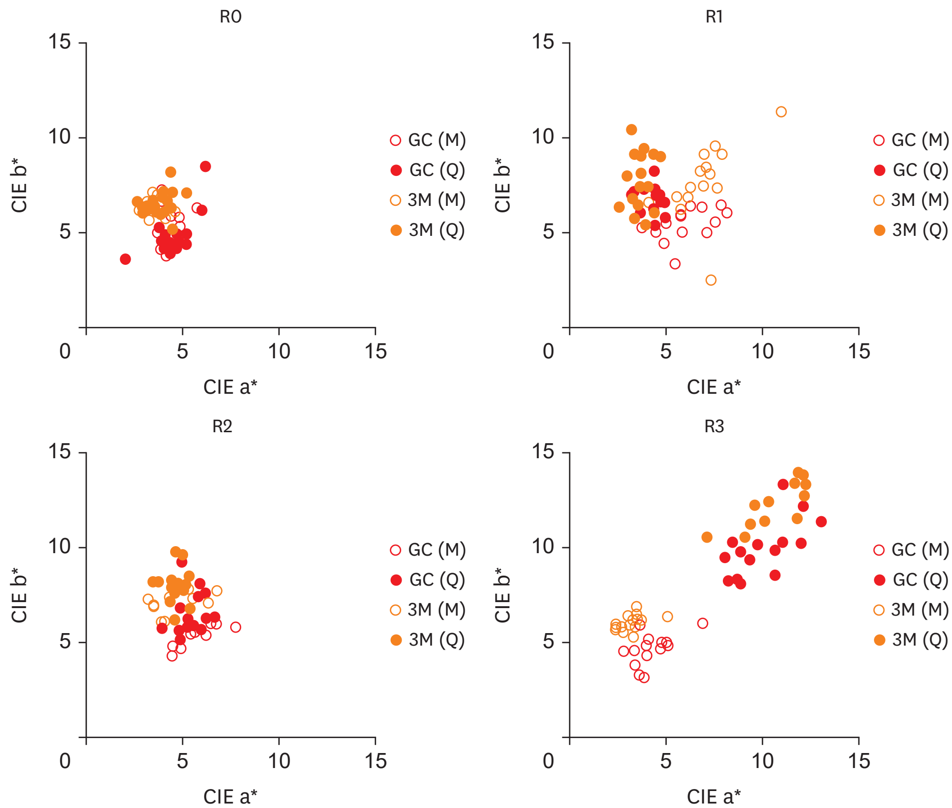

Figure 2. Changes in the a* and b* coordinates in the 4 subgroups at each reading period.
GC: G-ænial A´Chord; 3M: Filtek Universal; (M): mechanical aging; (Q): chemical aging; R: readings 0 (baseline), 1, 2 and 3.

Gloss retention, measured from baseline (R0) to the final reading (R3) was higher in G-ænial A´Chord than in Filtek Universal for both aging protocols. **Table 4** shows the percentage loss of gloss between the various reading periods and from baseline to the final reading in the 4 subgroups. Conversely to what was observed in the evaluation of color stability, there was no statistically significant effect of the aging protocol on the surface gloss or gloss retention of both resin composites ($p = 0.25$). (**Figure 3**)

A comparison among the 4 groups using 1-way ANOVA analysis complemented by the Scheffé test ($\alpha = 0.05$) identified 2 significantly different groups (G-ænial A´Chord, subjected to either chemical or mechanical aging; this material exhibited the least reduction of surface gloss at the final readings).

Table 4. Comparison of gloss retention between the 2 materials, measured as the percentage of reduction of gloss for the 4 subgroups

Aging protocol	Material	R0-R1	R1-R2	R2-R3	R0-R3
Mechanical	G-ænial A´Chord	2.6%	1.1%	1.6%	5.3%
	Filtek Universal	3.1%	1.7%	2.3%	7.2%
P		0.32	0.19	0.09	0.03
Chemical	G-ænial A´Chord	2.7%	1.6%	0.8%	5.2%
	Filtek Universal	2.8%	2.1%	2.8%	7.6%
P		0.99	0.11	0.17	0.01

Mean percentage of gloss reduction for each subgroup between readings (R) and from baseline (R0) to final reading (R3). The Student's *t*-test was used to calculate *p* values, corrected with the Scheffé test for $\alpha = 0.05$.

