



Original Article

# Comparison of Mechanical, Surface, and Chemical Properties of Different Thermoplastic Retainer Materials before and after Thermoforming: Scanning Electron Microscopy and Fourier Transform Infrared Spectroscopy Analyses

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**Cite this article as:** Tunca Y, Fahrzadeh N, Tunca M, Akinay Y. Comparison of mechanical, surface, and chemical properties of different thermoplastic retainer materials before and after thermoforming: scanning electron microscopy and fourier transform infrared spectroscopy analyses. *Turk J Orthod.* 2025; 38(4): 224-232

## Main Points

- Thermoforming significantly increases surface roughness in some polyethylene terephthalate glycol-based retainers, potentially affecting long-term performance.
- Scanning electron microscopy and Fourier transform infrared spectroscopy analyses revealed that molecular structure remains stable despite morphological changes.
- Surface roughness may influence bacterial adhesion, making material selection critical for clinical outcomes.
- Thermoplastic materials based on polyethylene terephthalate glycol copolyester with high surface homogeneity exhibit improved long-term stability.

## ABSTRACT

**Objective:** The aim of this study was to evaluate the surface hardness, surface roughness, and chemical structure changes of four vacuum-formed retainer (VFR) materials after thermoforming.

**Methods:** Four groups of VFR materials were evaluated: Taglus, Lumex-G, Atmos, and Duran. Each group consisted of 12 samples (n=12) that were thermoformed according to the manufacturer's guidelines. Surface roughness was measured using a high-precision profilometer, and hardness was assessed with a micro-Vickers hardness tester. Chemical structure analysis was conducted using Fourier transform infrared spectroscopy (FTIR), and surface morphology was examined using scanning electron microscopy (SEM).

**Results:** Hardness measurements demonstrated a general decrease across all groups following thermoforming. After thermoforming, surface roughness increased significantly in the Taglus and Lumex-G groups, whereas the Atmos and Duran groups maintained greater surface stability. FTIR demonstrated that all materials retained their chemical stability, and no significant changes in functional groups were detected. SEM results revealed more pronounced surface irregularities in the Taglus and Lumex-G groups.

**Conclusion:** Polyethylene terephthalate glycol copolyester-based materials with high surface homogeneity, such as Atmos and Duran, may be recommended for clinical use due to their superior surface stability and chemical resilience. By contrast, the surface irregularities observed in Taglus and Lumex-G may compromise their long-term clinical performance.

**Keywords:** Vacuum-formed retainer, surface hardness, surface roughness, Fourier transform infrared spectroscopy

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**Received:** July 25, 2025 **Accepted:** December 18, 2025 **Publication Date:** 30.12.2025



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INTRODUCTION

After completion of orthodontic treatment, retainers are commonly used to stabilize newly aligned teeth.<sup>1</sup> Retainers are generally classified into two main categories: removable and fixed.<sup>2</sup> Compared with fixed retainers, removable retainers, made of thermoplastic materials and known as vacuum-formed retainers (VFRs), are preferred for their superior comfort and aesthetic appeal.<sup>3,4</sup> Although patient compliance is a limitation of VFRs, they are cost-effective, easy to insert and remove, and require minimal clinical time.<sup>5</sup> VFRs are primarily manufactured from a variety of thermoplastic polymers, each with unique properties that substantially affect the final product's performance.<sup>6</sup>

Commonly used thermoplastic materials include polyurethane, polypropylene (PP), and polycarbonate (PC).<sup>7</sup> The current literature indicates that the majority of commercial VFRs are polyethylene terephthalate glycol (PET-G)-based; fewer materials belong to the thermoplastic polyurethane (TPU) class, while PC- and PP/polyolefin-based systems are less commonly represented.<sup>8-10</sup> These materials soften upon heating, can be thermoformed, and then harden, retaining their shape upon cooling. This thermal process directly influences the materials' mechanical properties, causing changes in surface roughness, hardness, and porosity due to the rearrangement of polymer chains.<sup>11,12</sup> Surface hardness determines a material's resistance to deformation and scratching, contributing to its longevity and wear resistance.<sup>13</sup> Surface roughness affects tactile sensations, gloss, friction, and light reflection, which are critical for both functional and aesthetic purposes.<sup>14</sup>

Structural changes in these materials may lead to issues such as discoloration, abrasion, and increased bacterial retention over time.<sup>15-18</sup> Although the recommended usage period for VFRs varies, an average of 12 months is typical.<sup>1,19</sup> Over time, changes in the surface properties of thermoplastic materials, including hardness, roughness, and porosity, significantly affect their performance and aesthetics.<sup>18</sup> Although researchers have explored these properties in thermoplastic retainer materials,<sup>8,11,20</sup> comprehensive evaluations using scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) are lacking in the literature.<sup>21</sup> The current study makes a key contribution by evaluating the mechanical properties of four commonly used VFR materials, using SEM and FTIR analyses, in their initial state and after thermal processing.

