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Physicomechanical Properties and Morphological Characterization of Several Universal Resin Composites After Different Aging Procedures

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ABSTRACT

Objectives: To evaluate flexural strength, elastic modulus, microhardness, and microstructure of several universal resin composites before and after artificial aging. In addition, filler characterization, water sorption, solubility, and degree of conversion were analyzed.

Methods: Five universal composites, Essentia Universal (EU), Filtek Universal (FU), G-ænial A'Chord (GA), Omnichroma (OM), and Tetric Prime (TP) were compared to a conventional composite, Filtek Supreme XTE (FS) used as a control. Flexural strength, elastic modulus, microhardness, and microstructure were analyzed at baseline and after three aging procedures: (1) Immersion in artificial saliva at 37°C for 2 weeks; (2) the same immersion followed by 10,000 thermocycles (5°C–55°C); and (3) the previous procedure with an additional 48-h storage in 75% ethanol at 37°C. SEM characterization of filler particles, water sorption, solubility, and degree of conversion were also assessed.

Results: FU showed the highest flexural strength and elastic modulus. Thermocycling reduced the flexural strength and elastic modulus of all composites except the elastic modulus of FU. FS was the hardest composite and EU the softest. FU, OM, and TP exhibited lower water sorption than FS. EU, GA, and OM exhibited lower solubility than FS. A similar degree of conversion was found for all universal composites.

Conclusion: Differences in mechanical behavior depended on the resin composite tested and the aging treatment.

1 | Introduction

Resin composites are the material of choice for direct dental restorations due to their high versatility and excellent mechanical and esthetic properties. Moreover, they can be bonded to hard dental tissues, allowing maximum preservation of tooth structure [1, 2]. Their improvement has been so profound, from an esthetic standpoint, that reproducing the challenging optical behavior of human enamel and dentin may be realistically achieved by combining composite masses with different shades

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and opacities [3]. However, this multilayer technique is a difficult process that demands a skilled operator and implies a longer chair time [4].

In recent years, all efforts have been directed toward the development of composite materials with an increasingly simplified clinical use. Accordingly, self-adhesive resin composites that do not require the prior application of an adhesive [5, 6], as well as bulk-fill composites that can be applied in one increment, have been launched [6, 7]. In a more recent development, newest resin composites that have fewer color shades can be placed with a nonlayering technique to mimic the esthetic properties of the surrounding dental tissues [8, 9]. These simplified resin composites are known as universal composites [5, 10]. They are available as (1) "grouped-shaded" materials that cover all VITA classical shades with fewer shades; (2) "cloud-shade" materials that include just three shades (light, medium and dark); and (3) "one shade," "single shade," or "monochromatic" composites, designed to mimic all the classical VITA shades [10–12], exhibit a color adjustment potential, also known as "chameleon effect" [9, 13]. This color adjustment potential in restorations is produced by the reciprocal color reflection between the dental tissues and the resin composite [14]. Therefore, resin composite brand, filler size, and content, as well as translucency, all influence the color adjustment potential. [8, 9, 13, 15–17]

Furthermore, characteristics of the cavity preparation and surrounding dental tissues, such as size and depth of the cavity, enamel margin configuration [10, 15, 16, 18, 19] and enamel prism orientation [18] have a relevant effect on resin composite color adjustment potential [20–23].

One-shade universal resin composites can be pigmentary or nonpigmentary. The method of color creation in nonpigmentary universal composites is innovative, as it is not only induced by selective light absorption via pigments, as usual in dental materials, but also by selective light reflection via particularized nanostructures [24]. This structural coloration can be obtained by means of uniform spherical nanofillers with a diameter smaller than the wavelength of visible light (approximately under 380 nm) [24, 25] that can provide natural color matching with hard dental tissues [26, 27].

Along with these modifications in the filler, some manufacturers have removed bisphenol A-glycidyl methacrylate (Bis-GMA) or bisphenol A ethoxylate dimethacrylate (Bis-EMA) from the composition of their universal resin composites due to public concern about the toxicity of bisphenol A (BPA) [28]. Bis-GMA is more rigid than other monomers, such as UDMA and TEGDMA, which may also lead to a decreased degree of conversion (DC) and crosslink density [29–31]. These changes in the matrix composition, as well as those in the filler to obtain the "chameleon effect" in universal resin composites, may also modify their mechanical behavior [32, 33] and may have potential implications in both short- and long-term performance [11, 34, 35]. Since cyclic chewing loads and temperature changes in a moist oral environment impact the long-term success of dental restorations [36], studies analyzing the esthetic properties of universal composites [8, 12, 37] should be complemented with mechanical behavior evaluations, particularly, after accelerated aging [28]. A combined hydrolytic and thermal degradation may cause filler debonding [38–41], while water plasticization further compromises their mechanical and physical properties [42].

Within these mechanical properties, flexural strength, and elastic modulus stand out as they have been correlated with wear resistance and marginal integrity of restorations [43, 44]. In addition, variations in filler shape and distribution may significantly influence the elastic modulus even among composites with a similar filler content [43, 45]. Other properties, such as surface hardness, have been proposed as an indirect predictor of wear, although this correlation remains weak [45, 46]. Finally, the degree of monomer conversion may influence [47] water sorption, solubility [42, 48, 49], and biocompatibility [50, 51], with a less straightforward impact on mechanical properties in certain composite formulations [52].

Accordingly, the aims of the present in vitro study were to characterize the mechanical behavior and microstructure of several universal resin composites in comparison with a conventional multishade nanofilled composite, and to analyze the effect of three artificial aging procedures on those properties (artificial saliva, thermocycling, and ethanol storage) [38–42].

Moreover, the filler component of these materials was characterized, and sorption, solubility, and DC were also evaluated. Therefore, the null hypotheses were that universal composites exhibit physicomechanical properties that are not different from those of a conventional nanofilled composite, and these properties are not affected by aging.

2 | Materials and Methods

Five universal resin composites were evaluated: Essentia Universal (EU, GC, Tokyo, Japan), Filtek Universal (FU, Solventum, St. Paul, MN, USA; shade A2), G-ænial A'Chord (GA, GC; shade A2), Omnichroma (OM, Tokuyama Dental, Tokyo, Japan), and Tetric Prime (TP, Ivoclar Vivadent, Schaan, Liechtenstein; shade A2). A nanofilled composite, Filtek Supreme XTE (FS, Solventum; shade Body A2), was also analyzed as a control. The composition of all the resin composites tested is detailed in Table 1, according to manufacturers and previous reports [53–55].

