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Physical Properties of Expired Composite Resins After Air Fryer Thermal Treatment

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Authors' contributions

This work was carried out in collaboration among all authors. Author MAAG contributed to the study by writing the original draft and handling resources, methodology, investigation, and conceptualization. Author JVRAA was responsible for writing, reviewing, and editing the manuscript, as well as conducting investigation and visualization. Author CSO and NLM contributed through writing, reviewing, and editing, in addition to methodology, investigation, and visualization. Author CMCT was responsible for writing, reviewing, and editing, and provided resources, supervision, and conceptualization. Author LCS contributed by writing, reviewing, and editing, as well as providing resources, conceptualization, supervision, project administration, formal analysis, and data curation. All authors read and approved the final manuscript.

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ABSTRACT

Aims: This study aimed to investigate the Physical properties of two expired composite resins after an experimental air fryer thermal treatment.
Methods: Three factors were assessed at two levels each: composite resin (Filtek Z350 XT, 3M ESPE, or Opallis, FGM), polymerization protocol (light only or light + heat), and expiration date (expired or non-expired). Resin bars (n = 6) measuring 2 × 2 × 12 mm were fabricated for three-point micro-flexural testing, and cylindrical specimens (n = 4) measuring 2 mm × 5 mm were prepared for Knoop microhardness testing. Additionally, cylindrical specimens (n = 4) measuring 1 mm × 15 mm were used for water sorption and solubility tests. Data were analyzed by three-way ANOVA and Tukey post-hoc test.
Results: Knoop microhardness differed significantly among groups (p = 0.024), with highest values for non-expired Z350 XT activated by light, expired Z350 XT activated by light + heat, and non-expired Opallis activated by light. No significant differences were found for flexural strength (p = 0.423), elastic modulus (p = 0.365), or water sorption and solubility (p = 0.893 and p = 0.193, respectively).
Conclusion: Except for microhardness, the physical properties analyzed of the expired composite resins were not affected by expiration date, polymerization protocol, or resin type.

Keywords: Composite resins; flexural strength; hardness tests; heat treatment; solubility.

1. INTRODUCTION

Composite resin is one of the most widely used materials for direct restorations in operative dentistry. Its popularity has increased due to advances in physical, mechanical and esthetic properties, as well as to the development of adhesive systems (Gungor et al., 2016; Shashikiran & Talreja, 2017; Dülger & Koşar, 2023).

In esthetic restorations, clinicians often select composites of multiple shades and saturation levels. Consequently, a broad palette of resin shades is purchased, even when only small volumes of certain shades are dispensed, which often leads to material expiration before complete consumption (Nagaoka et al., 2020; Oja et al., 2021). A similar scenario is observed in dental schools, where large material purchases aimed at cost reduction frequently result in unused, expired resin batches (Gungor et al., 2016; Shashikiran & Talreja, 2017).

The expiration date of a dental material is the interval during which the manufacturer guarantees optimal performance and assures its safety and non-toxicity. Although expired composite resins are not recommended for permanent restorations, they may be used for indirect provisional restorations (Shashikiran & Talreja, 2017). Previous investigations assessing microhardness, water sorption and shear strength of expired composites found no statistically significant differences (Gungor et al., 2016); Eliguzeloglu Dalkilic et al., 2019).

Although composite resins for indirect applications are commercially available, they share essentially the same chemical composition as direct composites. Their primary distinction is the monomer conversion stage, which occurs under controlled laboratory conditions (Miyazaki et al., 2009; Sideridou et al., 2002; Magne et al., 2015).

These conditions include increased irradiance during photoactivation and the application of heat for predetermined periods (post-cure heat treatment) (Ferracane & Condon, 1992; Ferracane et al., 1997; Almeida et al., 2017; Grazioli et al., 2019; Soares et al., 2006; Rocha et al., 2018). Heat application enhances the mechanical properties of cured composites by increasing monomer conversion and decreasing residual unreacted monomers (Sideridou et al., 2002; Grazioli et al., 2019; Lovell et al., 2001).

