

Research Article

Characterization of Esthetic Orthodontic Wires Made from Glass-Fiber-Reinforced Thermoplastic Containing High-Strength, Small-Diameter Glass Fibers

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In this work, we investigated the properties of a glass-fiber-reinforced thermoplastic (GFRTTP) composed of small-diameter ($\phi = 5 \mu\text{m}$), high-strength glass (T-glass) fibers and polycarbonate for esthetic orthodontic wires formed using pultrusion. After fabricating such GFRTTP round wires, the effects of varying fiber diameter (5 to 13 μm) on the mechanical properties, durabilities, and color stabilities were evaluated. The results showed that the mechanical properties of GFRTTPs tend to increase with decreasing fiber diameter. Additionally, it was confirmed that the present GFRTTP wires containing T-glass fibers have better flexural properties than previously reported GFRTTP wires containing E-glass fibers. Meanwhile, thermocycling did not significantly affect the flexural properties of the GFRTTP wires. Furthermore, the GFRTTP wires showed color changes lower than the acceptable threshold level for color differences on immersion in coffee. From these results obtained in the present work, the GFRTTP wires containing high-strength glass fibers have excellent properties for orthodontic applications. Our findings suggest that the GFRTTPs might be applied to all phases of orthodontic treatment because their properties can be tuned by changing the fiber properties such as fiber type and diameter.

1. Introduction

Orthodontic wires are widely used in orthodontic appliances throughout treatment. These wires are made from metal alloys such as stainless steel (SS), cobalt-chromium-nickel alloy (Co-Cr), β -titanium alloy (β -Ti), and nickel-titanium alloy (Ni-Ti) [1]. However, one disadvantage of these metallic wires is the opacity of the metal, which deters many patients from undergoing orthodontic treatment. There is therefore a demand for transparent esthetic orthodontic wires. The use of glass-fiber-reinforced thermoplastics (GFRTTPs) is one possible solution to this problem; the transparent or translucent

appearance of GFRTTP arch wires is considered to be especially attractive.

In a previous study, esthetic orthodontic wires were produced by pultrusion of GFRTTPs composed of a polycarbonate matrix reinforced with E-glass fibers [2, 3]. Polycarbonate has been widely used in orthodontic appliances such as esthetic brackets and clear retainers [4], and it was used as a matrix of the GFRTTP because of its high transparency, low weight, and high heat resistance [2, 5]. The previous study confirmed that the GFRTTP wires are more transparent than conventional metallic wires [2]. Moreover, field emission scanning electron microscopy and scanning

probe microscopy analysis indicated that the surface roughnesses of the GFRTTP wires are smoother than those of the β -Ti wire [3]. Furthermore, the GFRTTP wires had frictional and flexural properties similar to those of the Ni-Ti wires [2, 3]. These findings show that GFRTTP wires composed of polycarbonate and E-glass fibers are promising for use in orthodontic appliances.

The properties of GFRTTPs can be tuned by changing parameters such as the fiber type, diameter, and volume fraction [6, 7]. Materials reinforced with fibers such as carbon, glass, aramid, and boron are used as alternatives to metals in various industries [8]. Glass fibers are transparent and particularly suitable for esthetic dentistry [9]. Different types of glass fibers are available, and E-glass and high-strength glass (known as S-glass in the USA, T-glass in Japan, and R-glass in Europe) fibers are commonly used [8, 9]. High-strength glass fibers have a tensile strength one-third greater than that of E-glass fibers. These glass fibers are produced by flowing molten glass through holes in a high-temperature alloy bushing; the temperature, and thus the viscosity, is carefully controlled. The average filament diameter ranges from 4.44 to 13.3 μm [8]. The mechanical properties of GFRTTPs improve with decreasing fiber glass diameter [6, 10]. However, there are no research reports regarding the fabrication and characterization of the GFRTTP orthodontic wires containing high-strength, small-diameter glass fibers.

On the basis of these results, in the present study, GFRTTP wires composed of high-strength glass (T-glass) fibers of small diameter (5 μm) and polycarbonate were fabricated using a pultrusion method, for use in orthodontic treatment. The effects of the fiber type and diameter on the properties of the GFRTTP wires, such as mechanical properties, durabilities, and color stabilities, were evaluated. The hypothesis was that the mechanical properties of the present GFRTTP wires containing T-glass fibers would be superior to those of previously reported GFRTTP wires containing E-glass fibers.

