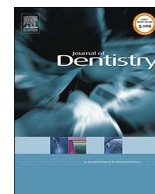




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## Polymerisation, antibacterial and bioactivity properties of experimental orthodontic adhesives containing triclosan-loaded halloysite nanotubes

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### ABSTRACT

**Objective:** To evaluate the immediate enamel bond strength, *in situ* degree of conversion and the polymerisation rate of three experimental orthodontic adhesives containing triclosan-loaded halloysite nanotubes. The antibacterial and bioactivity properties of such experimental materials were also assessed.

**Materials and methods:** Three experimental orthodontic adhesives were formulated by incorporating triclosan-loaded halloysite nanotubes (TCN-HNT) at different concentrations (5 wt%, 10 wt% and 20 wt%) into a resin blend (Control). The maximum polymerisation rate of the tested adhesives was evaluated through FTIR, while Raman was used to analyse the *in situ* degree of conversion (DC) at the bracket/enamel interface. The shear bond strength (SBS) of the enamel-bonded specimens was assessed at 24 h. The antibacterial properties of the experimental materials against *S. Mutans* were evaluated up to 72 h, while, their bioactivity was evaluated after 14 days of artificial saliva (AS) storage through SEM-EDS and Raman spectromicroscopy.

**Results:** Incorporation of TCN-HNT increased the polymerisation properties without interfering with the immediate bonding properties of the experimental adhesives. All experimental adhesives containing TCN-HNT inhibited bacterial growth at 24 h, and induced mineral deposition after 14 days of AS storage. At 72 h, only the experimental system containing 20% TCN-HNT maintained such a capability.

**Conclusions:** Adhesives doped with TCN-HNT present improved polymerisation properties and suitable bonding performance. However, only the adhesives containing TCN-HNT > 10% might promote long-term antibacterial activity and reliable mineral deposition.

**Clinical significance:** The use of adhesives containing triclosan-loaded halloysite represents a promising “smart” approach to bond orthodontic brackets and bands; these might prevent enamel demineralisation and induce enamel remineralisation during the treatment.

### 1. Introduction

Enamel demineralisation is one of the main causes responsible for the formation of white spot lesions (WSL) during fixed orthodontic treatments, in particular in those patients with limited oral hygiene

compliance [1]. Wide gaps are often observed at the adhesive–enamel interface around brackets [2]; this represents the most common site for demineralisation to occur due to accumulation of a biofilm rich in cariogenic species such as *Streptococcus mutans* [3,4].

It has been advocated that the use of ion-releasing materials such as

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glass ionomer cements may reduce the risk of enamel demineralisation and prevent excessive bacterial growth [5]. Based on this information, several therapeutic agents such as fluoride [6], chlorhexidine [7] and nano-silver [8] have been incorporated into experimental orthodontic bonding materials to overcome such a clinical issue. However, it seems that although their early effectiveness, the release of such active principles tends to reduce overtime. Moreover, some of these agents may induce tooth discoloration; for instance, the release of silver ions or chlorhexidine may often cause anaesthetics appearance when incorporated into orthodontic cements [9].

It has been reported that the use of triclosan nanoparticles may represent an alternative to conventional antibacterial agents due to its small particle size, as well as to the little amount needed to produce antibacterial effects [10]. Several researchers have been inspired to create innovative composites containing such inorganic nanoparticles due to their large surface area and to their high surface reactivity [11,12].

Nano-compounds based on mesoporous aluminosilicate clay (i.e. halloysite nanotubes – HNTs) have been incorporated into dental adhesives as nano-carriers for antibacterial agents [13]. Moreover, these nanotubes have an inner diameter of 30–70 nm, which could be infiltrated by resin monomers and increase the mechanical properties of resin-based materials due to their great elastic modulus (140 GPa) [14].

We hypothesised that triclosan-loaded HNTs might be used as a promising nano-filler for innovative therapeutic orthodontic adhesives to induce mineral deposition on the enamel around brackets.

Thus, the first objective of this study was to evaluate the enamel bond strength, and the polymerisation properties (i.e. *in situ* degree of conversion and polymerisation rate) of experimental orthodontic adhesives containing different concentrations of triclosan-loaded halloysite nanotubes. The antibacterial and bioactivity properties of such experimental materials doped with triclosan-loaded HNTs were also assessed.

The first hypothesis tested in this study was that the incorporation of triclosan-loaded HNTs within the composition of experimental orthodontics adhesives would not interfere with their immediate bonding performance to acid-etched enamel and polymerisation properties compared to the control experimental adhesive containing no TCN-HNT.

The second hypothesis was that the incorporation of different amount of triclosan-loaded HNTs would enhance the antibacterial, and bioactivity properties of such materials compared to the control experimental adhesive containing no TCN-HNT.