2.1 | Physicomechanical Properties

Specimens of each resin composite were prepared for flexural strength, elastic modulus, and microhardness testing as follows.

2.1.1 | Flexural Strength and Elastic Modulus

A three-point bending test was conducted according to ISO 4049:2019 (Dentistry-Polymer-based restorative materials, International Organization for Standardization) [56] and the Academy of Dental Materials guideline [57]. Ten specimens (2 mm wide × 2 mm high × 25 mm long) of each composite were made using a stainless-steel mold with two metal plates (Odeme

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TABLE 1 | Composition of the resin composites analyzed.

Resin composites (manufacturer) (abbreviation)	Type	Shade	Resin matrix	Particle size/content (wt%/vol%)	Batch no.
Essentia Universal (GC) (EU) [30, 31]	Microhybrid	Universal	UDMA, TCD, Bis-EMA Bis-GMA	PPF (17 μm): strontium glass (400 nm), lanthanide fluoride (100 nm), fumed silica (16 nm) FAISi glass (850 nm) 81 wt%/–	230522D
Filtek Universal[a] (Solventum) (FU)	Nanofilled	A2	AUDMA, FM, diurethane-DMA and 1,12-dodecane-DMA	Nonagglomerated particles (20 nm SiO_2, 4–11 nm ZrO_2). Agglomerate particles (20 nm SiO_2, 4–11 nm ZrO_2) filled with ErF_3 consisting of 100 nm agglomerated particles 76.5 wt%/58.4 vol%	9671807
Gænial A'Chord (GC) (GA) [32]	Nanohybrid	A2	UDMA, TEGDMA, Bis-EMA	Glass-filler (300 nm barium glass; 16 nm fumed silica) 82 wt%/–	230307A
Omnichroma (Tokuyama Dental) (OM) [31]	Nanofilled	Universal	Bis-GMA, TEGDMA	Uniform-sized supra-nano spherical filler (260 nm spherical SiO_2-ZrO_2) 79 wt%/68 vol%	113E53
Tetric Prime[a] (Ivoclar Vivadent) (TP)	Nanohybrid	A2	Bis-GMA, UDMA, Bis-EMA, D3MA	PPF (40 nm and 3 μm): barium glass, ytterbium trifluoride, mixed oxide (SiO_2/ZrO_2), and copolymers 76–77 wt%/54–56 vol%	Z057W6
Filtek Supreme XTE[a,b] (Solventum) (FS)	Nanofilled	A2 Body	Bis-GMA, UDMA, TEGDMA and Bis-EMA	Nonagglomerated particles (20 nm SiO_2, 4–11 nm ZrO_2). Particle agglomerate particles (20 nm SiO_2, 4 to 11 nm ZrO_2) Average particle size of 0.6–10 μm in dentin, body and enamel and 0.6–20 μm in translucent 72.5 wt%/55.6 vol% for translucent ones and 78.5 wt%/63.3 vol% for the rest	10026233

Abbreviations: 1,12-dodecane-DMA: 1,12-dodecane dimethacrylate; AUDMA: aromatic urethane dimethacrylate; Bis-EMA: bisphenol A-diglycidyl; Bis-GMA: bisphenol A-glycidyl methacrylate; D3MA: decanediol 1,10-dimethacrylate; Diurethane-DMA: diurethane-dimethacrylate; TCD: tricyclodecane dimethanol dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; UDMA: urethane-dimethacrylate; PPF: pre-polymerized filler.
[a]Composition of the composites provided by the respective manufacturer.
[b]Also available as Filtek Supreme Ultra and Filtek Z350 XT in other areas of the world.

Dental Research, Luzerna, SC, Brazil). Resin composites were inserted in the mold in one increment, covered with a Mylar strip, pressed with a microscope slide to remove the excess, and photopolymerized with five overlapping points (10 s each) with a SmartLite Pro LED curing light (Dentsply Sirona, Charlotte, NC, USA) with a power density of 1250 mW/cm^2. All specimens were then manually dry-polished with 600-grit silicon carbide paper discs (Buehler-Met II, Buehler, Lake Bluff, IL, USA) and stored for 24 h at 37°C in distilled water. Specimens were then subjected to a three-point flexural strength test in a universal testing machine (Instron 3345, Instron Co., Canton, MA, USA) at a crosshead speed of 0.5 mm/min until fracture to determine flexural strength (MPa) and elastic modulus (GPa) at baseline. The outcomes were calculated using the following equations:

$$\text{Flexural strength} = \frac{3FL}{2bh2.}$$

where F is the maximum load exerted on the specimen in Newtons (N), L is the distance between the supports (mm), b is the width, and h is the height, both in mm.

$$\text{Elastic modulus} = \frac{L3F}{4wh3d}$$

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where L is the support length (mm), F is the maximum load in N, w is the specimen width (mm), h is the specimen height, and d is the deflection (mm) recorded at F load [58].

2.1.2 | Microhardness

Five circular specimens of each composite were prepared in the same way using prefabricated Teflon molds (6 mm in diameter × 1.5 mm in thickness). The specimens were light cured for 20 s on the top surface without overlapping points, and with the same LED device. They were then dry polished on the side where irradiation was carried out using silicon carbide paper polishing discs of decreasing abrasiveness (600-, 1000-, 4000-grit) and, finally, with 1 μm alumina polishing paste (MetaDi, Buehler) with a polishing cloth. Specimens were polished with each disc or cloth for approximately 30 s. The samples were immersed in an ultrasonic bath with distilled water for 5 min and kept dry at 37°C for 24 h. Microhardness (Vickers Hardness Number, VHN) of each specimen at baseline was measured using a Vickers indenter (Buehler 2101) applied at 100 g of load and 30 s dwell time, ensuring a penetration depth of less than 10% of the sample thickness [59]. Five indentations were recorded on the top of each specimen. The mean Vickers hardness number was calculated and considered for statistical analysis, as a parameter that may predict abrasion as an indirect measure of the DC of resin composites [46, 60, 61]. The time elapsed from each test to the measurement of its hardness value was sufficient to guarantee complete relaxation of the material.