Manufacturers have also developed specialized ovens for thermal polymerization, but these devices are costly (Miyazaki et al., 2009; Grazioli et al., 2019; Santana et al., 2009; Zamalloa-Quintana et al., 2022). Other technologies commonly available in dental offices, such as autoclaves and microwaves, can likewise raise the temperature of photoactivated direct composites, thereby increasing monomer to polymer conversion (Miyazaki et al., 2009; Magne et al., 2015; Grazioli et al., 2019; Soares

et al., 2006; Santana et al., 2009; Zamalloa-Quintana et al., 2022).

An alternative device for this procedure is the air fryer, which offers precise temperature and time control at lower cost and with high portability. To date, no studies have evaluated its application in composite post-cure treatment. According to the manufacturer's manual, the air fryer uses an integrated turbine and resistive heating element system to circulate hot air within the chamber, rapidly cooking and baking foods. This air circulation technology ensures uniform heating from all sides.

Therefore, it is proposed that employing direct composite resins beyond their expiration date through a direct–indirect restoration technique that combines photoactivation with controlled thermal treatment in an air fryer may offer a novel approach for clinical restorative practice while contributing to the reduction of material waste.

The present study evaluated the physical properties (mini flexural strength, elastic modulus, Knoop microhardness, water sorption and solubility) of two expired composite resins after experimental thermal treatment in an air fryer. The null hypothesis was that air fryer thermal treatment following photoactivation would not significantly affect the physical properties of the expired composite resins.

2. MATERIALS AND METHODS

This study employed a factorial design with three factors assessed at two levels each: resin type (Filtek Z350 XT or Opallis); polymerization protocol (light only or light + heat); and expiration status (expired or non-expired). All expired resins were used within 12 months after their expiration date. The experimental design and material composition are detailed in Table 1.

For light curing, a Valo corded LED curing unit (1000 mW/cm²; Ultradent Products Inc., South Jordan, UT, USA) was used. Thermal treatment was performed with an air fryer (Mondial AF-03®, Bahia, Brazil). Specimens cured with light only were polymerized according to each manufacturer's instructions: Opallis, 40 s; Filtek Z350 XT, 20 s.

Specimens designated for thermal treatment were first photoactivated and, after a 10 min interval, placed in an air fryer preheated to 170 °C for 5 min and subsequently treated at the same temperature for 10 min, following Santana et al. (2009).

When more than one composite-resin increment was required during specimen fabrication, each subsequent photoactivation was performed with the light-curing unit's tip supported on the specimen holder. For the final layer, the resin was covered with a polyester strip and glass slide, then cured with the light tip resting directly on the assembly.

An illustrative scheme of the materials and methods employed to assess the physicochemical properties of expired and non-expired composite resins under different polymerization protocols is shown in Fig. 1.

Table 1. Material formulation and experimental group design used in this study.

Material	Composition	Groups	
Filtek Z350 XT 3M ESPE, Sumaré, Brasil	TEGDMA, Bis-EMA, UDMA, PEGDMA. Unagglomerated silica and zirconia particles (20 nm and 4 – 11 nm, respectively). Aggregated silica/zirconia (0.6 – 10 µm). 72.5% by weight and 55.5% by volume. Camphorquinone. **Shade:** A1B	**Polymerization protocol**	**Condition**
		Light only	Expired
			Unexpired
		Light + Heat	Expired
			Unexpired
Opallis FGM Produtos Odontológicos, Joinville, Brasil	Bis-GMA, Bis-EMA, TEGDMA, UDMA, co-initiator, silane. Silanized barium-aluminum-silicate glass filler (0.5 µm). 78.5 – 79.8% by weight and 57.0 – 58.0% by volume. **Shade:** DA1	Light only	Expired
			Unexpired
		Light + Heat	Expired
			Unexpired