2. Materials and Methods

2.1. Preparation of Materials. GFRTTP wires were fabricated using a previously reported pultrusion technique [2]. A polycarbonate (H4000; Mitsubishi Engineering-Plastics Corp., Tokyo, Japan) was used as the thermoplastic matrix. T-glass fiber filaments (Nittobo Co., Fukushima, Japan) were used for unidirectional reinforcement of the polycarbonate matrix. Table 1 shows the mass percentage compositions of E-glass and T-glass fibers [11]. We prepared GFRTTP wires with T-glass fibers of three diameters, that is, 13 μm (GFRTTP-13), 7 μm (GFRTTP-7), and 5 μm (GFRTTP-5), and investigated the effects of the fiber diameter on the GFRTTP wire properties. The volume fraction of glass fibers in the GFRTTPs was ~ 0.3 . Round GFRTTP wires of diameter 0.45 mm (0.018 inch) and length 36 cm were cut from the pultruded samples.

2.2. Frictional Tests. The frictional behaviors of the GFRTTP wires were investigated using a computer-controlled Instron

TABLE 1: Compositions (wt.%) of E-glass and T-glass.

Composition	E-glass	T-glass
SiO ₂	52–56	64–66
Al ₂ O ₃	12–16	24–26
CaO	20–25	—
MgO		9–11
R ₂ O	0–0.8	—
B ₂ O ₃	5–10	—

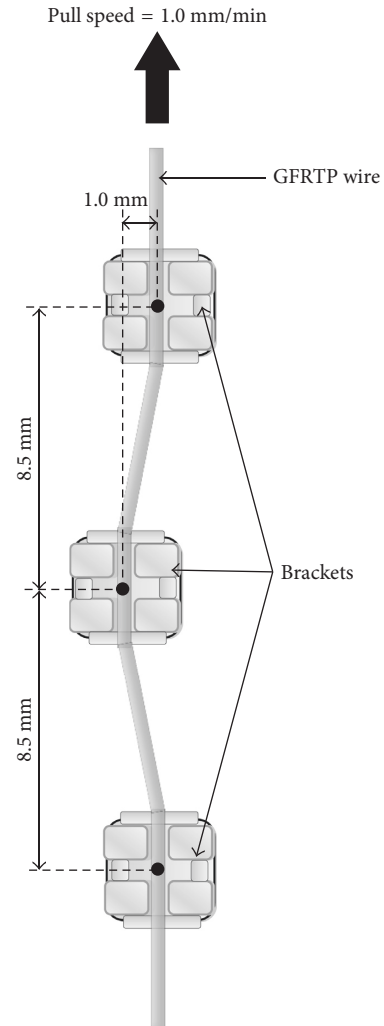


FIGURE 1: Illustration of the frictional testing system.

testing machine (TG-5kN; Minebea, Tokyo, Japan) with jig-fixed brackets, as described in a previous report [3]. Before the test, a jig was prepared using a 195 mm \times 54 mm \times 2.95 mm acrylic plate, on which three brackets (with a 0.022 inch \times 0.028 inch slot) were bonded using cyanoacrylate adhesive. As shown in Figure 1, the positions of the three brackets bonded on the acrylic jig were set. The distance between brackets was 8.5 mm, simulating the upper right lateral incisor, canine, and first premolar brackets. The central bracket was positioned 1.0 mm more to the left than the other two brackets along a vertical line

to give three unaligned horizontal brackets. A ceramic bracket (Sincere Brace, Kuraray Noritake Dental Inc., Niigata, Japan) made from zirconia was used as an esthetic bracket. For the friction tests, the wires were cut into segments of length 18 cm. The wires were ligated using elastomeric ligatures (Chain Elastic Ligature Clear, American Orthodontics). Tests were performed using an Instron machine with the cross head speed set at 1 mm/min; the wire was pulled through the brackets for 2 min, and the maximum loading, that is, the static frictional force, was measured using a load cell of 5 kN. A new wire, a bracket, and a fresh ligature were used for each combination and then discarded to eliminate the influence of wear. The frictional test results were reported as the average values for 10 specimens ($n = 10$).