2.2 | Observation Under Scanning Electron Microscopy (SEM)

2.2.1 | Morphological Analysis of Resin Composite Microstructure

Eight circular samples of each composite were also prepared, as previously described for microhardness testing, to be observed under SEM. Surfaces were etched during 30 s with 37% phosphoric acid (Total Etch, Ivoclar Vivadent), washed with water for 30 s, and air-dried for the same time. Specimens were observed under SEM (XL30 ESEM Philips, Amsterdam, Netherlands) at 15 kV accelerating voltage with a working distance of 8 mm. Representative images were obtained at ×500, ×1000, ×2500, ×5000, and ×10,000 magnification.

2.2.2 | Characterization of Filler Particles

Two specimens of 1 g of each resin composite were dissolved twice in acetone and centrifuged using a variation of a method previously described [62] to eliminate the resinous components until the supernatant became transparent. After drying in a vacuum chamber for 24 h, the residue was mounted on sandblasted aluminum stubs with carbon tape and conductive carbon glue (Ted Pella Inc., Redding, CA, USA) and left in a vacuum desiccator for at least 24 h. Specimens were sputter-coated with Pt, observed under a Hitachi FESEM (Hitachi Ltd., Chiyoda City, Tokyo, Japan), and the inorganic particles analyzed. Micrographs for each composite resin were exposed at magnifications of ×10,000, ×40,000, and ×60,000. ImageJ software (NIH, Bethesda, MD, USA) was used to measure the filler particles.

2.3 | Aging Procedures

Resin composites were tested for flexural strength and elastic modulus ($n = 10$), microhardness ($n = 5$), and SEM morphological analysis ($n = 2$) before and after being submitted to each aging treatment [41] as follows:

1. Storage in artificial saliva (pH: 6.9; 1000 mL: 1.2 g potassium chloride, 0.84 g sodium chloride, 0.26 g dipotassium phosphate, 0.14 g calcium chloride dihydrate) for 2 weeks at 37°C, as it has been considered the medium that best mimics the oral environmental conditions [41, 43, 63].
2. Storage in artificial saliva as described before, followed by thermocycling (10,000 cycles in distilled water, 5°C–55°C). The thermocycling process combines hydrolytic and thermal degradation, which is believed to produce debonding of the fillers [38–41].
3. Same as treatment 2 followed by storage in a mixture of ethanol (75 vol%; ethanol absolute, VWR International, Rosny-sous-Bois, France) and distilled water (25 vol%) at 37°C for 48 h. Ethanol penetrates and expands the polymer network, removing the unreacted, linear polymers and oligomers before breaking up their chains [64–66].

2.4 | Sorption and Solubility

Five discs from each composite resin were prepared as explained above. They were polymerized on both sides for 20 s following the ISO 4049 instructions [56] and kept dry at 37°C for 15 min. Therewith, they were removed from the mold and dry-polished with 1000-grit silicon carbide abrasive paper to eliminate irregularities. Specimens were weighed every 24 h with an analytical balance (GRAM FS, GRAM Precision S.L., Barcelona, Spain) until a constant mass m_1 was achieved, that is, until the mass loss of each specimen was less than 0.1 mg every 24 h. This usually took approximately 2–3 weeks. After a constant mass (m_1) was achieved, the volume (v) in mm^3 of each specimen was calculated according to their dimensions measured by a digital caliper (Mitutoyo Europe GmbH, Neuss, Germany). The specimens were then placed in distilled water at 37°C for 7 days, then washed with water and air-dried for 15 s. After being removed from the water, specimens were weighed for 1 min, and their mass recorded as m_2. The specimens were reconditioned in the desiccator in the same manner as m_1 until a constant mass (m_3) was achieved. Water sorption (W_{sp}) in μg/mm^3 was calculated using the following equation:

$$W_{sp} = \frac{m_2 - m_3}{v}$$

where m_2 corresponds to the mass of the specimen in μg after immersion in water for 7 days, m_3 represents the mass of the reconditioned sample in μg, and v is the volume of the specimen

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in mm^3 [58]; the solubility (W_{sl}) in $\mu g/mm^3$ was calculated using the following equation:

$$W_{sl} = \frac{m_1 - m_3}{v}$$

where m_1 is the conditioned mass in μg before immersion in water, m_3 represents the mass of the reconditioned sample in μg, and v is the volume of the specimen in mm^3.

2.5 | Degree of Conversion

DC was assessed using a Fourier transform infrared (FTIR) spectrometer (FT/IR-4600, JASCO, Tokyo, Japan) equipped with an attenuated total reflectance (ATR) accessory with a diamond crystal. Spectra from nonphotopolymerized and photopolymerized samples of each resin composite ($n = 3$) were collected within the range of 400-4000 cm^{-1} with a resolution of 2 cm^{-1} and 124 scans. Nonphotopolymerized material spectra were obtained after depositing approximately a 1.5 mm- thick increment on the ATR crystal. For photopolymerized sample preparation, a cylindrical nylon mold (5 mm in diameter, 1.5 mm in thickness) was slightly overfilled with an increment of nonpolymerized material, ensuring that air entrapment was minimized. Transparent polyester strips were used to prevent the formation of an air-inhibited layer. Glass slides were firmly pressed on the top and bottom of the mold to obtain flat resin composite surfaces. The material was light-cured from the top for 20 s using the Smart Lite Pro LED curing light (Dentsply Sirona), positioned in contact with the glass.