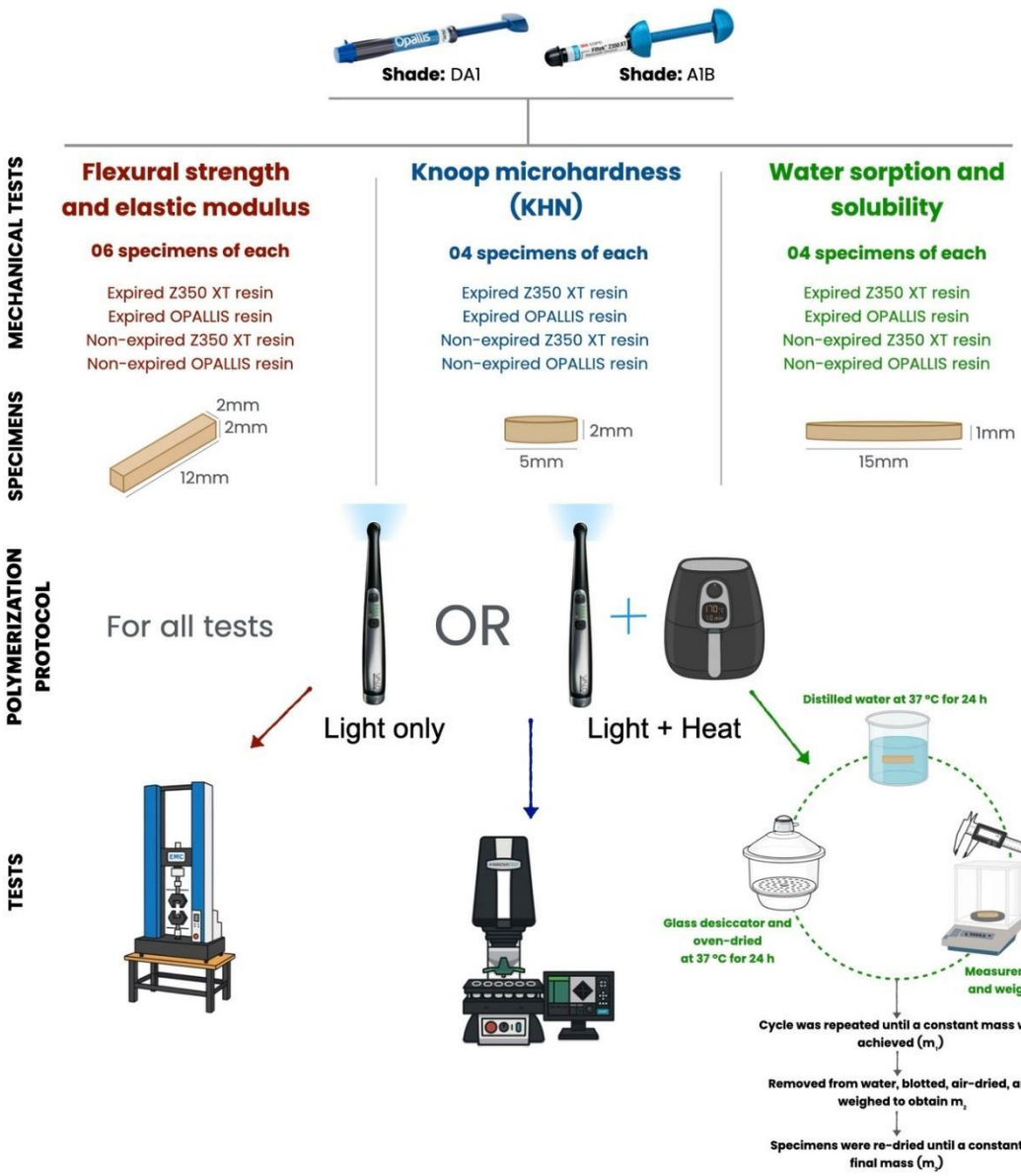

Fig. 1. Illustrative scheme of the materials, methods, and tests performed in this study

2.1 Flexural Strength and Elastic Modulus

Forty-eight composite resin bars were fabricated using an addition-silicone mold (Scan Putty; Yller, Pelotas, Brazil) placed inside a polyvinyl-chloride (PVC) cylinder (5 mm diameter × 4 mm height). The bars measured 2.0 mm (±0.1 mm) wide, 2.0 mm (±0.1 mm) thick, and 12.0 mm (±0.1 mm) long (Grazioli et al., 2019; Soares et al., 2006).