2.3. Three-Point Bending Tests. Three-point bending tests were performed at a constant loading rate of 1 mm/min with a span length of 16 mm using a computer-controlled Instron testing machine, according to the previous study [2]. The GFRTTP wires were of length 30 mm. The flexural properties of the GFRTTP wires such as the flexural strength and modulus were obtained from three-point bending tests. The experimental values for the GFRTTP wires were the averages for 12 specimens ($n = 12$).

2.4. Thermal Cycling. The stability of the mechanical properties of the GFRTTP wires was determined by thermocycling between 5 and 55°C in deionized water for 600 or 1200 cycles using a thermal cycling machine (Thomas, Tokyo, Japan), according to the previous study [2]. In these thermocycling tests, each GFRTTP wire was immersed in one bath for 60 s and then transferred to another bath within 5 s. After thermocycling, the flexural properties were again evaluated using three-point bending tests and compared with those before thermocycling; the experimental values were the averages for 12 specimens ($n = 12$).

2.5. Weight Change during Immersion. GFRTTP wires of length 30 mm were immersed for 4 weeks in 20 mL of distilled water (DW) of pH 6.0 at 37°C in a Teflon-sealed polystyrene bottle. The DW was replaced every week to expose the samples to a fresh solution. We determined the weight change by weighing the samples before and after immersion, using a precision digital balance (AG285; Mettler-Toledo GmbH, Greifensee, Switzerland). Water sorption and solubility were calculated using the following equations [12]:

$$\text{Water sorption (\%)} = \frac{(w_2 - w_3)}{w_1} \times 100, \quad (1)$$

$$\text{Water solubility (\%)} = \frac{(w_1 - w_3)}{w_1} \times 100, \quad (2)$$

where w_1 is the weight of the sample before immersion, w_2 is the weight of the sample after immersion, and w_3 is the weight of the sample which was dried after immersion. The

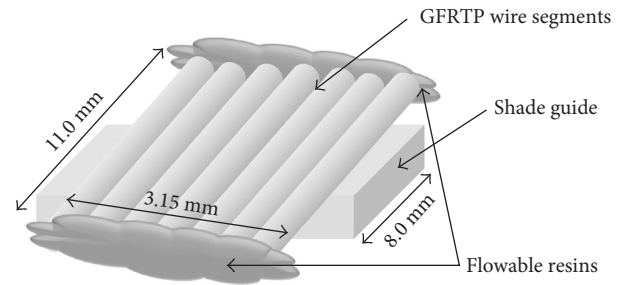


FIGURE 2: Illustration of the GFRTTP sample devised for colorimetric measurement.

experimental values were the averages of 5 measurements ($n = 5$).

2.6. Color Stability Evaluation. Direct colorimetric examination of the as-fabricated small-diameter GFRTTP wires was impossible; samples with a total width of at least 3 mm were needed for the color to be determined colorimetrically, as described later. Accordingly, GFRTTP samples were prepared by tightly arranging seven wire segments (length 11 mm and diameter 0.45 mm) by fixing both edges with a commercially available flowable resin (Filtek™ Flow, 3M ESPE, MN, USA), as shown in Figure 2.

According to the previous study [13], the GFRTTP samples were immersed for 4 weeks in 20 mL of coffee (NESCAFE Excella®, Nestlé Japan Ltd., Hyogo, Japan), which was used as a staining solution, in a Teflon-sealed polystyrene bottle at 37°C; the coffee solution was replaced weekly. After staining, the samples were washed with DW and dried with paper towels. The color changes after immersion for 24 h and 1, 2, and 4 weeks were determined colorimetrically (ShadeEye NCC, Shofu Inc., Kyoto, Japan) using a shade tab (A3, SOLARE, GC Corp., Tokyo, Japan). This device contains a pulsed xenon lamp as an optical light source and a three-component silicon photocell as the optical sensor. The colorimetric measurements were performed by contacting the measurement tip with the GFRTTP sample and using the shade tab as a reference. The values were the averages of 10 samples ($n = 10$), with each sample examined three times.

The color parameters were expressed using the Commission Internationale de l'Eclairage (CIE) $L^*a^*b^*$ color space system, relative to an illuminant standard, D65. In this three-dimensional color space, the three axes are L^* , a^* , and b^* . The L^* value is a measure of the lightness of an object and is quantified on a scale such that perfect black has an L^* value of 0 and a perfect reflecting diffuser has an L^* value of 100. The a^* value is a measure of redness ($+a^*$) or greenness ($-a^*$). The b^* value is a measure of yellowness ($+b^*$) or blueness ($-b^*$).