Each sample was immediately placed with its irradiated surface on the ATR crystal for spectra acquisition of the cured material. Spectra analysis was performed using Peakfit v4.12 (Systat Software, Chicago, IL, USA). A smoothing of 10% (Savitzky–Golay algorithm) and a Gaussian–Lorentzian mixed function were applied to fit curves. After using a standard baseline technique, the height of selected peaks was measured depending on the composite resin: internal standard (IS) aromatic carbon double bond (1532 cm^{-1} for OM, 1599 cm^{-1} for FU, and over 1608 cm^{-1} for GA, EU, TP, and FS) and methacrylate (aliphatic carbon double bond; 1637 cm^{-1}). DC was calculated by comparing the peak heights of the uncured (non-polymerized) and cured (polymerized) samples of each material, according to the following formula:

$$DC\,(\%) = \left[1 - \frac{\frac{1637\ cm^{-1}}{IS}\,cured}{\frac{1637\ cm^{-1}}{IS}\,uncured}\right] \times 100$$

2.6 | Statistical Analysis

Flexural strength, elastic modulus, and microhardness results did not follow a normal distribution ($p < 0.05$), confirmed by the Shapiro–Wilk test, nor variance homoscedasticity, after Levene's test ($p < 0.001$). Therefore, they were analyzed using Kruskal–Wallis with Bonferroni correction for multiple tests, and the Friedman test for related samples for two-way analysis of variance by ranks. In contrast, a normal distribution for water sorption and solubility, and DC data was confirmed, as well as homoscedasticity ($p > 0.05$). Therefore, results were analyzed by one-way ANOVA and post hoc comparisons using Tukey's test. All statistical tests were performed at a preset alpha of 0.05 using the IBM SPSS Statistics 27 package (IBM Corporation, Armonk, NY, USA).

3 | Results

3.1 | Physicomechanical Properties

3.1.1 | Flexural Strength and Elastic Modulus

Mean flexural strength and elastic modulus of all composite resins are shown in Tables 2 and 3, respectively, along with their corresponding standard deviations. Significant differences were detected for both properties ($p < 0.001$) at baseline and after the three aging procedures.

At baseline, FU yielded the statistically highest flexural strength. FS resulted in statistically lower flexural strength than FU, but statistically higher than all the remaining resin composites. TP resulted in statistically similar flexural strength compared to GA, but higher than those of EU and OM. The lowest flexural strength was measured for OM, which was not statistically different from that of EU.

After storage in artificial saliva for 2 weeks, FU resulted in higher flexural strength than all the other resin composites ($p < 0.001$). There was no statistical difference among the flexural strength of EU, GA, OM, TP, and FS.

Thermocycling had no effect on the trend observed at baseline, as the flexural strength of FU remained the highest while OM recorded the lowest. Similar intermediate flexural strengths were obtained for FS and TP ($p < 0.001$). The flexural strengths obtained after storage in ethanol were statistically similar for all the composites except for FU, which maintained the statistically highest flexural strength ($p < 0.001$).

Flexural strength of all resin composites was affected by aging procedures ($p = 0.003$ for FU and $p < 0.001$ for the remainder). Storage in artificial saliva for 2 weeks had no impact on the results, unlike thermocycling, which significantly reduced the flexural strengths of all materials except for FU. Thermocycling followed by storage in ethanol produced a significant decrease in the flexural strength of FS, FU, OM, and TP.

The Kruskal–Wallis test revealed statistically significant differences for the elastic moduli of the resin composites at baseline and after each aging procedure ($p < 0.001$). The elastic moduli at baseline were statistically higher for FU and FS. TP, GA, and EU showed similar intermediate elastic moduli, while OM yielded the lowest elastic modulus, which was not statistically different from that of EU. After 2-week storage in artificial saliva, FS yielded a statistically higher elastic modulus than all the other resin composites. FU and GA fell upon the intermediate statistical rank ($p < 0.001$). EU, TP, and OM obtained the lowest elastic moduli. When specimens were thermocycled, FS and FU obtained the highest elastic moduli

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TABLE 2 | Mean flexural strength in MPa and (standard deviation) of resin composites at baseline and after different aging procedures.

	Baseline	2 weeks in artificial saliva	Thermocycling	Thermocycling + Ethanol	
Resin composite	**Mean (SD)**	**Mean (sd)**	**Mean (sd)**	**Mean (sd)**	***p***
Essentia Universal	73.1 (4.5)^{de2}	79.7 (7.0)^{b1}	39.2 (8.0)^{cd3}	33.7 (6.5)^{b3}	< 0.001
Filtek Universal	129.1 (17.5)^{a1}	118.7 (4.9)^{a1}	109.4 (15.2)^{a1}	93.0 (20.0)^{a2}	0.003
G-ænial A'Chord	81.8 (17.5)^{cd1}	74.3 (12.1)^{b1}	45.8 (14.0)^{bcd2}	39.0 (9.9)^{b2}	< 0.001
Omnichroma	69.9 (18.3)^{e1}	71.8 (25.2)^{b1}	38.0 (7.2)^{d2}	33.6 (4.5)^{b3}	< 0.001
Tetric Prime	83.1 (6.6)^{c1}	78.8 (12.2)^{b1}	52.0 (8.9)^{bc2}	40.0 (3.2)^{b3}	< 0.001
Filtek Supreme XTE	108.0 (12.4)^{b1}	96.8 (23.3)^{b1}	54.4 (13.3)^{b2}	36.1 (6.8)^{b3}	< 0.001
p	< 0.001	< 0.001	< 0.001	< 0.001	

Note: Different lowercase letters indicate statistically significant differences among resin composites at baseline and after each aging procedure (per columns) according to the Kruskal–Wallis test ($p < 0.05$). Different numbers indicate statistically significant differences among aging procedures for each resin composite (per row) according to the Friedman test ($p < 0.05$).

TABLE 3 | Mean elastic modulus in GPa and (standard deviation) of resin composites at baseline and after different aging procedures.

	Baseline	2 weeks in artificial saliva	Thermocycling	Thermocycling + Ethanol	
Resin composite	**Mean (SD)**	**Mean (SD)**	**Mean (SD)**	**Mean (SD)**	***p***
Essentia Universal	4.9 (0.8)^{bc1}	4.7 (0.5)^{c1}	4.5 (0.9)^{b1,2}	3.5 (0.9)^{bc2}	0.008
Filtek Universal	8.07 (1.5)^{a1}	6.6 (0.3)^{b1}	7.1 (0.7)^{a1}	6.9 (1.7)^{a1}	> 0.05
G-ænial A'Chord	5.5 (1.0) ^{b1,2}	6.1 (1.4)^{b1}	4.8 (0.9)^{b2}	4.5 (1.1)^{b2}	0.012
Omnichroma	4.4 (1.1)^{c1}	4.4 (1.6)^{c1}	4.5 (1.0)^{b1}	3.2 (0.7)^{c2}	0.007
Tetric Prime	5.5 (0.9)^{b1}	4.4 (0.9)^{c1,2}	5.1 (1.0)^{b1}	3.6 (0.8)^{bc2}	0.001
Filtek Supreme XTE	8.6 (1.0)^{a1}	8.2 (1.7)^{a1}	7.2 (1.6)^{a1}	5.9 (0.7)^{a2}	< 0.001
p	< 0.001	< 0.001	< 0.001	< 0.001	

Note: Different lowercase letters indicate statistically significant differences among resin composites at baseline and after each aging procedure (per columns) according to the Kruskal–Wallis test ($p < 0.05$). Different numbers indicate statistically significant differences among aging procedures for each resin composite (per row) according to the Friedman test ($p < 0.05$).

in comparison with EU, GA, OM, and TP ($p < 0.001$). After ethanol storage, FS and FU yielded again the highest statistical mean values, followed by GA, EU, and TP. OM attained the lowest mean elastic modulus, although not statistically different from those of EU and TP ($p < 0.001$).