After 24 h of storage in distilled water at 37 °C in an incubator, elastic modulus and flexural strength were determined by a three point micro-flexural test on a universal testing machine (Instron 3345; Instron Inc., Canton, MA, USA) equipped with a 500 N load cell. Tests were performed at a crosshead speed of 0.5 mm/min and a support span of 10 mm.

2.2 Knoop Microhardness (KHN)

Cylindrical specimens were fabricated in a metal mold, resulting in specimens 5.0 mm (±0.1 mm) in diameter and 2.0 mm (±0.1 mm) thick. Each specimen's base was marked.

After 24 h of storage in distilled water at 37 °C, the specimens were mounted on acrylic plates with their tops facing upward. All specimens were polished in an automatic polishing machine (Buehler®, Lake Bluff, IL, USA) at 180 rpm clockwise under running water, using 400 grit wet silicon carbide paper (3M, St. Paul, MN, USA) for 20 s, followed by 1200-grit wet silicon carbide paper (Worker, São Paulo, Brazil) for 30 s.
The tops of the specimens were subjected to the Knoop microhardness test using a microhardness tester (HMV-2; Shimadzu, Tokyo, Japan). Five indentations were made under a 50 gf load for 15 s on each specimen.

2.3 Water Sorption and Solubility

Thirty-two cylindrical specimens were fabricated in a metal mold, yielding specimens 15.0 mm (±0.1 mm) in diameter and 1.0 mm (±0.1 mm) thick. Because the specimen diameter exceeded the curing-tip diameter (10 mm), light was applied centrally, followed by four additional peripheral exposures, as specified in ISO 4049:2019.

After 24 h of storage in distilled water at 37 °C, all specimens were measured and weighed. A single diameter and thickness measurement were recorded for each specimen using a digital caliper (Stainless Hardened; China) and a precision balance (Shimadzu; Tokyo, Japan). Specimens were then placed in a glass desiccator and oven-dried at 37 °C for 24 h. They were weighed again, and this cycle was repeated until a constant mass (m_1) was obtained.

After this step, specimens were immersed in distilled water and stored at 37 °C in an incubator for 7 days. At the end of the storage period, specimens were removed from the water, blotted with paper towels for 1 min, then placed on fresh paper towels for further drying, and weighed to record m_2 (water saturated mass). Thereafter, specimens were returned to the desiccator and the drying cycle was repeated until a constant final dry mass (m_3) was obtained.

Water sorption values *were calculated using the formula:*
Wsp **=** $(m_2 – m_3) / V$
Water solubility values *were calculated using the formula:*
Wsl **=** $(m_1 – m_3) / V$ *(ISO 4049:2019)*

where:

- ***Wsp*** *represents water sorption*
- ***Wsl*** *represents water solubility*
- m_1, m_2, *and* m_3 *are the constant masses obtained during the experiment*
- ***V*** *represents specimen volume* (mm^3).

2.4 Statistical Analysis

Statistical analyses were performed using Jamovi software (Jamovi Project; Sydney, Australia). Data normality was assessed by the Shapiro–Wilk test and variance homogeneity by Levene's test, with parametric assumptions considered met when $p > 0.05$. A three-way analysis of variance (ANOVA) was conducted to evaluate the effects of resin type, polymerization protocol, and expiration status, followed by Tukey's post-hoc test for pairwise comparisons. Statistical significance was set at $p < 0.05$.

3. RESULTS

3.1 Flexural Strength and Elastic Modulus

No significant differences were observed among the groups for flexural strength ($p = 0.423$) or elastic modulus ($p = 0.365$) when three-way ANOVA evaluated all interactions (Table 2).

However, three-way ANOVA revealed statistically significant main effects of resin type and polymerization protocol on elastic modulus. Specifically, Z350 XT exhibited a higher elastic modulus than Opallis ($p=0.003$), and specimens polymerized with light only exhibited a higher elastic modulus than those polymerized with combined light and heat ($p=0.04$).