The null hypothesis ( $H_0$ ) of our study is "There is no difference between VFR materials in terms of surface roughness, surface hardness, or chemical structure changes in the initial state and after thermoforming".

METHODS

Analyses of surface hardness and roughness were conducted at the Dentistry Research Laboratory of Van Yüzüncü Yıl University. As the study did not involve the use of human or animal materials, ethical approval was not required.

Sample Size

The sample size for this study was calculated to detect changes in two physical properties-surface hardness and surface roughness-through repeated measurements conducted before and after thermoforming. Assuming a medium effect size ( $f=0.25$ ), a significance level of  $\alpha=0.05$ , and a statistical power ( $1-\beta$ ) of 80%, a sample size of 48 was required. This design involved repeated measurements of four thermoplastic retainer materials, with a minimum of 12 samples per group ( $n\geq12$ ).

Sample Selection Criteria

Four main VFR groups, each consisting of 12 samples from different commercial brands, were selected. The thermoplastic retainer appliances were Duran (125×125×1 mm; Scheu Dental GmbH, Iserlohn, Germany), Taglus (125×125×1 mm; Laxmi Dental Export Pvt. Ltd, Mumbai, India), Atmos (approximately 127×127×1.016 mm; American Orthodontics, Sheboygan, WI, USA), and Lumex-G (125×125×1 mm; Foamalite Ltd, Loch Gowna, Cavan, Ireland). These four thermoplastic retainer materials were selected because they are among the most widely used in clinical orthodontics. To ensure standardization, all materials were used in their original commercial dimensions as provided by the manufacturers, without cutting or reshaping, to avoid altering their physical properties. The brands, compositions, and thermal processing durations of the thermoplastic materials are presented in Table 1. All tests were conducted in a controlled laboratory environment (23 °C±2 °C, 50%±5% relative humidity) following a minimum 24-hour stabilization period to mitigate the potential impact of moisture and temperature on the thermoplastics. Specimens were handled in accordance with established protocols, with powder-free nitrile gloves used as a standard precaution. Additionally, the surfaces under consideration were meticulously cleaned with

Table 1. Thermoplastic material brands and contents and thermal processing times				
VFR Brand	Content	Manufacturer	Heating time (s)	Cooling time (s)
Duran	Polyethylene terephthalate glycol copolyester (PET-G)	Scheu Dental GmbH, Iserlohn, Germany	30	60
Taglus	Polyethylene terephthalate glycol copolyester -polyethylene	Laxmi Dental Export Pvt. Ltd, Mumbai, India	70	40
Atmos	Copolyester	American Orthodontics, Sheboygan, WI, USA	40	30
Lumex G	Polyethylene terephthalate glycol copolyester	Foamalite Ltd, Loch Gowna, County Cavan, Ireland	30	25

lint-free wipes and isopropyl alcohol ( $\geq 99.5\%$ ), which is known for its effectiveness in removing contaminants and promoting a sterile environment.

### Micro-Vickers Hardness Measurement

The micro-Vickers hardness of the VFR samples was measured using a micro-Vickers hardness tester (HMV-G 21D; Shimadzu, Japan). A square-pyramidal indentation was created on the sample surfaces using a Vickers diamond indenter. The device was calibrated with a certified reference hardness block before performing three repeated measurements on each sample. The instrument settings were configured with a Vickers load of HV 0.025 (245.2 mN) and a dwell time of 15 s. Indentations were then photographed at 40 $\times$  magnification (Figure 1). The mean of three measurements, each taken from a different region of the sample, was calculated. The same procedure was repeated after thermoforming.

### Surface Roughness Measurement

The most common surface roughness parameters were evaluated: Ra (average roughness), Rq (root mean square roughness), and Rz (maximum height of the profile). A high-precision manual profilometer (SurfTest SJ-301, Mitutoyo, Kanagawa, Japan) equipped with a 5- $\mu$ m diamond stylus tip was used for the measurements. The device scanned 2 mm in a linear motion across the top surface of each specimen to detect irregularities (Figure 2). Three readings were taken at different locations on each specimen, and the mean value was used for statistical analysis.