## 2. Materials and methods

### 2.1. Preparation of TCN-loaded HNTs (TCN-HNT)

Halloysite nanotube (HNT) –  $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4 \cdot 2\text{H}_2\text{O}$  with a diameter of 30–70 nm and length of 1–3  $\mu\text{m}$  (Sigma-Aldrich, St. Louis, MO, USA) were submitted to a silanisation process using a solution containing 5 wt.% of 3-metacryloxypropyltrimetoxysilane and 95 wt.% acetone at 110 °C for 24 h. Subsequently, the treated nanoparticle were mixed [1:1 ratio] with 2,4,4-Trichloro-2-hydroxydiphenyl ether (TCN – Triclosan, Fagron, Rotterdam, SH, Netherlands) under continues agitation for 1 h as described in a previous study [15]. The mixture was then dispersed in 95 wt.% pure ethanol (0.03 mg ml<sup>-1</sup>) and sonicated for 1 h. Subsequently, the TCN-HNT nanotubes were desiccated for 10 days at 30 °C to ensure complete evaporation of residual solvents [15]. TCN-HNT was finally characterised using a Transmission Electron Microscope (TEM) JEM 120 Exll (JEOL, Tokyo, Japan) at 80 kV at a magnification X 300,000.

### 2.2. Formulation of experimental adhesive – incorporation of TCN-HNT

A monomer resin blend was created by mixing 75 wt.% bisphenol-

A-glycol dimethacrylate (BisGMA – Sigma-Aldrich Co., St. Louis, MO, USA) and 25 wt.% triethylene glycol dimethacrylate (TEGDMA- Sigma-Aldrich) and used as a control adhesive containing no TCN-HNT. Moreover, a photoinitiator (CQ: Camphorquinone, 0.5 mol%) and two co-initiators (EDAB: ethyl 4-dimethylaminobenzoate; DPIHFP: diphenyliodonium hexafluorophosphate) (Aldrich Chemical Co., Milwaukee, WI, USA) were also added at a concentration of 1 mol%.

The TCN-HNT filler was added at concentrations of 5, 10 and 20 (wt.%) into the resin blend in order to create three experimental adhesives. All formulations were mixed and maintained in an ultrasonic bath for 1 h. The four experimental resin adhesives were finally stored in a dark chamber.

### 2.3. Polymerisation rate evaluation (FTIR-ART)

Polymerisation rate of the experimental adhesives was evaluated using Fourier transform infrared spectroscopy (FTIR) using a spectrometer equipped with an attenuated total reflection (ATR) (Vertex 70; Bruker Optics, Ettlingen, Germany). Three specimens for each group were analysed by directly applying the tested materials (3  $\mu\text{L}$ ) onto the diamond crystal. These were light activated for 40 s using a light emitting diode unit (Radii Cal, SDI, Bayswater, VIC, Australia) with an irradiation value of 1200 mW/cm<sup>2</sup> at a standardised distance of 2 mm. Analysis was performed at a controlled room temperature of 23 ± 1 °C and 60 ± 1% relative humidity.

The IR-Solution software was used to standardise the assessment parameters such as scanning range (4000–800 cm<sup>-1</sup>), resolution (4 cm<sup>-1</sup>), and scanning time (40 s). This setup allowed the acquisition of each single scan, every 0.7 s during photo-activation procedure. The maximum polymerization rate [Rp. (s<sup>-1</sup>)] was also evaluated as described in a previous study [16] based on the intensity of the C=C stretching vibrations at 1635 cm<sup>-1</sup> (peak height) as a function of time, and using the symmetric ring stretching at 1608 cm<sup>-1</sup> from the polymerised and non-polymerised samples as an internal standard.

The data was plotted and a sigmoidal curve fitting method was applied with the linear regression method using Sigma Plot 12.0 for Windows (Systat Software Inc, San Jose, CA, USA).

### 2.4. Bonding procedures and bond strength evaluation

The buccal surface of 60 anterior bovine teeth were polished using a 600 grid SiC paper for 5 s in order to remove the aprismatic enamel only; this was checked by using a stereo microscope (×80). All specimens were then individually placed in cylinder-shape moulds and embedded in a self-curing polymethyl-methacrylate resin (Esschem Linwood, Pennsylvania, PA, USA), with buccal surface perpendicular to the horizontal plane; fifteen enamel specimens were used for each adhesive group (Table 1). The enamel surface of each specimen was first etched for 30 s with a 37% phosphoric acid gel (Acid Gel, Vilevie, Joinville, SC, Brazil) and rinsed with water for the same time. After air-drying for 5 s, orthodontic brackets were positioned on the enamel surface and kept under constant pressure (276 g); resin excess around the bracket was removed with a sharp dental probe.

Light-curing was performed for 40 s [17] using a light emitting diode unit (Radii Cal, SDI).

Specimens bonded to their brackets (Morelli Ltd, Sorocaba, SP, Brazil) with 11.18 mm<sup>2</sup> area, were stored in distilled water at 37 °C for 24 h. Subsequently, these were positioned in a Universal Testing Machine (Shimadzu EZ-SX, São Paulo, SP, Brazil) positioning the incisal edge of the bracket base parallel to a sharp chisel blade. They were stressed to failure using a 500 N load cell with a crosshead speed of 1.0 mm/min. The maximum force required for bracket's debonding was recorded in N, which was used to calculate the bond strength in MPa (N/mm<sup>2</sup>). Following debonding, the amount of adhesive on each enamel surface was with analysed using a stereomicroscope (×10) and the adhesive remnant index (ARI) calculated as described in a previous

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