3.2 Knoop Microhardness

Regarding microhardness test results, a statistically significant three-way interaction among the factors was detected ($p=0.024$) (Table 3). The non-expired Z350 XT resin activated by light exhibited significantly higher Knoop microhardness values than all other groups, except for the expired Z350 XT resin polymerized with light + heat ($p=0.79$) and the non-expired Opallis resin polymerized with light ($p=0.88$). Within the Opallis resin groups, a significant difference was observed only between non-expired and expired specimens when polymerized with light only ($p=0.001$).

Table 2. Mean (± standard deviation) values for flexural strength and elastic modulus according to experimental groups (MPa)

Composite Resin	Polymerization	Condition	Flexural Strength (MPa)	Elastic Modulus (MPa)
Filtek Z350 XT	Light only	Expired	233 (±50.1)	11798 (±1013)
		Unexpired	260 (±40.2)	11337 (±1187)
	Light + Heat	Expired	240 (±57)	10181 (±1670)
		Unexpired	291 (±50.1)	11746 (±1673)
Opallis	Light only	Expired	255 (±26)	10274 (±1070)
		Unexpired	273 (±55.9)	10899 (±600)
	Light + Heat	Expired	270 (±64.3)	8959 (±1611)
		Unexpired	264 (±51.4)	10232 (±1193)

Table 3. Mean Knoop microhardness values (± SD) according to experimental groups (gf)

Composite Resin	Polymerization	Condition	KHN (gf)
Filtek Z350 XT	Light only	Expired	62.8 ± (7.51) **B**
		Unexpired	76.5 ± (3.75) **A**
	Light + Heat	Expired	71.7 ± (6.16) **AC**
		Unexpired	54.2 ± (2.64) **B**
Opallis	Light only	Expired	57.2 ± (2.88) **B**
		Unexpired	72.4 ± (3.54) **AC**
	Light + Heat	Expired	64.5 ± (3.22) **BC**
		Unexpired	63.6 ± (3.17) **BC**

Different uppercase letters indicate statistically significant differences ($p < 0.05$)

Table 4. Mean values (± standard deviation) obtained for water sorption and solubility tests, according to the evaluated groups, in µg/mm³

Composite Resin	Polymerization	Condition	Water Sorption (µg/mm³)	Water Solubility (µg/mm³)
Filtek Z350 XT	Light only	Expired	18.2 (± 2.49)	0.201 (± 0.55)
		Unexpired	21.2 (± 0.72)	-2.96 (± 0.84)
	Light + Heat	Expired	20.9 (± 1.37)	2.70 (± 0.37)
		Unexpired	22.5 (± 3.05)	1.77 (± 1.27)
Opallis	Light only	Expired	8.12 (± 1.25)	-0.87 (± 1.03)
		Unexpired	9.28 (± 1.04)	-1.62 (± 0.84)
	Light + Heat	Expired	9.77 (± 1.02)	0.77 (± 0.23)
		Unexpired	9.80 (± 1.23)	0.60 (± 1.18)

3.3 Water Sorption and Solubility

The water sorption and solubility tests (Table 4) showed no statistically significant interaction among the three factors ($p=0.893$ for sorption and $p=0.193$ for solubility).

4. DISCUSSION

In the present study, no statistically significant differences were found for flexural strength, elastic modulus, or water sorption and solubility among the factors composite resin, polymerization method, and expiration status. Statistically significant differences were observed only in the microhardness test.

The three-point flexural test is designed to simulate in vitro clinical situations, such as how a material behaves under masticatory loads. However, specimen preparation for this test, as specified in ISO 4049:2019, presents limitations regarding the light-curing process. Because the specimens are relatively long (25 mm), irradiance is highest at the central irradiation site and diminishes toward the ends, increasing the

likelihood of fracture outside the intended flexural zone (Yap et al., 2018; Kumar, 2012; Palin et al., 2005).

The mini-flexural test, widely reported in the literature (Yap et al., 2018; Yap & Teoh, 2003), uses 12 mm bars, providing greater similarity to dental anatomy and allowing single-step light activation to ensure uniform irradiation across each specimen. Results from the three-point flexural and mini-flexural tests are strongly correlated. Thus, the mini-flexural test offers advantages: it is easier to perform and more accurately reflects clinical conditions (Yap & Teoh, 2003).