The instrument was calibrated with a manufacturer-supplied reference standard prior to each measurement session to ensure accuracy and reproducibility. A cut-off value of 0.8 mm and a scan speed of 0.5 mm/s were applied in accordance with international roughness measurement standards. The same procedure was repeated after thermoforming.

### SEM, DSC, and FTIR Analyses

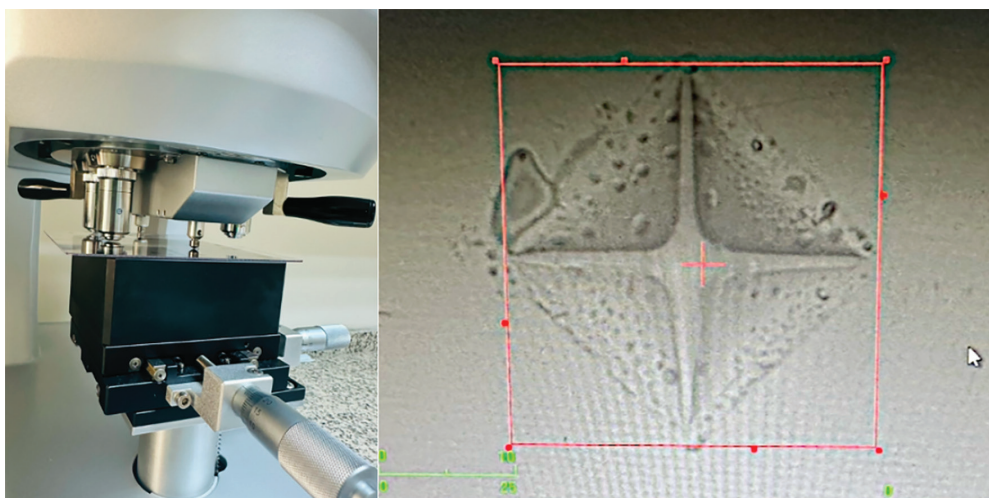
Morphological analyses of the VFR groups were conducted using field-emission SEM. All SEM images are presented at consistent magnifications with clearly visible scale bars (200  $\mu$ m for low magnification and 100  $\mu$ m for high magnification). The chemical bond types and thermal behaviors of the VFR groups were characterized by FTIR and differential scanning calorimetry (DSC). The thermal behavior and phase changes of the Taglus, Lumex-G, Atmos, and Duran groups were further investigated using DSC.

### Thermoforming

Thermoforming was performed on VFR samples after pre-thermoforming measurements. To standardize the process, each sample was pressed into a metal mold with a flat, round surface, and a single doctor monitored the processing time. Thermoforming was performed using an Essix-type vacuum-forming device (Dentsply Sirona, USA), which operates within a heating range of 160  $^{\circ}$ C–200  $^{\circ}$ C. The device applies a negative pressure (vacuum) of approximately -0.8 bar to adapt the softened sheet to the dental model, with a heater-to-sheet distance of approximately 25 mm. After thermoforming, the same mechanical property measurements (surface hardness and roughness) were repeated under the same conditions.

### Statistical Analysis

Statistical analyses were performed using IBM SPSS Statistics for Windows, version 23.0 (IBM Corp., Armonk, NY, USA) and the R statistical software (R Foundation for Statistical Computing, Vienna, Austria). A robust ANOVA was used to compare hardness and surface roughness measurements that were not normally distributed across brands and thermoforming conditions. Multiple comparisons following robust ANOVA were performed using robust pairwise t-tests, with Holm correction to control the family-wise error rate. Parameters that followed a normal distribution were compared across three or more groups using one-way ANOVA. Parameters that were not normally distributed were compared across three or more groups using the Kruskal-Wallis H test. Multiple comparisons



**Figure 1.** Micro-Vickers hardness of the vacuum-formed retainer samples.

were analyzed using Dunn’s test. The results are presented as mean ± standard deviation and as the median (minimum-maximum). The significance level was set at  $p<0.05$ .

RESULTS

Surface hardness values before and after thermoforming, and the comparative statistics by brand and thermoforming are presented in Table 2. Differences in median surface hardness by brand were not statistically significant ( $p=0.212$ ). However, the effects of thermoforming and thermoforming \*brand factors on median hardness values were statistically significant ( $p<0.001$ ). Comparative statistics for differences in surface hardness before and after thermoforming are presented. No statistically significant differences were observed among brands in mean hardness values ( $p=0.150$ ).