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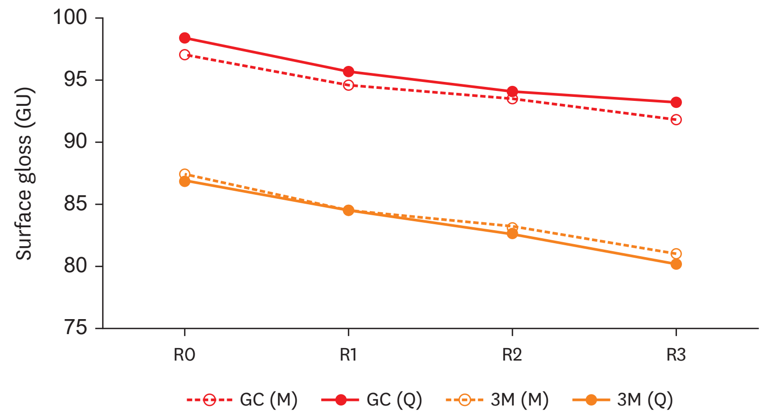

Figure 3. Surface gloss expressed in gloss units (GU) for each subgroup at different reading periods.
GC, G-ænial A´Chord; 3M, Filtek Universal; (M), mechanical aging; (Q), chemical aging; R, readings 0 (baseline), 1, 2 and 3.

DISCUSSION

The selection of resin composites for this evaluation was restricted to 2 contemporary universal resin composites that have been introduced to the market with claims that these materials have undergone innovations that improve their respective optical properties. Although this comparison could have been extended to other restorative materials within the scope of composites modified with nanotechnology, the aim of the study was to evaluate the effect of 2 aging protocols that are likely to affect optical properties, with the aim to test the veracity of the claimed innovations. The comparison between these materials was not aimed to demonstrate whether one material was better than the other, but to observe whether either test protocol was able to produce significant changes in color and surface gloss, thus testing the improvements claimed by the respective manufacturers. Some of the material improvements remain confidential under patented formulae, and discussions based on chemical modifications of their components would be a matter of hypothetical deduction and speculation. The optical properties of resin composites may be affected either by the chemical formulations of their respective organic phases or by the size, shape, and quantity of the filler particles. Moreover, other variables such as the curing source or polishing and aging protocols have been identified to have an influence on surface gloss, gloss retention, and color stability [7,8].

In the present laboratory study, these external variables were standardized to test 2 universal composites and to compare their performance under a controlled environment. Therefore, the methodology used by Rocha *et al.* [9] was replicated in terms of the preparation of samples and aging protocols to have a guiding reference to reduce any potential testing bias. One difficulty that needed to be well controlled was the calibration of the assessment instruments to obtain the most accurate and reliable values in comparison to the mentioned study and to comply with the ISO/TR 28642:2016 standards for color measurement.

There is a strong correlation between surface roughness and color stability/gloss retention, which was assumed when the study was designed [10]. A confocal laser scanning microscope was used to observe and describe the sample surfaces aiming to identify any potential defects that could have been interpreted as the cause of variations in color or gloss. The confocal images showed similar surface appearances for all subgroups, with few voids, but numerous scratches remaining from the abrasive papers.

This calls attention to the importance of assessing and comparing the surface roughness of the composites, and the use of alternative polishing protocols [11]. Once established protocols are used, the analysis of roughness may be added to establish or reject correlations between roughness and color change. Although nanohybrid resin composites present less surface roughness after polishing than microhybrid composites, the effect of the polishing system may influence the retention of optical properties when the material is subjected to chemical or mechanical challenges [12]. Smooth and glossy surfaces are desirable for long-term esthetic restorations and the final surface polish of resin composite restorations could be affected by many variables, as described above, as well as the finishing/polishing (F/P) system used [13,14]. In that sense, the baseline gloss values of Grandio and Filtek 350 XT (76.6 ± 4.7 and 73.9 ± 4.9 GU) with Mylar strips were similar to the initial values obtained in the present study [13]. Another study also recorded high gloss values for Filtek 350 XT (99.9 ± 1.2 GU) using mylar strips for the surface finish. That study concluded that gloss was affected by filler size distribution, refractive index of fillers, viscosity and refractive index of the resin matrix [15].

