Original papers

Influence of thermal cycles and disinfection on the roughness, microhardness and color of PETG/TPU and PMMA

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Abstract

Background. Occlusal splints can protect teeth during bruxism, preventing tooth wear, as well as during sports activities, shielding them from impacts.

Objectives. To verify the influence of thermal cycles and disinfection on the roughness, microhardness and color of polyethylene terephthalate glycol/thermoplastic polyurethane (PETG/TPU) and poly(methyl methacrylate) (PMMA).

Materials and methods. Thirty-six PETG/TPU samples and 36 PMMA samples were prepared (ø10 mm × 3 mm). Six groups were created according to the material and the disinfection method used (n = 12 each): PETG/TPU (glister), PETG/TPU (hypochlorite), PETG/TPU (soap), PMMA (glister), PMMA (hypochlorite), and PMMA (soap). Roughness, Knoop microhardness and color evaluations were performed before the experiments (T1), after thermocycling (T2) and after disinfection (T3). Three-way repeated measures analysis of variance (ANOVA) and Tukey's test were used for statistical evaluations.

Results. For roughness and color, ANOVA showed statistical significance based on the interaction between thermal cycling, material and disinfectant factors. In terms of Knoop microhardness, ANOVA showed statistical significance based on the interaction between thermal cycling and material factors.

Conclusions. Roughness results were clinically acceptable in all groups at all time points, except the PETG/TPU and PMMA groups disinfected with hypochlorite. Microhardness significantly increased for both materials after thermal cycling, and at all time points, the microhardness of PMMA was significantly higher than that of PETG/TPU. After thermal cycling, the color changes were clinically unacceptable in all groups.

Key words: polymers, polyurethane, occlusal splints, polymethyl methacrylate, polyethylene terephthalate glycol

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Background

Most occlusal splints are based on acrylic resin (poly(methyl methacrylate) (PMMA) – a thermoplastic polymer).[1] Such splints can protect teeth during bruxism, preventing tooth wear, as well as during sports activities, shielding them from impacts.[1–4] Additionally, the splints can be used after orthodontic movement to prevent teeth from returning to their pre-treatment positions,[5] and for orthognathic treatment for the repositioning of skeletal structures.[6–8]

In recent years, a new material has been used to manufacture splints.[2] This material combines polyethylene terephthalate glycol (PETG – a thermoplastic polymer) with thermoplastic polyurethane (TPU). The PETG/TPU splint has a hard outer part, which comes into contact with opposing teeth during bruxism, and a soft inner part, which fits over the teeth.[2] The PETG is a rigid polymer with excellent conformability, optical quality, resistance to mechanical stress, and dimensional stability. The TPU is a ductile elastomer that helps absorb impacts due to its elasticity, providing patient comfort.[2] It is worth mentioning that the PETG/TPU occlusal splint (Erkodent) can only be manufactured in a thermoplasticizer from the same company, due to the precise temperature control of this device, which is necessary to manipulate the PETG/TPU without damaging it.

Roughness, microhardness and color are important clinical factors for polymers used in dentistry (e.g., PMMA or PETG/TPU occlusal splints). The roughness of a material is related to the accumulation of microbes on its surface.[9] Thus, the greater the roughness of an occlusal splint, the greater the accumulation of microbes on its surface.[9] The microhardness of a material is related to its wear resistance.[9] Therefore, the greater the microhardness of a material, the greater its longevity.[9] The color of a material is one of the most important factors in dentistry. A change in the color of an occlusal splint over time can cause discomfort to its user, especially if people are around while the splint is in use.

This study, evaluating the physical and mechanical properties of PETG/TPU, is justified for several reasons. First, there is only 1 recent study (2021) evaluating the effects of thermal cycles and different disinfection methods on the physical and mechanical properties of PETG/TPU, and comparing them with those of PETG.[2] Second, no published study has compared the physical and mechanical properties of PETG/TPU with those of PMMA. Third, the manufacturing cost of a PETG/TPU splint is higher compared with the manufacturing cost of a PMMA splint. Thus, it is important for dentists to learn more about this new material (PETG/TPU) before recommending it to their patients.

The objective of the current study is to examine the impact of thermal cycles and disinfection on the roughness, microhardness and color of PETG/TPU and PMMA.