Comparative statistics of the Ra, Rz, and Rq surface roughness values before and after thermoforming, by brand, are presented in Table 3. The main effect of brand on median Ra values was not statistically significant ( $p=0.054$ ), whereas the main effect of thermoforming on median Ra values was statistically significant ( $p<0.001$ ). Furthermore, the interaction between brand and thermoforming for Ra values was statistically significant ( $p=0.021$ ). The highest median Ra value (0.12) was observed in the Taglus group after thermoforming, whereas the lowest median value (0.02) was found in the Duran, Atmos, Taglus, and Lumex-G groups before thermoforming. A statistically significant difference was observed in the median Ra values among brands ( $p=0.002$ ). The median values were 0.03 for Duran, 0.02 for Atmos, 0.09 for Taglus, and 0.06 for Lumex-G. The median Ra value for Atmos was significantly lower than that for Taglus, but no significant difference was observed between Duran and Lumex-G.

The main effect of brand on median Rz values was not statistically significant ( $p=0.667$ ), whereas the main effect of thermoforming on Rz values was statistically significant ( $p=0.037$ ). However, the interaction effect between brand and thermoforming on Rz values was not statistically significant ( $p=0.300$ ). For Rz surface roughness, the median values were: Duran 0.14 (0.10-0.52); Atmos 0.05 (0.32-0.4); Taglus 0.44 (0.11-2.57); and Lumex-G 0.3 (0.04-1.16). Median Rz values differed significantly among brands ( $p=0.004$ ). The median Rz value for



Figure 2. Surface roughness measurement using Surftest SJ-301 (Mitutoyo, Kanagawa, Japan).

Table 2. Comparison of surface hardness values by brand and thermoforming							
Brand	Thermoforming		Difference Mean±SD Median (Min-Max)		Q	η <sup>2</sup>	p
	Before Mean±SD Median (Min-Max)	After Mean±SD Median (Min-Max)					
Duran	13.26±1.52 13.47 (10.07-15.23) <sup>A</sup>	9.13±0.14 9.14 (8.85-9.38) <sup>B</sup>	4.13±1.51 4.34 (1.08-6.09)	B	1.50	0.049	0.212
Atmos	12.35±0.56 12.25 (11.12-13.07) <sup>A</sup>	9.22±0.18 9.22 (8.97-9.47) <sup>B</sup>	3.12±0.6 3.21 (1.65-3.98)	T	173.55	0.664	<0.001
Taglus	12.38±1.23 12.67 (9.77-14.27) <sup>A</sup>	9.30±0.39 9.34 (8.47-9.84) <sup>B</sup>	3.08±1.37 3.24 (0.36-5.36)	B*T	18.06	0.381	<0.001
Lumex-G	12.00±1.80 11.9 (7.72-15.27) <sup>A</sup>	9.10±0.21 9.02 (8.9-9.52) <sup>B</sup>	2.90±1.85 2.82 (-1.4-6.29)				
Test statistics			1.860				
η <sup>2</sup>			0.113				
p			0.150*				

<sup>A</sup>: Groups sharing this superscript before thermoforming are not significantly different.  
<sup>B</sup>: Groups sharing this superscript after thermoforming are not significantly different  
\*One-way analysis of variance (ANOVA). Statistical significance:  $p<0.05$ .  
Q, robust ANOVA test statistic; SD, standard deviation; η<sup>2</sup>, effect sizes; B, brand; T, thermoforming; Min, minimum; Max, maximum.



**Table 3.** Comparison of average roughness, root mean square roughness, and maximum height of the profile values by brand and thermoforming