The difference between the colors (ΔE^*) before and after immersion was calculated as [13, 14]

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}. \quad (3)$$

The ΔE^* values were converted to the National Bureau of Standards (NBS) units using the following equation [13, 15]:

$$\text{NBS units} = \Delta E^* \times 0.92. \quad (4)$$

The values are listed in Table 2 [13].

2.7. Statistical Analysis. The experimental results were evaluated using analysis of variance and tested using Scheffe's multiple comparison test among the means, with $p < 0.05$ considered significant.

3. Results and Discussion

In the present study, the properties of GF RTP wires containing T-glass fibers of various diameters and polycarbonate for esthetic orthodontic appliances were investigated.

3.1. Frictional Properties. In terms of biomechanics, the frictional properties of orthodontic wires are an important parameter in achieving optimum tooth movement. In particular, the efficiency of tooth movement in the leveling stage can be adversely affected by friction generated between the wire and bracket [16]. Table 3 shows the frictional forces of the GF RTP wires against esthetic brackets made from zirconia. There were significant differences between the frictional forces of GF RTP-5 and GF RTP-13 and between those of GF RTP-5 and GF RTP-7 ($p < 0.05$). Generally, the frictional properties of orthodontic wires are affected by their surface characteristics [3, 17]. The surfaces of all the fabricated GF RTP wires are the same, that is, polycarbonate, because most of the reinforcing glass fibers are embedded in the polycarbonate matrix [2, 3]. This suggests that the surface properties of GF RTP wires do not cause differences among their frictional properties.

The frictional forces are affected not only by the surface roughness but also by the material's stiffness [3], that is, contact forces between the two surfaces (i.e., the wire and the bracket) during sliding in frictional tests increase with increasing wire's stiffness [17]. In terms of the flexural properties of the GF RTP wires, described later, the flexural modulus tends to increase with decreasing fiber diameter (Table 4). It is therefore thought that the frictional force of GF RTP-5 is significantly higher than that of GF RTP-13 and GF RTP-7 (Table 3).

A comparison with the results of a previous study of currently used metallic wires such as SS and Co-Cr [3] shows that the frictional forces (3.62–4.73 N) of GF RTP wires containing T-glass fibers of various diameters (5 to 13 μm) are lower than those of SS wires (7.53 N) and Co-Cr wires (7.62 N). Generally, minimizing the friction between the wire and bracket during orthodontic tooth movement is important for optimum orthodontic treatment. GF RTP wires therefore give superior sliding mechanics with low frictional resistance between the wire and bracket during orthodontic treatment.

3.2. Flexural and Durability Properties. The flexural properties of the GF RTP wires before and after thermocycling are

TABLE 2: National Bureau of Standards (NBS) ratings.

NBS unit	Critical remarks of color differences	
0.0–0.5	Trace	Extremely slight change
0.5–1.5	Slight	Slight change
1.5–3.0	Noticeable	Perceivable
3.0–6.0	Appreciable	Marked change
6.0–12.0	Much	Extremely marked change
12.0 or more	Very much	Change to other color

TABLE 3: Frictional forces of GF RTP wires against brackets.

Sample	Frictional force (N)
GF RTP-13	3.62 ± 0.33^a
GF RTP-7	3.86 ± 0.21^b
GF RTP-5	$4.73 \pm 0.28^{a,b}$

Mean values with the same superscripts are significantly different from each other ($p < 0.05$).

shown in Table 4. The flexural strengths of GF RTP-13, GF RTP-7, and GF RTP-5 after different numbers of cycles ranged from 913.3 to 958.5, from 999.2 to 1120.1, and from 1043.5 to 1126.2 MPa, respectively. The flexural moduli of GF RTP-13, GF RTP-7, and GF RTP-5 after different numbers of cycles ranged from 34.5 to 35.4, from 38.4 to 40.5, and from 38.3 to 42.6 GPa, respectively. Before thermocycling, the flexural properties of GF RTP-5 were better than those of GF RTP-13 ($p < 0.05$), that is, the flexural properties of GF RTP wires tended to improve with decreasing fiber diameter. This is because the number of defects in a fiber decreases with decreasing fiber diameter [6]. The trends in the flexural properties of GF RTP wires containing T-glass fibers with changes in the diameter agreed with those previously reported for GF RTP wires containing E-glass fibers [2].