The perceptibility and acceptability thresholds used to interpret the tolerance of the variations observed in the present study can also be considered a limitation in terms of a gray area for discussion. Perceptibility and acceptability tolerance thresholds need to be discussed to establish which differences in color or surface gloss may significantly affect the esthetic outcome of a dental restoration [6,16]. The wide variability of values depends upon the evaluators' subjective criteria, the assessment tools, and the systems of coordinates that are used for evaluation. The acceptability and perceptibility limits for surface gloss have been reported within a range of 6.4 and 35.7 GU, respectively [17]. In the present study, regardless of this wide spectrum, gloss retention and color stability values of both evaluated restorative materials were within the range of tolerance, reflecting an acceptable performance, compatible with their marketing claims.

Regarding chemical aging, it has been reported that differences in inorganic fillers influence the diffusion of aqueous solutions and ethanol within the resin bulk, resulting in different aging patterns [18]. Irrespective of the 2 different materials evaluated, the combined influence of the inorganic phase and the aging agent penetration did not appear to have a significant impact on the initial readings [19]. However, color differences were observed when the samples were subjected to chemical aging for a long period, probably due to the degradation caused by the chemical agent of the organic matrix of both test materials. In a study reporting changes in the mechanical properties of resin composites caused by fluid sorption, a significant change was detected in aging protocols when ethanol was used [20]. In that study, along with the organic matrix degradation, the degradation of the ester bonds and the silane coupling agent were also discussed as being responsible for the deterioration in strength [20]. According to the manufacturers´ specifications, the main differences in the matrix composition are the presence of bisphenol groups (bisphenol-A glycidyl dimethacrylate [Bis-GMA] and ethoxylated bisphenol-A dimethacrylate [Bis-EMA]) and polyethyleneglycol dimethacrylate (PEGDMA) in Filtek Universal instead of bismethacrylic acid isopropylidenebis(p-phenyleneoxyethylene) ester (Bis-MEPP) in G-ænial A´Chord.

As an improvement of nano-filled resin composites, nanohybrid materials contain a range of different sizes of macrofillers and microfillers, which also include nanometer-sized fillers (1–100 nm) that occupy the spaces between larger particles, leading to reduced spacing among particles [21]. However, it has been demonstrated that composites with smaller filler sizes do not necessarily present low surface roughness and discoloration, implying that staining may depend upon monomer structure, as well as surface irregularities [14,21]. Monomer release may have an impact on color stability, and seemed to have led to reduced color stability in direct nanohybrid and nano-filled resin composites compared to indirect resin-based materials, irrespective of the staining agents and the F/P systems [22].

A study reported that resin immersion in ethanol resulted in polymer softening due to polymer swelling and the subsequent weakening of the polymeric chains' cohesive forces [23]. In that study, a lower cross-link density resulted in greater surface degradation. Although light-curing was standardized in the current study, differences in the cross-link density of different resin composites associated with different possible patterns of ethanol infiltration might explain the present results, in which both tested resin composites presented the greatest gloss alterations after the first aging period [23,24].

In the mechanical aging groups, the pattern of surface gloss reduction was similar between both resin composites. Although the initial polishing procedures were standardized, it is known that resin wear depends on the inorganic and organic components [25,26]. Conversely, the results of gloss retention obtained after toothbrushing simulation in a study that evaluated indirect resin composite samples showed that surface gloss values remained above 80 GU with less than a 10% reduction after 10,000 cycles [10]. In that study, the baseline surface gloss values started above 80 GU but drastically dropped by more than 50% after 5,000 cycles and 75% after 10,000 [10].

The results described in the current study concur with those obtained by Gurgan *et al.* [27] for the color stability of Filtek Universal and G-ænial A´chord. Those authors also found that G-ænial A´chord presented excellent optical properties, which are features supported by the improved polishing properties of these nano-hybrid composite resins [27].

Brushing produces microscopic and macroscopic surface alterations, with an impact on the reflection of the incident light, thus reducing surface gloss [28]. The present gloss reduction from the initial reading until the last cycles agrees with other studies that have shown a correlation between wear and progressively increased roughness and an increased number of cycles for simulated brushing. It is important to highlight that the same toothpaste was used in the present study as in the study of Rocha *et al.* [9], with an average particle size close to 70 µm.

Unlike the results obtained in that study, brushing resulted in a similar percentage of gloss reduction compared to the effect of ethanol immersion for both resin composites. Although it is believed that surface polishing is strongly related to surface gloss, the present data show that other aging protocols, such as ethanol immersion, might have a greater influence on surface gloss [26]. Moreover, mechanical aging did not affect color stability as much as chemical aging did.