	Brand	Thermoforming			Difference Mean±SD Median (Min-Max)
		Before Mean±SD Median (Min-Max)	After Mean±SD Median (Min-Max)		
Ra	Duran	0.02±0.01 0.02 (0.02-0.04) <sup>AB</sup>	0.06±0.02 0.06 (0.03-0.09) <sup>AC</sup>		0.04±0.02 0.03 (0.01- 0.07) <sup>ab</sup>
	Atmos	0.03±0.02 0.02 (0.02-0.10) <sup>B</sup>	0.05±0.03 0.04 (0.02-0.13) <sup>ABC</sup>		0.02±0.04 0.02 (0.06-0.11) <sup>b</sup>
	Taglus	0.02±0.01 0.02 (0.02-0.04) <sup>B</sup>	0.15±0.11 0.12 (0.05-0.4) <sup>ABC</sup>		0.13±0.11 0.09 (0.01-0.38) <sup>a</sup>
	Lumex-G	0.03±0.02 0.02 (0.02-0.10) <sup>B</sup>	0.10±0.06 0.09 (0.05-0.26) <sup>C</sup>		0.07±0.07 0.06 (0.05-0.24) <sup>ab</sup>
		<b>B</b>	<b>T</b>	<b>B*T</b>	
	Q	2.55	17.04	9.74	
	Test statistics				14.761
	η <sup>2</sup>	0.080	0.162	0.249	0.304
	p	0.054	<0.001	0.021	0.002*
Rz	Duran	0.24±0.10 0.23 (0.14-0.49)	0.45±0.24 0.35 (0.25-0.67)		0.18±0.23 0.14 (0.10-0.52) <sup>ab</sup>
	Atmos	0.28±0.09 0.26 (0.19-0.54)	0.34±0.16 0.3 (0.17-0.64)		0.06±0.19 0.05 (0.32-0.4) <sup>b</sup>
	Taglus	0.18±0.05 0.16 (0.13-0.3)	0.96±0.88 0.6 (0.27-2.73)		0.82±0.86 0.44 (0.11-2.57) <sup>a</sup>
	Lumex-G	0.25±0.10 0.23 (0.14-0.45)	0.65±0.30 0.54 (0.3-1.53)		0.38±0.35 0.3 (0.04 -1.16) <sup>ab</sup>
		<b>B</b>	<b>T</b>	<b>B*T</b>	
	Q	0.522	4.358	3.664	
	Test statistics				13.355
	η <sup>2</sup>	0.017	0.047	0.111	0.235
	p	0.667	<b>0.037*</b>	0.300	<b>0.004*</b>
Rq	Duran	0.04±0.02 0.03 (0.02-0.05)	0.08±0.02 0.07 (0.04-0.11)		0.04±0.03 0.03 (0-0.09) <sup>b</sup>
	Atmos	0.03±0.01 0.03 (0.02-0.13)	0.06±0.04 0.06 (0.03-0.15)		0.03±0.05 0.02 (0.08-0.12) <sup>b</sup>
	Taglus	0.03±0.01 0.03 (0.02-0.05)	0.33±0.23 0.19 (0.06-0.52)		0.18±0.14 0.16 (0.02-0.5) <sup>a</sup>
	Lumex-G	0.04±0.02 0.03 (0.02-0.07)	0.13±0.07 0.12 (0.07-0.34)		0.1±0.08 0.08 (0.01-0.3) <sup>ab</sup>
		<b>B</b>	<b>T</b>	<b>B*T</b>	
	Q	2.00	15.57	7.25	
	Test statistics				16.375
	η <sup>2</sup>	0.064	0.150	0.198	0.304
	p	0.112	<b>&lt;0.001</b>	0.064	<b>0.001*</b>

\*Kruskal-Wallis H Test. Statistical significance: p&lt;0.05.

<sup>A-B</sup>: Interactions with the same superscript letter are not significantly different.<sup>a-b</sup>: Interactions with the same superscript letter are not significantly different.<sup>A-C</sup>: Same uppercase superscripts in a column indicate no significant difference before thermoforming.<sup>a-b</sup>: Same lowercase superscripts in a column indicate no significant difference after thermoforming.Q, robust ANOVA test statistics; Min, minimum; Max, maximum; η<sup>2</sup>, effect sizes; B: brand; T, thermoforming.

Atmos was significantly lower than that for Taglus and did not differ significantly from those for Duran and Lumex-G.

