

TALITA SUELEN DE QUEIROZ

**Impact Resistance and Microstructural Response of EVA and 3D
Printed KeyGuard resin for Sports Mouthguards under Hydrothermal
Ageing**

PHD STUDENT: Talita Suelen de Queiroz

INTERNATIONAL ADVISOR: Profa. Dr^a. Flávia Pires Rodrigues

BRAZILIAN ADVISOR: Prof. Dr. Tarcísio José de Arruda Paes Junior

INTERNATIONAL CENTER: University of Leeds – UK.

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ABSTRACT

This study aims to compare the impact resistance and microstructural response of EVA and KeyGuard 3D-printed resin, before and after hydrothermal ageing. Additionally, it investigates the feasibility of KeyGuard as an alternative material for fabricating personalised sports mouthguards. The project will be divided into two complementary studies. **Study A:** Rectangular, V-notched specimens ($10.2 \times 63.5 \times 4.0$ mm) of Bio-Art EVA, Keystone EVA and KeyGuard resin ($n = 10$) will be tested in a pendulum impact apparatus adapted from the Dynstat method. Specimens subjected to ageing will undergo 5,000 thermal cycles ($5\text{ }^{\circ}\text{C}/55\text{ }^{\circ}\text{C}$, 30 s dwell). Absorbed energy and failure modes will be recorded, and representative specimens will be examined by scanning electron microscopy (SEM) to analyse the failure mode. **Study B:** Square specimens ($15 \times 15 \times 3$ mm; $n = 10$) of each material, with and without thermal cycling, will be analysed by micro-computed tomography for porosity and layer integrity, optical profilometry for surface roughness (S_a , S_z) and Vickers hardness testing. The data will be subjected to descriptive and inferential statistics after checking homogeneity and homoscedasticity to determine the appropriate test.

Keywords: Mouth Protectors; 3D printing; Mechanical Tests and Materials Testing.

OBJECTIVE

STUDY A

This study aims to **evaluate and compare the impact resistance and failure patterns** of various materials employed in the fabrication of sports mouthguards by means of a **pendulum impact test** adapted from the Dynstat method with notched specimens.

H0: No statistically significant differences will be presented in absorbed impact energy or failure patterns among the three materials tested.

STUDY B

This study aims to **assess the internal microstructure, surface topography, and hardness of specimens fabricated from ethylene–vinyl acetate (EVA) and a 3D printed resin for sports mouthguards (KeyGuard), both before and after hydrothermal aging.**

H0: Thermal cycling will not present statistically significant differences in microstructure, surface topography or hardness among EVA and 3D printed resin for sports mouthguard.

3. Schedule of Activities

Activities	Nov 25	Dec 25	Jan 26	Feb 26	Mar 26	Apr 26
Literature review and alignment with supervisors	X					
Protocol definition and equipment training	X					
Specimens Fabrication		X	X			
Pilot impact tests (Study A)		X	X			
Impact tests with non-cycled specimens (Study A)			X	X		
Thermal cycling (Studies A & B)			X			
Impact tests with cycled specimens (Study A)				X		
SEM analysis before and after impact (Study A)				X		
Micro-CT, profilometry, and hardness testing (Study B)					X	
Data processing and statistical analysis					X	X
Report writing and supervisor review					X	X
Scientific article drafting						X

4. MATERIALS AND METHODS

4.1 STUDY A

Impact Behaviour of Ethylene–Vinyl Acetate and a Flexible 3-D-Printed Resin Used in Sports Mouthguards: Effects of Hydrothermal Ageing

4.1.1 Materials Selection

The materials chosen for the present study will be subdivided into ethylene–vinyl acetate (EVA) and a flexible 3-D-printed resin indicated for sports mouthguard fabrication, as detailed in Table 1.

Table 1. Experimental groups and corresponding materials evaluated.

Group	Material
EVA.B	Ethylene vinyl acetate copolymer (EVA) without hydrothermal ageing - Bio-art Equipamentos Odontológicos Ltda., Brazil (n=10)
EVA.K	Ethylene vinyl acetate copolymer (EVA) without hydrothermal ageing –Proform, Keystone Industries, USA (n=10)
EVA.KR	Ethylene vinyl acetate copolymer reinforced (EVA) without hydrothermal ageing –Proform, Keystone Industries, USA (n=10)
3D.K	Keyguard Resin – Keystone Industries without hydrothermal ageing, USA (n=10)
aEVA.B	Ethylene vinyl acetate copolymer (EVA) with hydrothermal ageing - Bio-art Equipamentos Odontológicos Ltda., Brazil (n=10)
aEVA.K	Ethylene vinyl acetate copolymer (EVA) with hydrothermal ageing –Proform, Keystone Industries, USA (n=10)
a.EVA.KR	Ethylene vinyl acetate copolymer reinforced (EVA) with hydrothermal ageing –Proform, Keystone Industries, USA (n=10)
a3D.K	Keyguard Resin – Keystone Industries with hydrothermal ageing, USA (n=10)