Materials and methods

Groups

Thirty-six PETG/TPU samples and 36 PMMA samples were fabricated. Next, 6 groups were created according to the material and disinfection method used (n = 12 each): PETG/TPU (glister), PETG/TPU (hypochlorite), PETG/TPU (soap), PMMA (glister), PMMA (hypochlorite), and PMMA (soap). All manufactured samples (colorless and translucent) had the same dimensions (ø10 mm × 3 mm).[2,10]

Evaluation times

Assessments were performed at 3 time points (T1, T2 and T3):

– T1 (initial): roughness, microhardness and color change tests were performed in all groups;

– T2 (after thermal cycling): all groups underwent thermal cycling and, subsequently, all tests performed at T1 were repeated;

– T3 (after disinfection): each group was exposed to its respective disinfectant, and the tests performed at T1 were subsequently repeated (note: disinfection was performed on specimens that had already been subjected to thermal cycling).

All tests were performed by the same operator.

Sample manufacturing

In this study, samples of PMMA (Clássico, São Paulo, Brazil) and PETG/TPU (Erkoloc-Pro Clear; Erkodent, Pfalzgrafenweiler, Germany) were fabricated.

Thermopolymerizable PMMA samples with dimensions of ø10 mm × 3 mm[2,10] were prepared according to a study by Goiato et al.,[11,12] using the conventional resin polymerization method.[13] After manufacturing, the samples were submitted to finishing with Maxi-Cut burs to remove excess acrylic resin. Next, the samples were polished using a sequence of abrasive papers in a universal automatic polisher (APL-4; Arotec SA Indústria e Comércio Ltda., Cotia, Brazil).[11] The abrasive papers were used in ascending order (600-, 800- and 1200-grit) for 1 min each, at a speed of 300 rpm and under constant irrigation.[11] After finishing and polishing, the samples were stored in distilled water inside an incubator (CIENLAB, Campinas, Brazil) at a temperature of 37°C for 24 h to hydrate them while residual monomers were eliminated.[11] Each group was stored separately.

The thermoplastic PETG/TPU samples were manufactured from sheets of material measuring ø120 × 3 mm.[2] Before cutting the samples, the PETG/TPU sheets were thermoprocessed using the Erkoform 3D Motion (Erkodent), according to the manufacturer's recommendations.[2] After this process, metallic cylinders were used to cut the heated PETG/TPU sheets.[2] The PETG/TPU groups were stored in the same way as the PMMA groups.

All PETG/TPU and PMMA samples were prepared by the same operator.

Roughness

The roughness of all samples was determined using a profilometer (Dektak 150; Veeco, Plainview, USA). The samples were placed on a support and the profilometer tip was used on their surface.[10] The roughness average (Ra) values were measured using a cutoff of 500 µm in a 12-second time constant.[10] Three readings were taken on each sample and the average value was calculated.[10] The roughness was measured in micrometers [µm].[10] For the PETG/TPU samples, this evaluation was performed only on the PETG part.[2]

Knoop microhardness

The Knoop microhardness test was performed with a load of 25 g for 10 s (HMV/2T; Shimadzu, Kioto, Japan).[2] Three readings were taken on each sample.[2] The distance between penetrations, and between penetrations and the edges of the sample was 500 µm.[2] Values were recorded as Knoop hardness numbers (KHN). An average of the 3 readings was taken.[2] For the PETG/TPU samples, this evaluation was performed only on the PETG part.[2]

Color change

The color change (ΔE^*) was calculated using the Commission Internationale de l'Eclairage (CIE) $L^*a^*b^*$ system (UV/2450; Shimadzu).[2] The formula used was: $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{½}$. The L^* variable represents luminosity and varies from 0 (black) to 100 (white).[2] The a^* variable represents the amount of red (positive values) or green (negative values), while the b^* variable represents the amount of yellow (positive values) or blue (negative values).[2]

For the PETG/TPU samples, this evaluation was performed only on the PETG part, as this is the part that is visible when the patient is using an occlusal splint.[2]

Thermal cycles

The samples were submitted to 2000 cycles of immersion in alternating baths of distilled water at 5°C and 55°C for 60 s (MSCT/3; Convel, São Paulo, Brazil).[2]

Disinfection

Each group was disinfected with one of the following: Glister™ Concentrated Multi-Action Oral Rinse (Amway, São Paulo, Brazil), 1% hypochlorite[9] and liquid soap[2] (liquid soap with glycerin, physiologic pH, Johnson's Baby; Johnson & Johnson, São Paulo, Brazil).[2]

The liquid soap was not diluted with any substance. The Glister™ oral rinse was diluted with water according to the manufacturer's recommendations (5 jets were applied inside the product dosing cup and then the cup was filled up to the 1-ounce-line with water).