A previous thermocycling study of GF RTP wires with E-glass fibers of diameter 13 μm showed that the flexural strength and modulus were 690.3 MPa and 25.4 GPa, respectively [2]. The flexural strength and modulus of the GF RTP wire with T-glass fibers of diameter 13 μm in the present study were 958.5 MPa and 35.0 GPa, respectively. The flexural properties of GF RTP wires with T-glass fibers are therefore superior to those of GF RTP wires with E-glass fibers. Generally, most of the glass fibers used for reinforcement are of E-glass. In dental applications, there are only a few reports regarding the properties of GF RTPs containing high-strength glass (S-glass) fibers [18]. Such S-glass fibers have a higher strength, modulus, and hardness than E-glass fibers, and their composition (about 65% SiO_2 , 25% Al_2O_3 , and 10% MgO) is similar to that of T-glass fibers included in the high-strength glass group [8, 9, 11, 18, 19]. As expected, the properties of the present GF RTP wires containing T-glass fibers are better than those of previously reported GF RTP wires containing E-glass fibers. Our hypothesis was confirmed. In other words, GF RTP wires as tailored orthodontic appliances might be used during all phases of orthodontic treatment, since their properties can

TABLE 4: Flexural properties of GFRTPs after thermocycling.

	0 cycles		600 cycles		1200 cycles	
	Strength (MPa)	Modulus (GPa)	Strength (MPa)	Modulus (GPa)	Strength (MPa)	Modulus (GPa)
GFRTTP-13	958.5 ± 137.4 ^{a,b}	35.0 ± 5.2 ^a	938.9 ± 102.2	34.5 ± 5.6	913.3 ± 122.1 ^a	35.4 ± 5.9
GFRTTP-7	1120.1 ± 197.0 ^a	40.5 ± 9.2	1037.0 ± 123.2	39.7 ± 6.9	999.2 ± 56.8	38.4 ± 6.5
GFRTTP-5	1126.2 ± 92.5 ^b	42.6 ± 5.3 ^a	1043.5 ± 81.5	40.0 ± 3.6	1060.4 ± 102.4 ^a	38.3 ± 5.9

Mean values with the same superscripts are significantly different from each other ($p < 0.05$).

TABLE 5: Water sorption values and solubilities after immersion for 4 weeks.

Sample	Water sorption (%)	Water solubility (%)
GFRTTP-13	0.67 ± 0.12	0.07 ± 0.11
GFRTTP-7	0.56 ± 0.18	0.10 ± 0.07
GFRTTP-5	0.56 ± 0.20	0.10 ± 0.10

No significant differences in both water sorption and water solubility were observed after 4 weeks of immersion ($p > 0.05$).

TABLE 6: Color differences measured in ΔE^* and NBS units after immersion in coffee solution.

	24 hours		1 week		2 weeks		4 weeks	
	ΔE^*	NBS units	ΔE^*	NBS units	ΔE^*	NBS units	ΔE^*	NBS units
GFRTTP-13	1.47 ± 0.52	1.36 ± 0.48	1.05 ± 0.46 ^a	0.97 ± 0.42 ^a	1.10 ± 0.59 ^a	1.01 ± 0.54 ^a	0.83 ± 0.52 ^a	0.77 ± 0.48 ^a
GFRTTP-7	1.34 ± 0.39	1.23 ± 0.36	1.11 ± 0.23 ^b	1.02 ± 0.21 ^b	1.00 ± 0.36 ^b	0.92 ± 0.32 ^b	0.84 ± 0.36 ^b	0.77 ± 0.33 ^b
GFRTTP-5	1.84 ± 0.62	1.70 ± 0.57	2.20 ± 0.51 ^{a,b}	2.02 ± 0.47 ^{a,b}	2.07 ± 0.43 ^{a,b}	1.90 ± 0.39 ^{a,b}	1.94 ± 0.48 ^{a,b}	1.79 ± 0.45 ^{a,b}

ΔE^* values were converted to NBS units using the following equation: NBS units = $\Delta E^* \times 0.92$; mean values with the same superscripts are significantly different from each other ($p < 0.05$); for each GFRTTP, there were no significant differences in color change data across all immersion periods ($p > 0.05$).

be tuned by changing the fiber properties such as fiber type and diameter.