Similar to flexural strength, sequential aging influenced the elastic modulus of all resin composites with the exception of FU (*p*-values are detailed in Table 3). Storage in saliva for 2 weeks had no impact on elastic modulus, while thermocycling reduced the mean elastic modulus of GA. However, when storage in saliva was followed by thermocycling and ethanol storage, EU, GA, OM, and FS exhibited a significant reduction in mean elastic modulus compared with the respective means at baseline and after saliva storage. Elastic modulus remained constant for FU ($p > 0.05$).

3.1.2 | Microhardness

Results from the microhardness analysis are displayed in Table 4. Statistically significant differences among resin composite microhardness were detected at baseline and after sequential aging ($p < 0.001$).

The multi-shade resin composite control, FS, registered the highest mean microhardness at baseline and after all the aging procedures, while EU resulted in the lowest mean microhardness. For the remaining universal composites, TP resulted in statistically higher mean microhardness at baseline than the other materials. FU and GA resulted in statistically similar mean microhardness, while OM was ranked in the lowest statistical subset. After 2 weeks of immersion in saliva, FU and TP resulted in statistically higher mean microhardness than GA and OM. When specimens were thermocycled, FU attained statistically higher mean microhardness than the other universal composites. GA obtained statistically higher mean values than OM and TP. After ethanol storage, the mean microhardness of FU was again statistically higher than that of GA, OM, and TP. These three universal composite resins attained statistically similar mean microhardness.

The microhardness of all resin composites tested underwent modifications after the sequential aging procedures

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TABLE 4 | Mean microhardness (VHN) and (standard deviation) as function of resin composites at baseline and after different aging procedures.

	Baseline	2 weeks in artificial saliva	Thermocycling	Thermocycling + Ethanol	
Resin composite	**Mean (SD)**	**Mean (SD)**	**Mean (SD)**	**Mean (SD)**	***p***
Essentia Universal	32.4 (5.35)^{e1,2}	26.5 (3.2)^{e3}	29.1 (3.7)^{e2,3}	38.6 (6.4)^{d1}	< 0.001
Filtek Universal	47.2 (4.5)^{c2}	43.5 (2.3)^{b3}	47.6 (3.2)^{b1,2}	52.5 (6.8)^{b1}	< 0.001
G-ænial A'Chord	41.4 (3.3)^{d1}	35.5 (2.9)^{d2}	42.7 (3.7)^{c1}	43.1 (5.5)^{c1}	< 0.001
Omnichroma	41.9 (4.1)^{d1}	37.3 (2.8)^{c2}	37.3 (3.0)^{d2}	44.2 (7.2)^{c1}	< 0.001
Tetric Prime	55.4 (2.1)^{b1}	44.9 (2.8)^{b2}	36.1 (3.1)^{d3}	44.3 (4.7)^{c2}	< 0.001
Filtek Supreme XTE	62.4 (2.3)^{a1}	55.0 (2.0)^{a2}	53.6 (6.2)^{a2}	57.6 (7.3)^{a1,2}	< 0.001
p	< 0.001	< 0.001	< 0.001	< 0.001	

Note: Different lowercase letters indicate statistically significant differences among resin composites at baseline and after each aging procedure (per columns) according to the Kruskal–Wallis test ($p < 0.05$). Different numbers indicate statistically significant differences among aging procedures for each resin composite (per row) according to the Friedman test ($p < 0.05$).

(Friedman tests, $p < 0.001$). Intra-material comparisons showed that 2-week storage in saliva resulted in a significant surface softening for all composites. Whereas thermocycling did not change the mean microhardness of OM, EU, and FS, it caused a decrease for TP and an increase for FU and GA. Thermocycling followed by ethanol softened the surface of TP compared to its initial hardness, while the microhardness of the other composites remained stable, with the exception of an increase for FU.

3.2 | Morphological Analysis of Resin Composite Microstructure and Filler Characterization

Representative SEM images of all the resin composites at baseline and after the three aging procedures are shown in Figure 1. Although all composites included prepolymerized filler particles, their shape and distribution were noticeably different. The filler shape in GA, EU, and TP was irregular, with sharp edges and a wide particle size range. GA exhibited nanoparticles and clusters of these particles ranging from 5 to 20 μm and, unlike the other composites, a boundary of these higher particles was clearly observed with a similar chemical composition according to the similar shades of gray. In the case of EU and TP, dark and light gray prepolymerized filler particles suggest the presence of elements with lower and higher atomic numbers, respectively. Rhomboidal-shaped particles reach 50 μm in these resin composites.

The filler characterization at high magnification in Figure 2 complements the findings described in Figure 1 for the resin composites with different filler arrangement ("nanofilled" and "suprananofilled," as per the respective manufacturers' description). FU contains individual nanoparticles ranging from 10 to 30 nm and particle clusters measuring from 100 nm to 5.0 μm, which are made of two types of nanoparticles, ranging from 5 to 15 nm and from 25 to 40 nm. FS displays particle clusters measuring from 250 nm to 7.5 μm made of nanoparticles from 10 to 60 nm with a few brighter nanoparticles measuring 50–90 nm. Individual nanoparticles outside the clusters were scarce. OM displays rounded microclusters measuring from 210 to 320 nm made of nanoparticles ranging from 20 to 35 nm.

Regarding the effect of the different aging procedures on composite structure, no relevant changes were detected. Only in the case of OM, some gaps around the clusters were observed after thermocycling (Figure 1).