Disinfection was performed for 60 days, 3 times a week.[9] During disinfection, each group remained immersed in its respective disinfectant for 15 min at 37°C. After each disinfection, the samples were washed in running water for 30 s.[9]

Statistical analyses

Three-way repeated measures analysis of variance (ANOVA) and Tukey's test were used for statistical evaluations ($p < 0.05$; Jamovi, v. 2.2.5.0; Jamovi Project, Sydney, Australia).

Results

Roughness

For roughness, ANOVA showed statistical significance based on the interaction between thermal cycling, material and disinfectant factors ($p < 0.001$; Table 1). At T1 (Table 2), the PMMA (hypochlorite) group showed a significantly higher roughness value than the other groups. At T2, the PMMA (hypochlorite) and PMMA (soap) groups showed significantly higher roughness values than the other groups. At T3, the PETG/TPU (hypochlorite) group showed a significantly higher roughness value than the other groups, and the PETG/TPU (glister) and PMMA (hypochlorite) groups showed significantly higher roughness values than the PETG/TPU (soap), PMMA (glister) and PMMA (soap) groups.

When measurements taken at different time points were compared (Table 2), the PMMA (hypochlorite) group showed a significant increase in roughness at T2 (T2 compared with

Table 1. Three-way repeated measures analysis of variance (ANOVA) for the roughness test

Factors	Sum of squares	df	Mean square	F	p-value
Thermal cycling	0.1011	2	0.05053	116.64	<0.001*
Thermal cycling × material	0.0146	2	0.00731	16.87	<0.001*
Thermal cycling × disinfectant	0.0178	4	0.00444	10.24	<0.001*
Thermal cycling × material × disinfectant	0.0145	4	0.00363	8.37	<0.001*
Residue	0.0572	132	4.33e-4	–	–

* interaction between the factors was statistically significant or the analyzed factor was statistically significant; df – degrees of freedom.

Table 2. Mean (standard deviation) of the roughness values (μm) of the evaluated groups

Groups	Initial (T1)	After thermal cycling (T2)	After disinfection (T3)
PETG/TPU (glister)	0.173 (0.0172) Aa	0.195 (0.0291) Aa	0.248 (0.0219) Ab
PETG/TPU (hypochlorite)	0.186 (0.0309) Aa	0.194 (0.0274) Aa	0.288 (0.0350) Bb
PETG/TPU (soap)	0.185 (0.0216) Aa	0.193 (0.0199) Aa	0.200 (0.0179) Ca
PMMA (glister)	0.163 (0.0208) Aa	0.193 (0.0214) Aa	0.200 (0.0224) Ca
PMMA (hypochlorite)	0.207 (0.0289) Ba	0.233 (0.0384) Bb	0.251 (0.0355) Ab
PMMA (soap)	0.173 (0.0381) Ab	0.210 (0.0260) Bb	0.217 (0.0262) Cb

Different lowercase letters horizontally represent a statistically significant difference. Different capital letters vertically represent a statistically significant difference (Tukey's test, $p < 0.05$). PETG – polyethylene terephthalate glycol; TPU – thermoplastic polyurethane; PMMA – poly(methyl methacrylate).

T1). The PETG/TPU (glister) and PETG/TPU (hypochlorite) groups showed a significant increase in roughness at T3 (T3 compared with T2).

Knoop microhardness

For microhardness, ANOVA showed statistical significance based on the interaction between thermal cycling and material factors ($p < 0.001$; Table 3). There was a significant increase in microhardness after thermal cycling (T2 compared with T1) for PETG/TPU and PMMA (Table 4). At all time points (T1, T2 and T3), the microhardness of PMMA was significantly higher than that of PETG/TPU (Table 4).

Color change

For color change, ANOVA showed statistical significance based on the interaction between thermal cycling, material and disinfectant factors ($p < 0.001$; Table 5). Based on $\Delta E1$, there were no significant differences between groups (Table 6). Based on $\Delta E2$, the PETG/TPU (hypochlorite) group showed a significantly greater color change compared with the other groups (Table 6).