In terms of durability, no significant differences in the flexural properties of the GFRTTP wires were observed after any number of cycles ($p > 0.05$). This indicates that thermal cycling did not degrade the GFRTTP wires. Furthermore, the data in Table 5 show that there were no significant differences among the water sorption abilities and solubilities of the GFRTTP wires ($p > 0.05$). This is because polycarbonate, which was used as the matrix resin for the GFRTTPs, is a low-sorption material [20]. The results of the durability tests suggest that the mechanical properties of the GFRTTP wires would remain stable during the course of orthodontic treatment.

3.3. Color Stability. It is difficult to discern small degrees of color change caused by penetration of colored solutions, using the human eye. Among colored solutions such as coffee, tea, and coke, coffee is the most chromogenic agent [21]. Therefore, applying the same method as used by a previous study [13], the color stability of the GFRTTP wires during the 4 weeks of immersion in coffee solution, in terms of two units (ΔE^* and NBS), was evaluated. Table 6 shows the color differences reported using ΔE^* and NBS units for the GFRTTP wires after various immersion periods. The ΔE^* values after different immersion periods for GFRTTP-13, GFRTTP-7, and GFRTTP-5 ranged from 0.83 to 1.47, from 0.84 to 1.34, and from 1.84 to 2.20, respectively. Values of 3.7 ΔE^* or 2.6 ΔE^* units have been suggested to be clinically

unacceptable [15, 22]. All the experimental values obtained for the GFRTTPs in this study were lower than 2.2 ΔE^* units, ranging from 0.83 to 2.20 (Table 6). Additionally, there were no significant differences among the measured ΔE^* values for any of the GFRTTP wires during different immersion periods ($p > 0.05$).

The degrees of color difference for the GFRTTP wires were also assessed in terms of NBS units. The NBS units after different immersion times for GFRTTP-13, GFRTTP-7, and GFRTTP-5 ranged from 0.77 to 1.36, from 0.77 to 1.23, and from 1.70 to 2.02, respectively. The data in Table 2 show that the color change values for GFRTTP-13 and GFRTTP-7 across all immersion periods were lower than 1.5, and only slight color changes were observed in terms of NBS units. The color change values for GFRTTP-5 across all immersion periods were lower than 3.0, and noticeable color changes were observed in NBS units. All the GFRTTP wires gave color change values lower than the threshold level of 3.0 in NBS units; therefore, the color changes were clinically acceptable [23]. Additionally, there were no significant differences among the color change values for the GFRTTP wires across all immersion periods ($p > 0.05$). The results obtained in both ΔE^* and NBS units indicate that discoloration of GFRTTP wires will not occur during orthodontic treatment.

4. Conclusions

This study investigated the properties of GFRTTP wires composed of T-glass fibers and polycarbonate, for use in

orthodontic treatment. The authors' interpretation of the results is summarized as follows:

- (1) The frictional force of GFRTTP-5 against zirconia brackets is significantly higher than that of GFRTTP-13 and GFRTTP-7; however, the frictional forces of these GFRTTP wires are lower than those previously reported for SS and Co-Cr wires.
- (2) The flexural properties of the present GFRTTP wires containing T-glass fibers are better than those previously reported for GFRTTP wires containing E-glass fibers. Additionally, the flexural properties improve with decreasing fiber diameter, that is, GFRTTP-5 has better flexural properties than GFRTTP-13 or GFRTTP-7.
- (3) The results of thermocycling tests show no significant differences in the flexural properties of the GFRTTP wires after 600 or 1200 cycles. Moreover, the weight changes for all the GFRTTP wires were stable during 4 weeks of immersion in DW at 37°C.
- (4) After immersion in coffee solution for 4 weeks, the color change values for all the GFRTTP wires were lower than the threshold levels in ΔE^* and NBS units, that is, the color changes were clinically acceptable. Moreover, there were no significant differences for any of the GFRTTP wires during different immersion periods.

Within the limitations of this study, it was confirmed that GFRTTP wires containing T-glass fibers have excellent mechanical properties and color stability and are suitable for use in orthodontic treatment. Further investigation of the *in vivo* behavior of GFRTTP wires containing T-glass fibers, such as biomechanics and tissue responses, needs to be performed before they can be used for esthetic orthodontic wires in orthodontic treatment.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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