CONCLUSION

According to the results obtained in the present study, the null hypotheses were partially rejected, as these 2 universal restorative materials demonstrated acceptable retention of surface gloss and color stability after the aging protocols were applied, although both protocols impacted the evaluated optical properties negatively. Nonetheless, G-ænial A´Chord presented higher surface gloss values and better color stability than Filtek Universal.

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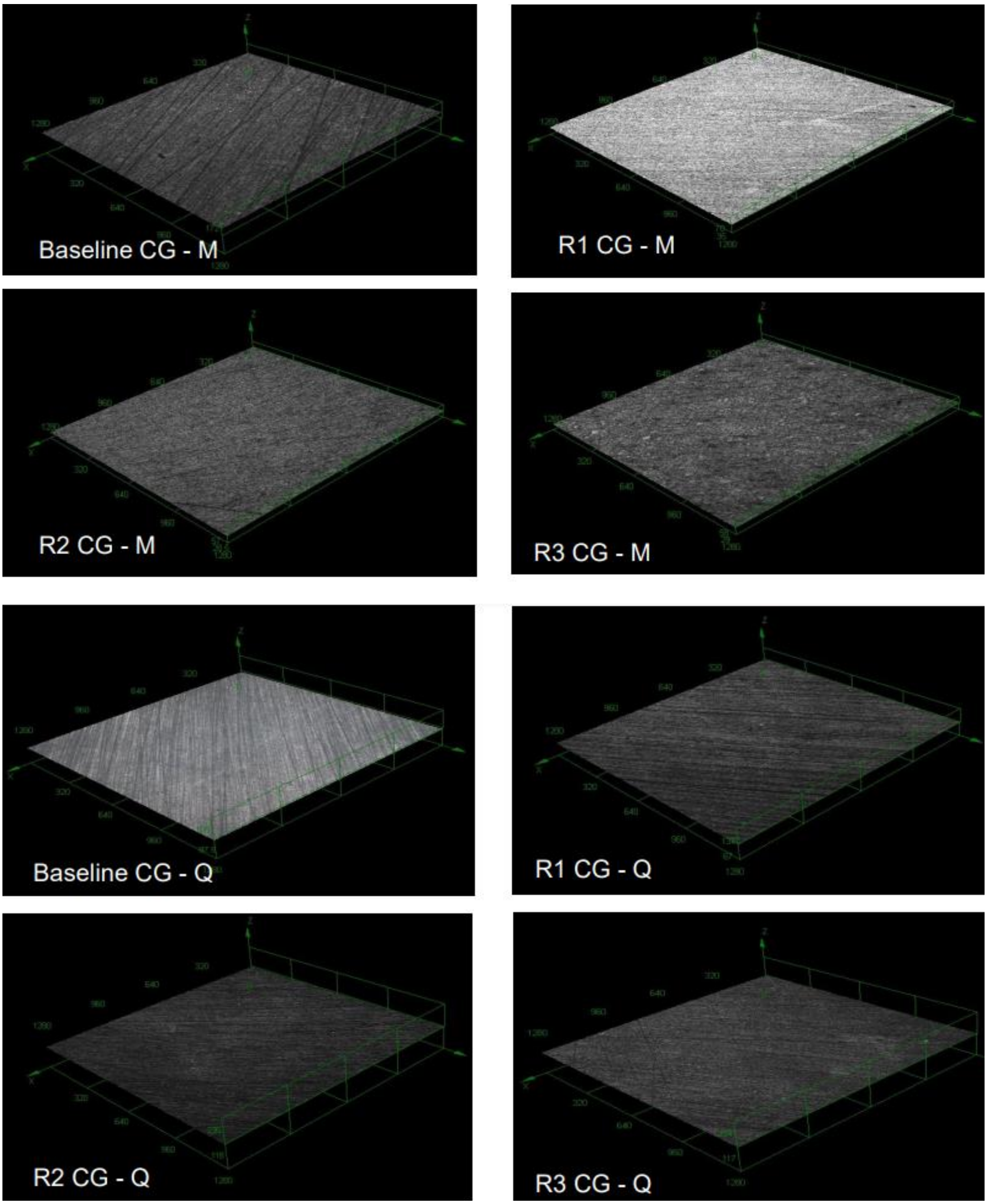
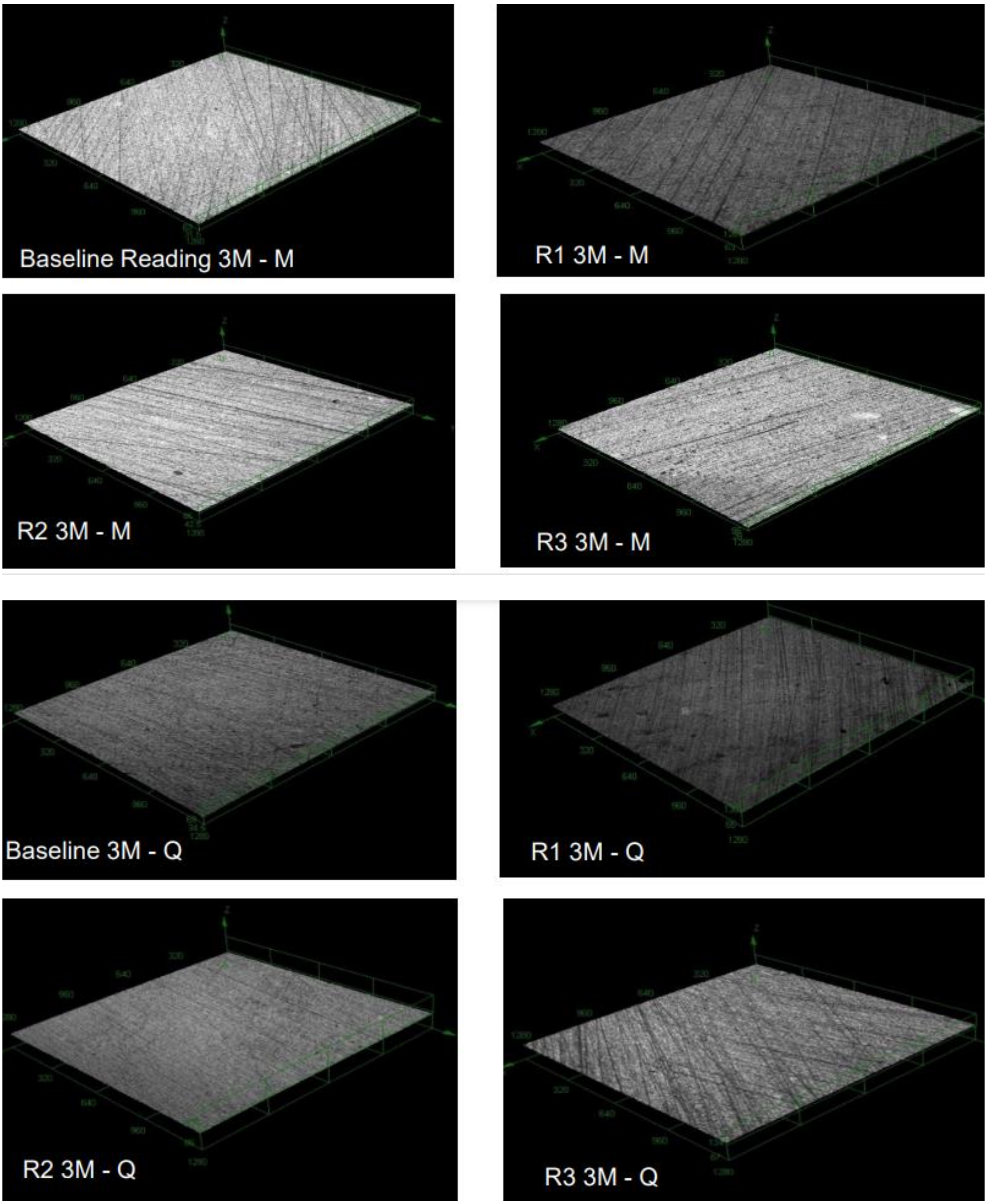
Appendix 1. Images from the two resin composites at each reading period for the two ageing protocols obtained by means of a Confocal Laser Scanning Microscope (CLSM)



(continued to the next page)

Appendix 1. (Continued) Images from the two resin composites at each reading period for the two ageing protocols obtained by means of a Confocal Laser Scanning Microscope (CLSM)



GC, G-ænial A´Chord; 3M, Filtek Universal; (M), Mechanical ageing; (Q), Chemical ageing; R, readings 0 (baseline), 1, 2 and 3.

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