3.3 | Sorption and Solubility

Results of both properties are shown in Table 5. ANOVA tests showed statistically significant differences among resin composites for sorption ($p < 0.001$) and solubility ($p < 0.001$). Regarding water sorption, all universal composites obtained statistically similar means. FS recorded a statistically higher mean water sorption than FU, OM, and TP. In terms of solubility, FS exhibited the highest mean, which was statistically higher than those of EU, GA, and OM, and not significantly different from those of FU and TP. No statistically significant differences were found among the mean solubility of EU, GA, OM, and TP.

3.4 | Degree of Conversion

The mean DC ranged from 52.31% to 61.57% (Table 5). One-way ANOVA detected statistically significant differences for the mean DC ($p = 0.004$). FS showed the lowest mean DC without significant differences with those of EU and FU. All universal resin composites displayed a statistically similar mean DC.

4 | DISCUSSION

The mechanical properties analyzed, as well as the microstructure of the universal composites evaluated, were not similar at baseline. The effect of the three aging procedures on the mechanical properties was material-specific. Therefore, the first null hypothesis was rejected.

In the present study, one of the methods used to determine the mechanical behavior of the resin composites was the three-point bending test to measure flexural strength, according

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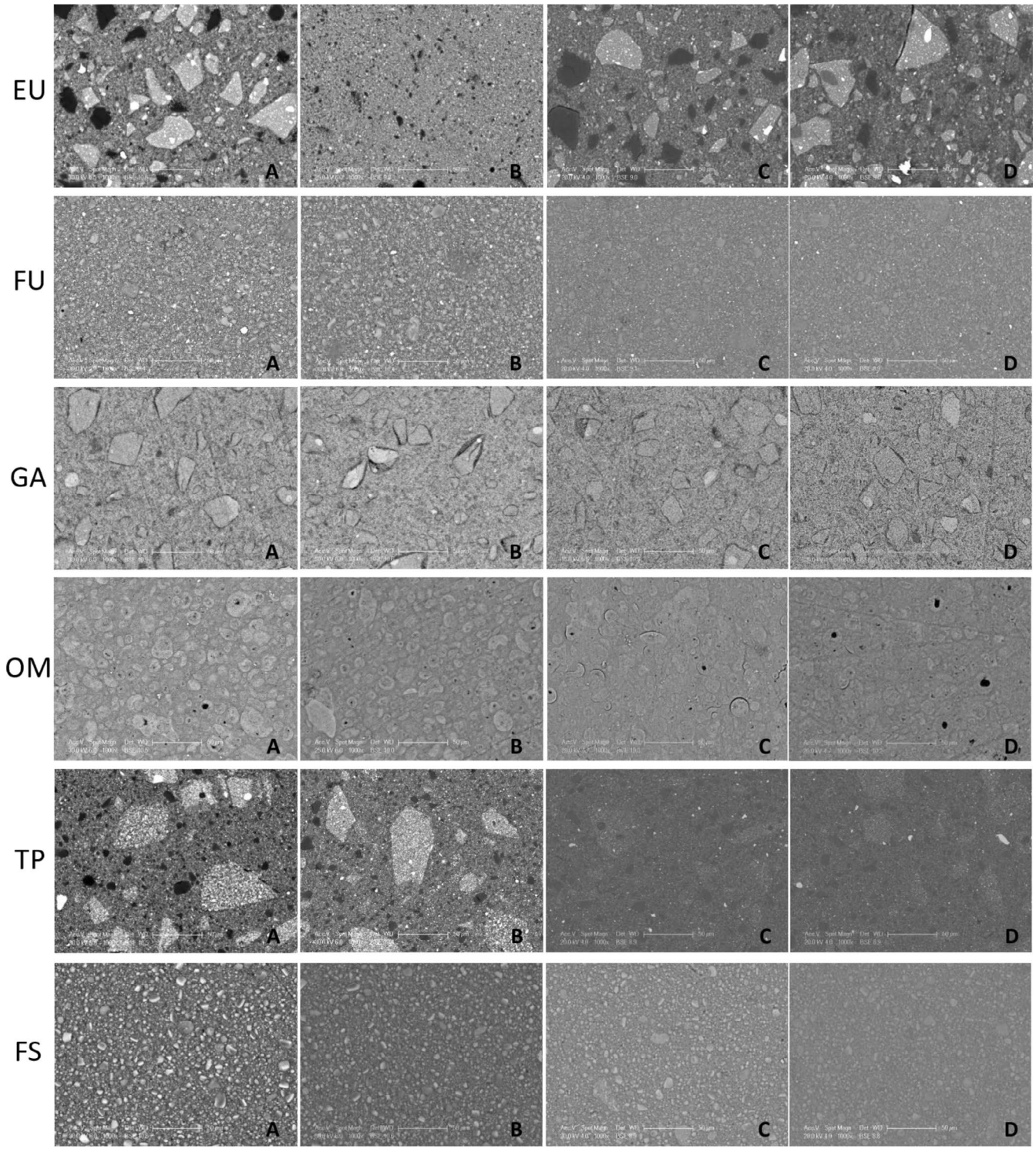

FIGURE 1 | SEM images (×1000) of all the composite resins at baseline (A) and after the three aging procedures: 2 weeks (B), thermocycling (C), and thermocycling and ethanol storage (D). Composite resins: (EU) Essentia Universal, (FU) Filtek Universal, (GA) G-ænial A'Chord, (OM) Omnichroma, (TP) Tetric Prime, and (FS) Filtek Supreme XTE.

to the ISO recommendations [56, 57]. The minimum flexural strength accepted for resin composites according to ISO standards is 80 MPa [56]. At baseline, FU attained the highest mean flexural strength followed by FS, both surpassing the cut-off value, unlike GA and TP for which the respective means were around 80 MPa, or composites such as OM and EU yielding even lower levels. As flexural strength of resin composites has been correlated with their clinical wear [43] but not with their fracture resistance, OM and EU may undergo more wear clinically than other resin composites tested in this project. In a previous study, no significant differences were detected between the flexural strength of FU and FS,