4.1.2 Specimen Design

Specimens will be fabricated from the materials listed in Section 3.1.1. **Rectangular bars 10.2 mm x 63.5 mm x 4.0 mm (width × length × thickness) for each group (n=10)** will be prepared and receive a centrally positioned notch at the geometric centre of the sample. The notch will be V-shaped, cut at a 45° angle to a depth of 0.3 mm (ISO D256 adapted), using a V-Notch Cutting Cutter in accordance with ISO 179/180 to ensure symmetry and reproducibility across groups. For the printed-resin specimens, the notches will be made after post-curing, in order to avoid that the procedure will not induce micro-cracks or chipping in the

specimens.

4.1.3 Hydrothermal ageing

Specimens assigned to the hydrothermal ageing groups will be subjected to a thermal cycling protocol in an automated thermocycler (THE 1400 Thermocycler - SD mechatronic, Germany). Alternating immersions in water baths at 5 °C and 55 °C, with a duration of 30 s at each temperature and a 5 s transfer between baths; a total of 5,000 cycles will be completed.

4.1.4 Pilot Tests

Prior to the main test, pilot trials will be carried out to calibrate the optimal impact energy (J) until a measurable degree of significant deformation or partial fracture be observed, to establish the failure criteria for the definitive assays. The impact energy determined in the pilot phase will be fixed and applied uniformly to all experimental groups.

4.1.5 Experimental test

After conditioning the specimens in a room maintained at $23\text{ °C} \pm 3\text{ °C}$ for 48 h, each one will be positioned between the two supports of the microimpact tester (Zwick 5102 Pendulum Impact Tester, UK), and the pendulum will be released from the standardised height. For each impact, the absorbed energy (J) will be recorded, and the failure depth (mm) will be measured from the notch with a digital calliper.

4.1.4.1 Morphological Analysis by Scanning Electron Microscopy (SEM)

Fractography will be made through SEM analysis, to analyse the origin and propagation of the failure. Three specimens from each group ($n = 3$) will be examined through an environmental scanning electron microscopy (SEM) (Hitachi SU5000 SEM, Japan), working in high or low voltages, both before and after the impact tests. The images will assess the topographic presence of cracks, direction of the crack propagation, and delamination, at magnifications of up to 5000 x.

4.1.5 Statistical Analysis

The data after each impact will include absorbed impact energy (in joules) with and

without hydrothermal ageing. These data will be subjected to statistical analysis. Comparisons between the groups will be carried out using ANOVA or Kruskal-Wallis' test, depending on data normality.

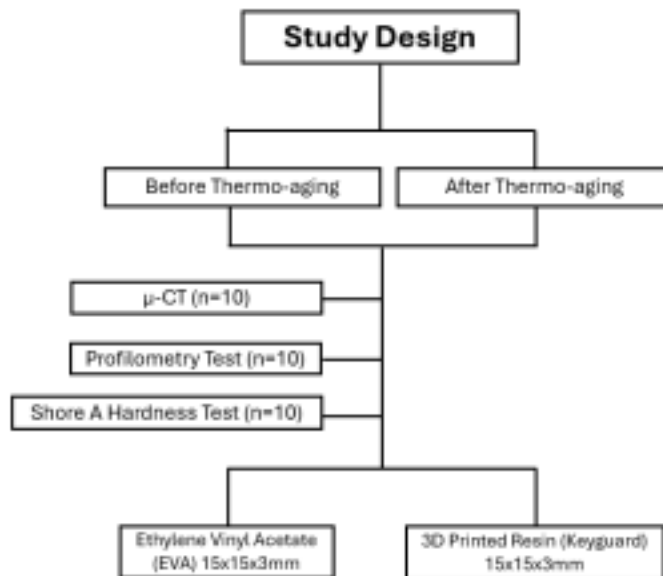
4.2 STUDY B

Hydrothermal ageing effects on Microstructure, Surface Topography, and Hardness of EVA and 3-D-Printed KeyGuard Resin for Sports Mouthguards

4.2.1 Study Design

Pre-fabricated EVA plates (3 mm thick) will be used and KeyGuard 3-D-printing resin will be employed in its liquid formulation together with the manufacturer-recommended post processing materials and equipment, including a washing solution and a UV-light final-curing unit. **The proposed tests for microstructural characterisation will comprise micro-computed tomography, profilometry, and hardness measurements performed before and after hydrothermal ageing**, as presented in Flowchart 1.

Flowchart 1 - Study Design.