From $\Delta E1$ to $\Delta E2$, there was a significant reduction in color change values in all groups, except for PMMA (glister) ($p > 0.05$) and PETG/TPU (hypochlorite – this group showed a significant increase in color value from $\Delta E1$ to $\Delta E2$; Table 6).

Table 3. Three-way repeated measures analysis of variance (ANOVA) for the microhardness test

Factors	Sum of squares	df	Mean square	F	p-value
Thermal cycling	239.08	2	119.538	102.573	<0.001*
Thermal cycling × material	53.21	2	26.607	22.831	<0.001*
Thermal cycling × disinfectant	1.80	4	0.451	0.387	0.818
Thermal cycling × material × disinfectant	2.87	4	0.718	0.661	0.652
Residue	153.83	132	1.165	–	–

* interaction between the factors was statistically significant or the analyzed factor was statistically significant; df – degrees of freedom.

Table 4. Mean (standard deviation) of the Knoop hardness numbers (KHN) of the evaluated materials

Material	Initial (T1)	After thermal cycling (T2)	After disinfection (T3)
PETG/TPU	11.5 (1.17) Aa	12.5 (0.91) Ab	12.8 (0.90) Ab
PMMA	18.7 (1.47) Ba	21.7 (1.43) Bb	22.2 (1.42) Bb

Different lowercase letters horizontally represent a statistically significant difference. Different capital letters vertically represent a statistically significant difference (Tukey's test, $p < 0.05$). PETG – polyethylene terephthalate glycol; TPU – thermoplastic polyurethane; PMMA – poly(methyl methacrylate).

Table 5. Three-way repeated measures analysis of variance (ANOVA) for the color change test

Factors	Sum of squares	df	Mean square	F	p-value
Thermal cycling	89.4	1	89.39	24.70	<0.001*
Thermal cycling × material	14.2	1	14.17	3.92	0.052
Thermal cycling × disinfectant	93.8	2	46.92	12.97	<0.001*
Thermal cycling × material × disinfectant	201.6	2	100.78	27.85	<0.001*
Residue	238.8	66	3.62	–	–

* interaction between the factors was statistically significant or the analyzed factor was statistically significant; df – degrees of freedom.

Table 6. Mean (standard deviation) of the color values (ΔE) of the evaluated groups

Groups	ΔE1	ΔE2
PETG/TPU (glister)	6.35 (1.60) Aa	3.67 (2.62) Ab
PETG/TPU (hypochlorite)	4.23 (1.94) Aa	8.46 (1.75) Bb
PETG/TPU (soap)	6.90 (2.58) Aa	2.51 (2.20) Ab
PMMA (glister)	4.22 (1.69) Aa	4.23 (2.41) Aa
PMMA (hypochlorite)	5.65 (2.00) Aa	1.96 (0.84) Ab
PMMA (soap)	4.72 (2.15) Aa	1.79 (1.30) Ab

Different lowercase letters horizontally represent a statistically significant difference. Different capital letters vertically represent a statistically significant difference (Tukey's test, $p < 0.05$). PETG – polyethylene terephthalate glycol; TPU – thermoplastic polyurethane; PMMA – poly(methyl methacrylate).

Discussion

The purpose of thermal cycling was to simulate the extreme temperature changes that occur during the clinical use of an occlusal splint. In this study, 2000 thermal cycles were performed, which represents 2 years of clinical use of an occlusal splint.[2] This procedure can promote successive expansions and contractions of the material causing microcracks.[2] The literature shows that temperature variations combined with high humidity, as well as the use of disinfection methods over time, are extrinsic degradation factors for a polymer.[2,9,11,14] It is noteworthy that the intrinsic degradation factor for a polymer is represented by a change in its matrix over time.[2,9,11,14]

When comparing the groups at T1 (Table 2), there was no significant difference between the PETG/TPU groups, while the PMMA groups showed significant differences between them. Furthermore, the roughness of the PMMA (hypochlorite) group samples was significantly higher than those of the PETG/TPU groups (T1; Table 2). These results may suggest that the industrial polishing of PETG/TPU was more standardized than the manual polishing of PMMA. Based on the statistical analysis, comparing the groups at T2, it is possible to verify that in most cases, the roughness values of PMMA were significantly higher than those of PETG/TPU (Table 2). Thus, this may suggest that, clinically, PETG/TPU accumulates less bacterial plaque on its surface over time.