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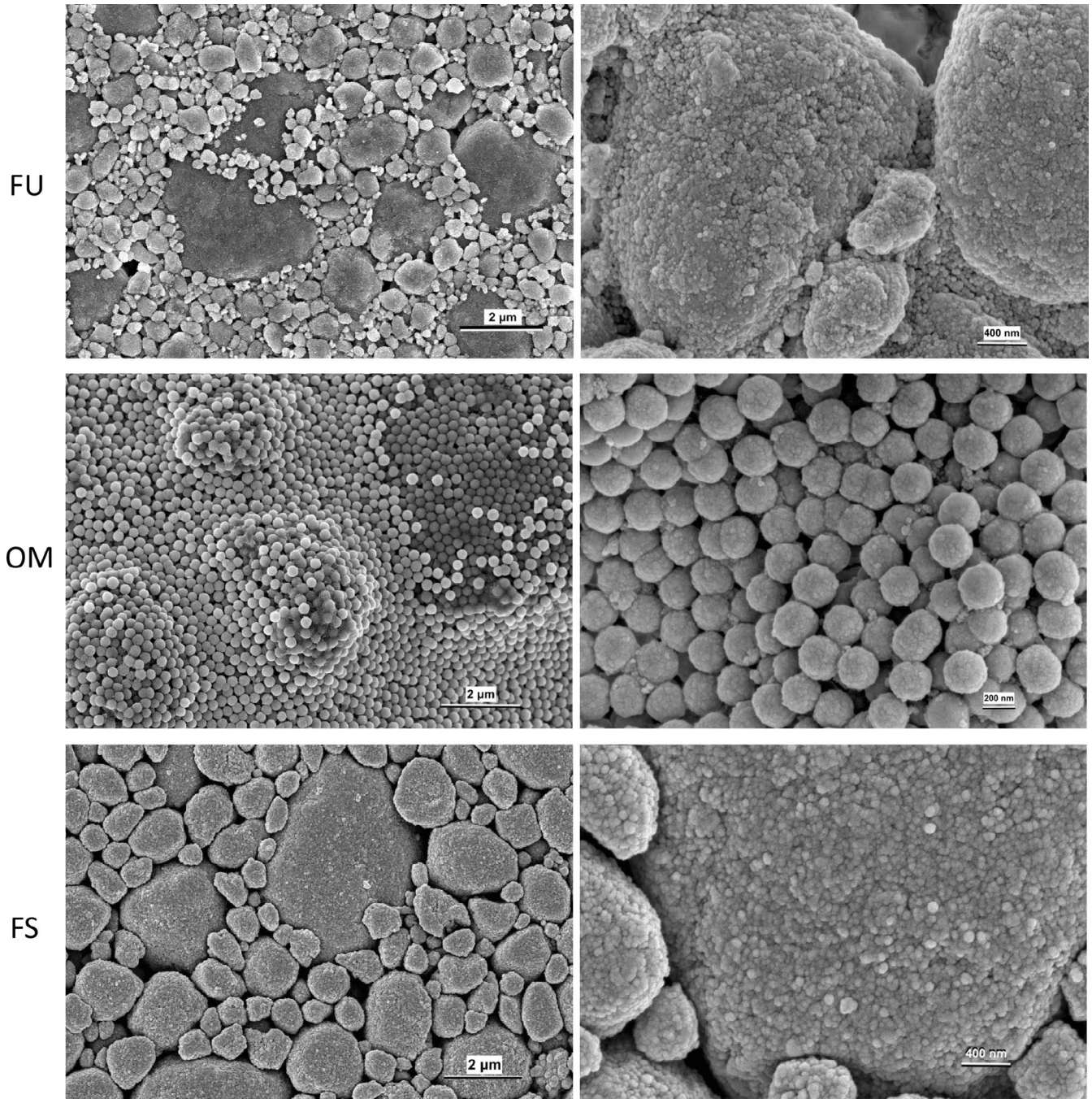

FIGURE 2 | Representative SEM images of the filler of Filtek Universal (FU) (×10,000, ×40,000), Omnichroma (OM) (×10,000, ×60,000), and Filtek Supreme XTE (FS) (×10,000, ×40,000).

although, as with our findings, the values measured for FU were higher than those for FS [20]. The authors attributed this similarity to the identical filler content in volume and distribution in both resin composites.

It is worth noting that OM exhibited the lowest mean flexural strength, which may compromise its clinical wear in posterior teeth. This behavior has been reported in previous studies [23, 33–35] and attributed to its distinctive structure, with microclusters made of nanospheres surrounded by a polymer layer, as revealed in Figure 2 [11]. This organic shell is, perhaps, considered in the inorganic percentage reported by the manufacturer, thus overestimating its real presence [11, 34]. Moreover, this structural-induced coloring would resemble the microstructure of microfilled composites [28] and would be related to lower Weibull modulus [23], easier crack propagation, and lower fracture resistance [11, 23].

Elastic modulus is another property traditionally considered to characterize the mechanical performance of resin composites, which was derived from the same three-point bending test. Resin composites with higher modulus of elasticity have been considered to perform better clinically with improved marginal quality of the restorations [44]. Mean elastic moduli attained by

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TABLE 5 | Mean water sorption and solubility ($\mu g/mm^3$) (standard deviation) and degree of conversion (%) of resin composites tested.

	Water sorption	Water solubility	Degree of conversion (%)
Resin composite	**Mean (SD)**	**Mean (SD)**	**Mean (SD)**
Essentia Universal	18.4 (2.9)^{ab}	1.1 (12)^{a}	55.1 (3.9)^{ab}
Filtek Universal	18.0 (3.0)^{b}	5.8 (2.6)^{bc}	58.5 (3.5)^{ab}
G-ænial A'Chord	19.1 (0.8)^{ab}	0.9 (1.0)^{a}	60.5 (0.5)^{a}
Omnichroma	17.9 (2.3)^{b}	2.4 (2.9)^{ab}	61.4 (1.7)^{a}
Tetric Prime	14.6 (2.0)^{b}	3.7 (1.12)^{abc}	61.6 (2.4)^{a}
Filtek Supreme XTE	23.1 (2.6)^{a}	7.2 (1.7)^{c}	52.3 (2.0)^{b}
p	0.001	<0.001	$=0.004$

Note: Lowercase letters indicate statistically significant groups within the resin composites (per columns) according to the Tukey test ($p<0.05$).