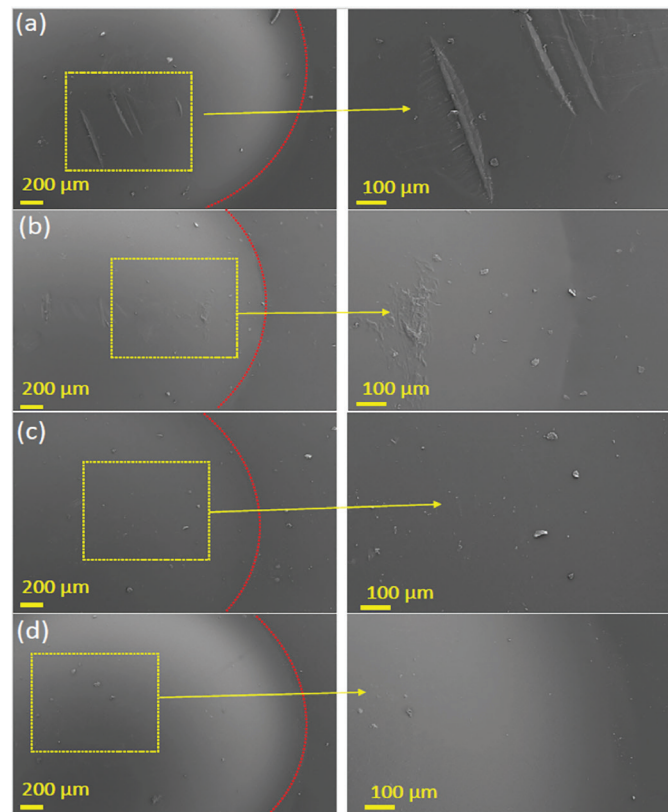
The main effect of brand on median Rq values was not statistically significant ( $p=0.112$ ). By contrast, the main effect of thermoforming on Rq values was statistically significant ( $p<0.001$ ). However, the interaction effect of brand and thermoforming on Rq values did not reach statistical significance ( $p=0.064$ ). A statistically significant difference was also observed in the median Rq values among brands ( $p=0.001$ ). Furthermore, median Rq values for Duran, Atmos, Taglus, and Lumex-G were 0.03 (0-0.09), 0.02 (0.08-0.12), 0.16 (0.02-0.5), and 0.08 (0.01-0.3), respectively. The Rq value for Taglus was similar to that of Lumex-G, but was significantly different from those of Duran and Atmos.

The morphology of Taglus, Lumex-G, Atmos, and Duran after thermoforming was examined by SEM (Figure 3). Surface roughness measurements were obtained from the heat-treated centers. The Taglus and Lumex groups exhibited greater surface roughness than the Atmos and Duran groups.

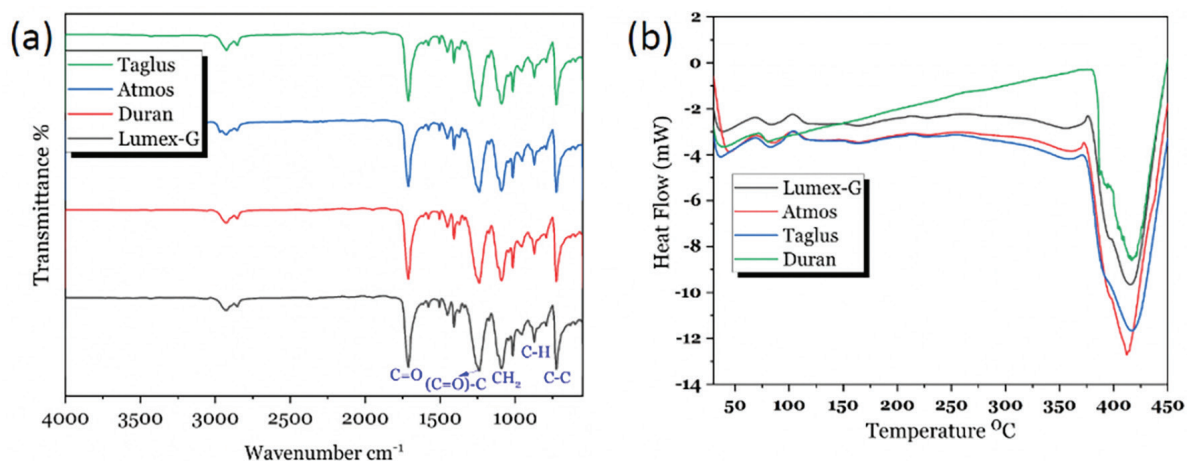
Characteristic bond types in Taglus, Lumex-G, Atmos, and Duran were identified by FTIR analysis (Figure 4a). The peaks indicate that all groups have similar bond types, consistent with polyethylene (PE) terephthalate, as specified in the manufacturers' product datasheets.<sup>22</sup> The peaks observed at 1,712 and 1,238  $\text{cm}^{-1}$  were attributed to the C=O and (C=O)-C functional groups, whereas  $\text{CH}_2$ , C-H, and C-C bond vibrations were detected at 1,095, 875, and 721  $\text{cm}^{-1}$ , respectively. The FTIR spectra confirmed that the four materials have comparable chemical structures.