Table 2 shows that from T1 to T2, only 1 group (PMMA (hypochlorite)) showed a significant increase in the roughness value. This result can be explained by a degradation of this type of material. The degradation of a polymer, due to extrinsic and intrinsic factors, is represented, for example, by a significant change in its roughness, microhardness and color values.[2,9,11,14] Despite this, as previously reported, the increase in roughness occurred in 1 group only (Table 2), which shows the excellent quality of both types of materials.

At T3 (Table 2), the following information on roughness values can be observed: 1) comparing the PETG/TPU groups: soap < glister < hypochlorite ($p < 0.05$); and 2) comparing the PMMA groups: soap = glister < hypochlorite ($p < 0.05$). Thus, hypochlorite was the disinfectant that most increased the roughness of these materials. Furthermore, still based on the T3 time point (Table 2), the glister and the hypochlorite generated a significantly greater roughness on the PETG/TPU surface than on the PMMA surface. Thus, PMMA is likely to be less negatively influenced by these disinfectants than PETG/TPU. It is noteworthy that, based on another analysis of Table 2 (i.e., from T2 to T3), PMMA roughness was not influenced by any disinfectant, unlike PETG/TPU roughness, which increased significantly after disinfection with hypochlorite and glister.

The level of roughness that prevents the accumulation of microbes on the surface of a material is ≤0.2 μm (clinically acceptable value).[15,16,17] A greater roughness than this promotes bacterial adhesion to the surface of the material, which may result in gingival inflammation and caries.[16,17] In this study, values <0.25 μm were considered clinically acceptable. Thus, despite the statistically significant differences in Table 2, all results were clinically acceptable, with the exception of 2 groups (T3 – PMMA (hypochlorite) and T3 – PETG/TPU (hypochlorite)). Therefore, hypochlorite was the most aggressive disinfectant for the materials used.

For PETG/TPU or PMMA, there was a significant increase in microhardness at T2 when compared with T1 (Table 4). This shows that, at T2, both materials showed degradation based on microhardness. It should be noted that, at all time points, the microhardness values of PMMA were significantly higher than those of PETG/TPU. Therefore, clinically, PMMA is more resistant to wear than PETG/TPU, contributing to the greater longevity of PMMA splints (Table 4). Based on this result, it is possible to recommend PMMA splints for severe cases of bruxism and PETG/TPU splints for milder cases of bruxism.

Specification number 12 of the American Dental Association (ADA) indicates that the microhardness of acrylic resins for denture bases should not be lower than 15 KHN.[18] Based on this recommendation, all PMMA microhardness values (T1, T2 and T3) were clinically acceptable (Table 4).[18]

Based on ΔE1 (Table 6), there were no significant differences between PETG/TPU or PMMA groups; and between PETG/TPU and PMMA groups. This demonstrates that thermal cycling generated similar effects in all evaluated groups (ΔE1), and that occlusal splints made of PMMA or PETG/TPU may show similar color changes over time. Based on ΔE2 (Table 6), the PETG/TPU (hypochlorite) group showed a significantly higher color change value than the other groups, and from ΔE1 to ΔE2, the PETG/TPU (hypochlorite) group was the only group that showed a significant increase in color change. Thus, the chronic use of this disinfectant can clinically change the color of a PETG/TPU splint.

The $\Delta E < 3.7$ is considered clinically acceptable for a material.[19,20] Thus, based on ΔE1 (Table 6), all observed values were clinically unacceptable, demonstrating that a large color change can be expected for both materials over time. Based on ΔE2 (Table 6), in most situations, disinfectants acceptably changed the color of the materials used (PETG/TPU (glister), PETG/TPU (soap), PMMA (hypochlorite), and PMMA (soap)). However, this only shows that, in most situations, disinfectants acceptably changed the color of materials that have already undergone a clinically unacceptable color change.

A limitation of this study is that only 3 disinfectants were used. Thus, more studies are needed using other disinfectants.

Conclusions

Roughness results were clinically acceptable in all groups at all time points, except for the PETG/TPU and PMMA groups disinfected with hypochlorite. The microhardness was increased for both materials after thermal cycling, and, at all time points, the microhardness of PMMA was significantly higher than that of PETG/TPU. The color change was clinically unacceptable after thermal cycling in all groups.

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