FU and FS at baseline were the highest, while that yielded by OM was the lowest (not statistically different from EU), in agreement with previous reports [23, 33, 54]. This could be explained by the strong correlation between the elastic modulus and the filler wt%, that in the case of OM would be much lower than the one reported by the manufacturer [32]. In the case of comparable filler content, shape and distribution could directly influence the elastic modulus of composites [45], unlike the organic matrix, explaining why the elastic modulus was similar for FU and FS despite the differences in their polymeric composition [20].

In the present study, microhardness was also used for the characterization of resin composites, as it is related to the resistance to plastic deformation. It has also been used to predict abrasion, although there is not a strong correlation between wear and surface hardness [45, 46].

The highest microhardness was achieved with FS, followed by TP and FU, which were also the materials with higher flexural strength. Consequently, OM yielded low results in comparison with other universal composites, as previously reported [23, 34], which were even lower for EU [54]. Although filler load is deemed to be responsible for surface hardness, this correlation was not observed in our study as FS resulted in higher microhardness than FU. Additionally, microhardness is a valid parameter to indirectly measure the DC of resin composites [60, 61]. However, according to our results, this does not seem to be the case. All universal resin composites exhibited statistically similar percentages of the DC, despite their different hardness. Additionally, FS was statistically the hardest resin composite while displaying the lowest DC, in accordance with previous studies [20, 52]. Interestingly, unlike FU, FS contains Bis-GMA in its composition, which may explain the lower DC [29–31]. Both an inorganic content of 80% or higher and the presence of fillers of irregular shape can hinder the transmittance of light, leading to a decrease in the DC [17]. Based on our results, we cannot attribute any effect of the filler content on the DC, as previously reported for other universal composites [54]. In contrast, the shape of the filler may have made a difference, explaining why OM, homogeneously loaded with spherical particles, showed the highest DC. Moreover, the refractive indices of the filler and the photopolymerized resin match in OM provide a distinctly high translucency [10, 12, 32].

As previously mentioned, not all aging procedures resulted in changes to the mechanical properties of resin composites. The variations that occurred were material-specific.

Storage in artificial saliva for 2 weeks was the first method used to simulate aging of the resin composites, as it is the medium that best mimics the oral environmental conditions. A higher ion exchange at filler surface, in comparison with distilled water, may affect the mechanical properties [41, 43, 63]. Surface deterioration was observed and microhardness decreased after this aging procedure, in contrast with flexural strength, elastic modulus, and microstructure, which remained unaltered in line with previously published results [54].

Subsequently, samples were submitted to a thermocycling process combining hydrolytic and thermal degradation, which is believed to produce debonding of the fillers and create microcracks in resin composites [38–41]. Water exerts a plasticizing effect on the resin matrix network that negatively impacts mechanical and physical properties [42]. Flexural strength of all resin composites, apart from FU, was nearly reduced by half, while the elastic modulus remained stable, except for GA. Regarding microhardness, TP was the only material that underwent surface softening after thermocycling. A stable microhardness was reported for OM, likely due to its smaller filler [23]. Moreover, electron microscopy images of most composites showed no relevant surface changes, although other authors found a significant decrease in Knoop hardness for FU, FS, and GA after 30,000 cycles, which is three times the number of cycles applied in our study.

Immersion in ethanol for 2 weeks was part of the last aging method, as it simulates intraoral degradation after a prolonged period [43, 64–66]. Ethanol storage was the most aggressive aging procedure, as it significantly affected the mechanical stability of all composites, even decreasing the flexural strength of FU. In contrast, it is worth highlighting that all resin composites underwent surface hardening after immersion in ethanol, with FU even attaining higher microhardness than at baseline. The gaps previously observed between clusters and the matrix in OM were more frequent, and surface microcracks were also evident. An issue with the silane coupling agent might be responsible for this finding, as water sorption of OM was not higher than that observed for the other composites. A particular situation

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occurred with TP, since after immersion in ethanol the values did not recover and reach those initially recorded, but those determined after immersion in saliva for 2 weeks. It should be mentioned that TP was the universal composite that suffered the most significant reduction in surface hardness after thermocycling compared to the initial values and was also the one with the highest solubility values.

Water sorption and solubility were evaluated because water uptake by resin-based composites leads to hydrolytic breakdown of the filler-matrix interface, undermining the polymeric network and decreasing the mechanical properties [49]. Nevertheless, water sorption of all universal composites was similar in our study, while statistically higher values were determined for FS. Since water sorption is closely related to solubility, the higher solubility recorded for FS comes as no surprise [49]. Although sorption and solubility are considered relevant properties that may be related to the mechanical behavior and the degradation of resin composites, this relationship was not validated in our study. FU and FS were the materials with the most stable mechanical behavior, as opposed to OM, for which sorption and solubility were not particularly high compared to the other resin composites.

The authors acknowledge the in vitro nature of this study as a limitation, which precludes our findings from being directly translated into an expected clinical performance of the analyzed materials. Moreover, the overall mechanical behavior of the universal resin composites was material dependent. The multifactorial influence on the behavior of these materials prevents us from attributing their in vitro performance to a single characteristic due to the lack of validity. Further studies should include medium- and long-term clinical trials in posterior teeth to assess the effect of intraoral mechanical, chemical, and thermal stresses on the clinical longevity of universal resin composites.

5 | Conclusions

Within the limitations of the current study, it was concluded that:

- FU recorded higher flexural strength and elastic modulus than all other universal composites. In contrast, OM scored the lowest in physical-mechanical properties.
- Thermocycling, followed or not by immersion in ethanol, reduced the flexural strength and elastic modulus of the universal composites tested, although these changes were material dependent, being less pronounced for FU.

Author Contributions

J.P. conceived the idea. L.C. and V.F. defined the methodology. E.G.-A., B.B., D.J.-D., L.C., V.F., and J.P. performed the specimen preparation and laboratory testing. V.F. and D.J.-D. performed the statistical analysis, and E.G.-A., L.C., V.F., P.Á.-L., and M.Á.G. contributed to the interpretation of the data. B.B. prepared the figures. E.G.-A. and L.C. wrote the original draft that was critically reviewed by J.P. All authors revised the manuscript, gave final approval, and agreed to be accountable for all aspects of the scientific work. This study did not receive external funding. The authors do not have any financial interest in the companies whose materials are included in this article.

Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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