To evaluate thermal behavior, two complementary thermal analysis techniques were employed. DSC was used to assess the phase transition properties of the materials. The DSC results indicated that all materials had similar melting behavior, with the onset of melting occurring at around 370  $^{\circ}\text{C}$ -375  $^{\circ}\text{C}$  and

peak melting temperatures reaching approximately 410  $^{\circ}\text{C}$ -415  $^{\circ}\text{C}$ . Thermogravimetric analysis (TGA) was performed to determine the thermal degradation characteristics. According to the TGA results, the onset of thermal degradation began at approximately 370  $^{\circ}\text{C}$  and was completed by 440  $^{\circ}\text{C}$ -445  $^{\circ}\text{C}$ , indicating that all the tested materials possessed high thermal stability before substantial decomposition occurred.



**Figure 3.** Scanning electron microscopy images of the vacuum-formed retainer materials: (a) Taglus, (b) Lumex-G, (c) Atmos, and (d) Duran.



**Figure 4.** Fourier transform infrared spectroscopy analysis of the vacuum-formed retainer materials. (a) FTIR spectra of the groups are presented. (b) DSC thermograms of the groups are presented. FTIR, Fourier transform infrared spectroscopy; DSC, differential scanning calorimetry.

## DISCUSSION

Maintaining the final tooth positions achieved through orthodontic treatment is critical for long-term treatment success. Although the duration of retention protocols varies with malocclusion type, an average retention period of one year is generally accepted. During this period, VFRs are exposed to masticatory forces and oral fluids, and their mechanical and surface properties play a key role in meeting aesthetic and functional expectations.<sup>6,16</sup> Therefore, analyzing changes in the surface hardness, roughness, and chemical structure of VFRs both in their initial state and after thermoforming is essential for evaluating their clinical effectiveness.<sup>6,12</sup> This study compared the mechanical properties of different VFR materials before and after thermoforming. Four thermoplastic retainer materials (Duran, Taglus, Atmos, and Lumex-G) were selected because they are among the most widely used in clinical orthodontics and represent different polymer types (PET-G, PET-G/ PE blend, PC, and copolyester, respectively). The findings of this study indicate that certain material properties are sensitive to thermoforming, resulting in a partial rejection of the null hypothesis.

Compositional analysis revealed that Duran and Lumex-G are primarily composed of PET-G, whereas Taglus contains both PET-G and PE. In contrast, Atmos is composed of copolyester. Differences in material composition may substantially affect mechanical stability, as PET-G has been reported to confer durability owing to its amorphous structure, chemical resistance, and thermal stability.<sup>6-8,16</sup> Albilali et al.<sup>8</sup> reported that PET-G-based materials maintained high hardness values even after thermal treatment. Although no statistically significant differences were observed between groups in the present study, hardness generally decreased following thermoforming. This reduction can be attributed to rearrangements of polymer chains in amorphous thermoplastics that compromise mechanical durability.<sup>16</sup> Furthermore, long-term exposure to oral fluids and prolonged water sorption may further accelerate reductions in hardness and elasticity, suggesting that *in vitro* thermoforming alone may underestimate the extent of clinical degradation.<sup>23</sup>

Additionally, the Taglus brand's PET-G and PE content may have contributed to its lower hardness values. PE is suggested to have lower hardness than PET-G, potentially reducing the blend's overall hardness. In contrast, the Atmos and Duran groups retained higher hardness values even after thermoforming. This finding may be attributed to the molecular homogeneity and thermal stability of pure copolyester and PET-G within their polymer structures. Our findings are largely consistent with the literature; however, the lower hardness values observed in the Taglus group may be related to its PE content and warrant further investigation.

The impact of thermoforming on the surface roughness of VFR materials is critical to both aesthetic performance and clinical application. Thermoforming may increase surface

roughness due to the rearrangement of polymer chains. Such morphological alterations can lead to surface irregularities that ultimately reduce the long-term durability of orthodontic appliances.<sup>8,11</sup> Moreover, increased surface roughness values has been associated with increased bacterial adhesion and plaque accumulation on VFRs, increasing potential periodontal risk.<sup>17</sup> SEM analysis supported these findings, as the Taglus and Lumex-G surfaces exhibited greater surface irregularities than Atmos and Duran, consistent with the profilometry results. Recent studies have also investigated new thermoplastic materials, such as TPU and polyolefins, which may offer improved resistance to surface degradation compared with that of PET-G; however, clinical validation remains limited.<sup>20</sup>

In the present study, a significant increase in surface roughness was observed after thermoforming, particularly in the Taglus and Lumex-G groups. This finding suggests that the molecular homogeneity of these materials might be disrupted during thermal processing. Specifically, the PE content in Taglus, which is less hard than PET-G, may have contributed to this result by weakening the material's structural stability. Lumex-G, although PET-G-based, may be more susceptible to thermal rearrangement due to differences in polymer chain alignment or manufacturing processes.