Studies using accelerated aging protocols have reported a significant decrease in the flexural strength of Filtek Z350 XT resin when comparing unexpired specimens to aged ones. In contrast, the elastic modulus of the same resin did not differ significantly between unexpired and expired groups, corroborating the findings of the present study (Nagaoka et al., 2020; D'Alpino et al., 2014).

A study using a hydrothermal aging protocol evaluated the expiration period of the tested resins and reported that up to three years after the expiration date, no statistically significant differences were observed in flexural strength, elastic modulus, or wear resistance. However, a reduction in microhardness was observed in the expired composites (Oja et al., 2021), which is consistent with the results of this study.

Another study evaluated the flexural properties of five composite resins that were stored for up to 30 months beyond expiration (Nagaoka et al., 2020). The tested materials retained their flexural strength for up to 15 months post-expiration. Since the resins used in the present study were expired for no more than 12 months, these findings are consistent with our results.

Some laboratory studies have investigated the influence of filler particle size and filler weight on the flexural properties of resin composites (Tanimoto et al., 2006; Rodrigues Junior et al., 2007). Tanimoto et al. (2006) analyzed the effect of particle size on flexural strength and observed that strength decreased as particle size increased. However, no statistically significant differences were detected in the elastic modulus among the tested materials.

Rodrigues Junior et al. (2007) found a low but significant correlation between the mechanical properties and the weight percentage of inorganic filler in composite resins. This percentage was determined by ashing the organic matrix of three resin types (universal nanoparticulate, hybrid, and microparticulate). Subsequently, the influence of the inorganic content on flexural strength and elastic modulus was evaluated by three-point flexural testing. In the present study, although the analyzed composites differ slightly in particle size (nanoparticulate Z350 XT versus microhybrid Opallis), they have an equivalent filler volume percentage, thus providing a meaningful context for comparison.

Previous studies have also reported a reduction in microhardness values when composite resins are past their expiration date (Garcia et al., 2009; Tirapelli et al., 2004). In the current investigation, similar behavior was observed: within each composite group (Opallis and Z350 XT activated by light only), expired resins exhibited significantly lower microhardness than non-expired ones ($p = 0.004$ for Opallis; $p = 0.001$ for Z350 XT).

Similarly, Tirapelli et al. (2004) assessed the radiopacity and microhardness of two composite resins and one compomer by comparing specimens that were non-expired with those beyond their expiration date. A statistically significant difference was observed in one of the composite resins, exhibiting decreased values in the expired group.

In 2009, Roberti Garcia et al. (2009) observed that microhardness of composite resins decreases when tested 180 days post-expiration. They evaluated the degree of conversion, microhardness, and surface roughness of expired resins subjected to direct application and light curing only. Nevertheless, when thermal treatment was combined with light activation, expired Z350 XT resin exhibited a statistically significant increase of approximately 14% in surface microhardness compared to light activation alone.

Evidence in the literature suggests that, in general, the application of heat results in increased mechanical properties of cured composites due to improved conversion and reduced unreacted monomers (Sideridou et al., 2002; Grazioli et al., 2019). However, in this work, such enhancement was detected in the heat-treated group only during the Knoop microhardness test and was absent in the other mechanical assessments.

In the non-expired Z350 XT group, heat and light curing was associated with an approximately 30% decrease in Knoop microhardness compared with light-only curing. Although heat has been linked to a higher degree of conversion, excessive temperature may promote composite degradation, which can reduce surface hardness (Miyazaki et al., 2009).
The glass transition temperature (Tg) is a material dependent parameter that can guide the selection of an appropriate thermal treatment protocol (Santana et al., 2011). Above Tg, secondary molecular interactions weaken, permitting greater molecular relaxation and enabling trapped free radicals to react, thereby increasing the degree of conversion (Miyazaki et al., 2009). Reported Tg values for composite resins range from approximately 157 to 162 °C, and the initial loss of volatile components may begin at temperatures starting from 180 °C (Santana et al., 2011).

Santana et al. (2009) observed that an experimental thermal treatment at 170 °C for 10 min significantly increased the mechanical properties of the tested composites. In the present study, the thermal curing protocol was aligned with literature recommendations (≤170 °C/10 min). Therefore, this condition alone is unlikely to explain the decrease in microhardness observed in the non-expired Z350 XT group subjected to heat and light curing.