4.2.2 Specimens Preparation

Square specimens (15 mm × 15 mm × 3 mm) will be fabricated. They will be randomly allocated to six experimental groups (n = 10 per group): Group 1, EVA-B Control (no hydrothermal ageing); Group 2, EVA-B Cycled (after hydrothermal ageing); Group 3, EVA K Control (no hydrothermal ageing); Group 4, EVA-K Cycled (after hydrothermal ageing); Group 5, KeyGuard Control (no hydrothermal ageing); and Group 6, KeyGuard Cycled (after

hydrothermal ageing). The same group distribution will be used for each analysis: micro-CT, profilometry and Hardness Testing.

EVA specimens: Pre-fabricated EVA sheets 3 mm thick will be cut into 15 mm × 15 mm squares with a scalpel. A thermal-polishing protocol will then be applied with a butane torch: the flame will be passed rapidly and uniformly over each surface to melt the surface layer and re solidify it into a smoother and more homogeneous topography.

KeyGuard specimens: A 15 mm × 15 mm × 3 mm STL model will be designed in CAD (Rhinoceros 7; McNeel North America, Seattle, WA, USA). For printing, the file and resin preset “KeyGuard – Keystone Industries” will be selected in the Asiga printer software, with all parameters set according to the manufacturer’s instructions. The resin will be shaken for 5 min, and will rest for 5 min to dissipate bubbles. After the printing procedure, the specimens will be removed from the build platform and pre-cleaned with isopropyl alcohol and a soft-bristle brush. They will then undergo two consecutive 1min ultrasonic washes in a pure isopropyl alcohol, with compressed-air drying between the washes. After 20 min air-drying, post-curing will be performed in an Otofash G171 chamber (2 000 flashes per side). Surfaces will be finished and polished with descending-grit Scotch-Brite wheels followed by felt wheels and polishing pastes to obtain smooth, uniform surfaces that minimise artefacts in subsequent measurements.

4.2.2 Hydrothermal ageing

Specimens assigned to the hydrothermal ageing groups will be subjected to a thermal cycling protocol in an automated thermocycler (THE 1400 Thermocycler - SD mechatronic, Germany). Alternating immersions in water baths at 5 °C and 55 °C, with a duration of 30 s at each temperature and a 5 s transfer between baths; a total of 5,000 cycles will be completed.

4.2.4 Materials Characterization

4.2.4.1 Micro-computed Tomography (μ-CT)

The internal microstructure of specimens from all groups **will be evaluated by micro computed tomography (μ-CT)**. The specimens will be scanned in a micro-computed tomograph (model 1132, Bruker, Belgium) with a beam accelerating voltage of 100 kV and X-ray beam current of 96 μA, 207 TAG Image File Format (TIFF) 16-bit images, with vertical and horizontal resolutions of 1336-dpi and 2-Mb size file. Microtomography images of these sections will be obtained using an isotropic voxel size of 11.24 μm or lower. The three-dimensional reconstructions generated by μ-CT will be exploited for both qualitative and quantitative assessments. Qualitatively, the datasets will visualise the layer-by-layer

architecture, filler distribution, and the presence of pores or voids. Quantitatively, they will allow calculation of global and local pore-volume fractions and size distributions, as well as evaluation of filler

dispersion homogeneity where discernible. Segmentation and morphometric analyses will be performed in NRecon and CTAnalysis (Bruker, Belgium).

4.2.4.2 Profilometry

The surface topography of specimens from all groups will be assessed using optical profilometry (Proscam 2200, Scantron, UK). Three measurements will be taken on representative regions of each specimen surface. Three-dimensional surface-roughness parameters, such as Sa (arithmetical mean height) and Sz (maximum peak-to-valley height) will be calculated to analyse the surface characteristics.

4.2.4.3. Hardness Test

The test will be performed with a microhardness (Duramin 40, Struers, UK) following ISO 48-4 and ASTM D2240 at circular specimens (30mm x 30mm x 4mm), before and after thermal cycling (5 000 cycles alternating between 5 °C and 55 °C for 30 s).

One hour before the test, specimens will be conditioned in a room maintained at 23 °C ± 3 °C. A standard Vickers indenter will then be applied with a constant contact force of approximately 200 kgf for 10 s. Indentation depth is not measured directly; instead, the hardness value is inferred from the material's resistance to penetration under this standardised load.

4.5 Statistical Analysis

The quantitative data obtained for porosity (volume/distribution), surface-roughness parameters (Sa, Sz) and hardness will be subjected to statistical analysis. Comparisons between Control and Hydrothermal ageing groups for each material will be performed for each parameter using the appropriate statistical test, after verifying data normality and homoscedasticity. A 5 % significance level ($p < 0.05$) will be adopted for all statistical analyses.

7 REFERENCES

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MATERIALS REQUEST

4mm EVA plates PROFORM: 36 plates

4mm EVA reinforced plates PROFORM: 36 plates

3mm EVA plates PROFORM: 20 plates

3mm EVA plates PROFORM: 20 plates