These results align with previous studies indicating that thermoforming often leads to surface irregularities, which can reduce the long-term durability of orthodontic appliances. For example, Ahn et al.<sup>16</sup> demonstrated that thermal aging processes significantly altered the molecular structure and surface morphology of VFR materials, resulting in increased surface roughness. Furthermore, this roughness may serve as a site for bacterial adhesion, compromising the appliance's clinical effectiveness.<sup>15-18</sup> Gardner et al.<sup>11</sup> reported that rough surfaces could interact with oral fluids and food residues, leading to increased microbial plaque accumulation, which may negatively impact periodontal health. Therefore, minimizing the effects of thermoforming on surface roughness is critical for material selection and manufacturing process design.

In the present study, changes in surface and chemical structure of VFR materials after thermoforming were evaluated using SEM and FTIR. The SEM results revealed that the surfaces in the Taglus and Lumex-G groups exhibited greater irregularities than those of the Atmos and Duran groups. This finding is consistent with the observed increases in surface roughness and may be associated with disruptions in the Taglus and Lumex-G materials' molecular homogeneity. These surface irregularities are thought to result from microscopic rearrangements of polymer chains, and the SEM observations are consistent with the surface roughness results.

Moreover, FTIR analysis revealed that all materials shared similar functional groups, with characteristic peaks consistent with PET-G-based structures. Specifically, the peaks observed at 1,712 and 1,238 cm<sup>-1</sup> correspond to the C=O and (C=O)-C functional groups, respectively. However, the surface and

thermal properties of materials can vary due to processing parameters such as time, temperature, and pressure as well as other physical effects. Using the same PET-G raw materials may produce identical bond types in FTIR analysis, whereas different processing parameters such as temperature, pressure, and duration may produce different thermal and physical properties. These minor differences were evident in the SEM and DSC results. Even if FTIR indicates chemical stability after thermoforming, small differences in surface roughness, hardness, and decomposition temperatures can be attributed to physical defects such as porosity. This observation also highlights a limitation of FTIR analysis: while it effectively detects major chemical structures, it may not capture subtle molecular-level differences, especially in copolymer blends. For example, the increase in surface roughness observed in the Taglus group could be due to microscopic rearrangements in amorphous regions induced by the presence of PE. Such minor chemical changes may not be detectable by FTIR, but can still manifest as alterations in surface morphology.

### Study Limitations

The inability to evaluate, *in vivo*, the physical changes induced by chewing forces on VFR materials, the lack of long-term assessment of the effects of saliva and other oral fluids, and the fact that thermoforming was performed by a single operator represent the primary limitations of this study. In addition, no artificial aging procedures (e.g., thermal cycling, mechanical loading, or water sorption tests) were applied, limiting extrapolation of the findings to long-term clinical performance. Furthermore, the assessment of material properties was limited to Vickers hardness and two-dimensional profilometry. The absence of advanced characterization techniques, such as three-dimensional surface topography analysis or nanoindentation, may have restricted a more detailed understanding of microstructural changes. These limitations should be taken into consideration when interpreting the present findings, and future studies addressing these aspects would provide a more comprehensive evaluation of thermoplastic retainer materials.

### CONCLUSION

Following thermoforming, a significant increase in surface roughness was observed in the Taglus and Lumex-G groups, whereas the Atmos and Duran groups largely maintained surface stability. DSC and FTIR analyses indicated that no major chemical or thermal degradation occurred in any of the materials, while SEM analysis confirmed surface-level morphological changes, particularly in the Taglus and Lumex-G groups. Within the limitations of this *in vitro* study, PET-G-based materials with high surface homogeneity appeared to demonstrate better surface stability.

### Ethics

**Ethics Committee Approval:** As the study did not involve the use of human or animal materials, ethical approval was not required.

**Informed Consent:** Not applicable.

### Footnotes

**Author Contributions:** Concept - Y.T., N.F.; Design - Y.T., M.T.; Data Collection and/or Processing - Y.T., M.T.; Analysis and/or Interpretation - M.T., Y.A.; Literature Search - Y.T., M.T.; Writing - Y.T., N.F.

**Conflict of Interest:** The authors have no conflicts of interest to declare.

**Financial Disclosure:** The authors declared that this study received no financial support.

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