In 2019, Eliguzeloglu Dalkilic et al. reported no statistically significant differences in Vickers hardness or water solubility between expired and non-expired composite resins. In that study, four composites were evaluated, and all expired specimens were at most six months past the expiration date (Eliguzeloglu Dalkilic et al., 2019). This short post-expiration period, as well as the use of a specific LED curing unit, may have influenced the results (Eliguzeloglu Dalkilic et al., 2019; Gungor et al., 2016).

With advancements in dental technologies, third generation light curing units (LCUs), often referred to as polywave or multi peak devices, can deliver irradiance from 1,000 to 3,000 mW/cm² (Pelissier et al., 2011). These devices are considered universal because they emit light across multiple spectral peaks that activate camphorquinone and alternative photoinitiators (e.g., TPO, PPD) (Pelissier et al., 2011). The VALO LED curing light (Ultradent Products Inc., South Jordan, UT, USA) is an example of a third generation LCU. In the present study, the device's high irradiance and broader spectral coverage may have contributed to the observed outcomes, including for expired composites polymerized with light only. This spectral output likely supported a high degree of conversion.

Muniz et al. (2013) investigated the influence of thermal treatment on the water sorption and solubility of two direct composite resins by using a dry heat oven (170 °C, 10 min). They found statistically significant differences in water sorption ($p = 0.026$) and in solubility ($p = 0.01$) (Muniz et al., 2013). Fabre et al. (2007) also documented negative solubility values, which do not indicate the absence of this property. Rather, water sorption exceeded mass loss, leading to an increase in specimen mass after immersion. This behavior is frequently associated with high material hydrophilicity, which favors the retention of water molecules through interactions with polar sites along the polymer chain (Muniz et al., 2013; Malacarne et al., 2006).

No statistically significant changes were detected in water sorption and solubility after the experimental air fryer thermal treatment. One plausible explanation is the comparatively lower hydrophilicity of the formulations evaluated here relative to those in previous studies, including the microhybrid FillMagic and the hybrid Filtek P60 (Muniz et al., 2013) and the universal Filtek Ultimate (Dülger & Koşar, 2023). Filtek Z350 XT is primarily formulated with Bis-GMA, UDMA and TEGDMA, whereas Opallis contains Bis-GMA and Bis-EMA. Although these monomers contain polar groups, the overall formulations in this study appear to show only moderate water uptake.

To the best of our knowledge, there are no published reports using air fryers to provide heat for postcuring composite resin restorations, which limits direct comparison with previous studies. Based on the results obtained, the air fryer may be a viable heat source for thermal polymerization under the conditions tested. Further studies are necessary, including cytotoxicity assessments of expired composite resins and in situ or clinical investigations, to ensure safety and support clinical applicability of the materials.

5. CONCLUSION

Within the limitations of this study, the interaction among resin type, polymerization protocol and

expiration status did not significantly affect flexural strength, elastic modulus, water sorption or solubility. No main effect of expiration status was detected for these outcomes, supporting the study hypothesis. In contrast, microhardness showed a statistically significant difference, indicating that thermal treatment influenced this property in expired resins.

ETHICAL APPROVAL

This study involved only inert materials and did not include human participants or animals; therefore, submission to Brazil's national ethics system (Plataforma Brasil) and review by the Research Ethics Committee (CEP) and the Ethics Committee for Animal Use (CEUA) were not required, in accordance with Brazilian National Health Council (CNS) Resolutions 466/2012 and 510/2016.

DISCLAIMER (ARTIFICIAL INTELLIGENCE)

Author(s) hereby declare that generative AI technologies were used exclusively for English language correction and improvement of fluency. The manuscript was originally written in English by the authors. No part of the data, results, or scientific interpretation was generated or modified by AI.

Details of the AI usage are provided below:

1. Technology used: ChatGPT (GPT-5 model, OpenAI, 2025 version).
2. Purpose: English grammar correction and enhancement of scientific writing style.
3. Input data: Original English text written by the authors.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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