



## Physical, chemical and antimicrobial evaluation of a composite material containing quaternary ammonium salt for braces cementation



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### ABSTRACT

The antibiofilm effect of iodide quaternary ammonium methacryloxy silicate (IQAMS) in Transbond XT Light Cure Adhesive resin used for braces cementation was evaluated. Fourier Transform Infrared (FTIR) spectroscopy confirmed IQAMS formation and Scanning Electron Microscopy coupled to Energy-Dispersive X-ray Spectroscopy (SEM-EDS) revealed that as coating, the quaternary ammonium groups from IQAMS were homogeneously dispersed throughout the surface. When incorporated, the composite material presented homogeneous dispersion throughout the resin. Assays with *Streptococcus mutans* demonstrated enhanced antibiofilm effect for the IQAMS coated resin, with much lower colony-forming units (CFU), in comparison to incorporated IQAMS. Such a difference was assigned to low availability of quaternary ammonium groups at the surface of resin when IQAMS was incorporated, hindering its antibiofilm effect. Additionally, the incorporation of IQAMS led to slight decrease in ultimate bond strength (UBS) and shear bond strength (SBS), in comparison to the neat commercial resin. Thus, the synthesized IQAMS displays great potential as antibiofilm coating or sealant to prevent oral infections in brackets during orthodontic treatment.

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### 1. Introduction

The goal of orthodontic treatment is to provide complete and integrated therapy for patients' oral health; restoring function as well as aesthetics [1–3]. Many orthodontic treatments available involve cementation of brackets and other metallic devices combined with elastic ligatures and arc wires, hindering hygiene. This results, for instance, in large *Streptococcus mutans* [4,5] and *Candida albicans* [6] biofilm accumulation and colonization, which it can lead to dental caries, compromising the oral health of patients [1,7–9]. The prevention of dental caries requires commitment and effort of the patient to properly cleanse the orthodontic devices, in addition to make use of a less cariogenic diet [10]. In order to support the patients and become the orthodontic treatment more effective, researchers have developed new materials with antimicrobial (bactericidal/bacteriostatic) properties [11,12].

Organically modified silicates (ORMOSIL) are hybrid materials [13] that contain organic functionalities covalently bonded to the silicate network providing especial properties [14,15]. Sol-gel process is a suitable approach to obtain ORMOSIL-based materials under mild

conditions with multifunctional properties [13,14,16–18]. In a typical sol-gel process, silicon alkoxides precursors undergo acid or basic-catalyzed hydrolysis forming silanol groups (Si–OH) that further react to form siloxane bonds (Si–O–Si) and hence lead to the development of a tridimensional network [13,17,18]. Given its low toxicity and biocompatibility, ORMOSIL have been used as biomaterial via immobilization of biomolecules or modification of the silicate network with functional groups with biological activity [16,18,19]. Quaternary ammonium salts are among the groups most used to aggregate antimicrobial properties to the silicate network, since they are already widely used as disinfectant in food production and medical appliances to prevent bacterial contaminations [20]. In this regard, dimethyloctadecyl[3-(trimethoxysilyl)propyl] ammonium chloride, an ORMOSIL precursor, has emerged as an effective compound for development of antibacterial dental composites [19,21–24].

The antimicrobial activity of quaternary ammonium groups is attributed to their ionic part combined to a lipophilic chain [25]. Palermo et al. found that the alkyl chain can penetrate into the cell causing lysis and death [26]. Li et al. [27] showed a large decrease in colony-forming unity (CFU) on samples containing quaternary ammonium groups with 12 carbons length. Additionally, Gong et al. [28] observed that the modification of the ORMOSIL network with methacrylate groups led to development of quaternary

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ammonium methacryloxy silicates (QAMS), that incorporated and dispersed better in orthodontic resin after establishment of covalent bonds with the acrylate groups present in the resin via copolymerization [19,24,28].

Based on the previous statements, this study focus in the synthesis of iodide quaternary ammonium methacryloxy silicate (IQAMS) and evaluation of its antibiofilm properties in the presence of *Streptococcus mutans*, when it is applied as coating or incorporated in different concentrations into the commercially available Transbond XT Light Cure Adhesive resin for braces cementation. The chemical and physical properties of the obtained materials were also evaluated.

## 2. Experimental

### 2.1. Materials

(3-Iodopropyl)trimethoxysilane (IPTMS, Sigma-Aldrich,  $\geq 95\%$ ), *N,N*-Dimethyldodecylamine (DMDA, Sigma-Aldrich, 97%), Tetraethyl orthosilicate (TEOS, Sigma-Aldrich, 98%), 3-(Trimethoxysilyl)propylmethacrylate (TMSPM, Sigma-Aldrich, 98%), Hydrochloric acid (HCl, J.T. Baker, 36.5–38%), ultrapure water deionized water and *N,N*-Dimethylformamide (DMF, Sigma-Aldrich, 99.8%). All compounds were used as purchased.

### 2.2. Synthesis, deposition and incorporation of IQAMS in Transbond XT Light Cure resin

The synthesis of IQAMS followed two steps:

In *step 1*, a round-bottomed 50 mL flask was protected from light and put into silicone oil bath. Then, 25 mL of DMF were added, followed by addition of 1158  $\mu\text{L}$  of IPTMS and 1753  $\mu\text{L}$  of DMDA. A condenser was attached to the flask and the solution was put under stirring and heated up to 110  $^{\circ}\text{C}$  and kept it for 24 h under reflux. A scheme of step 1 is presented in Fig. 1.

In *step 2*, after 24 h, still under stirring, the heating was turned off and once the temperature reached 25  $^{\circ}\text{C}$ , the condensation system was removed and 646  $\mu\text{L}$  of TEOS were added and after 5 min, 714  $\mu\text{L}$  of TMSPM. Then, 15 min later, 700  $\mu\text{L}$  of HCl 0.3 mol L<sup>-1</sup> were added to catalyze the hydrolysis and condensation process of the alcoxysilanes initiating the formation of the ORMOSIL network. The reaction proceeded for 1 h when the stirring was turned off. A scheme of step 2 is presented in Fig. 2.

The solution containing the IQAMS was transferred to a Teflon® mold and taken to a pre-heated furnace at 80  $^{\circ}\text{C}$  to evaporate the solvent for 48 h. The solid obtained product was put in a Soxhlet extractor deperated in ethanol for 24 h and then rota-evaporated. All these procedures were to ensure the quality of the product.

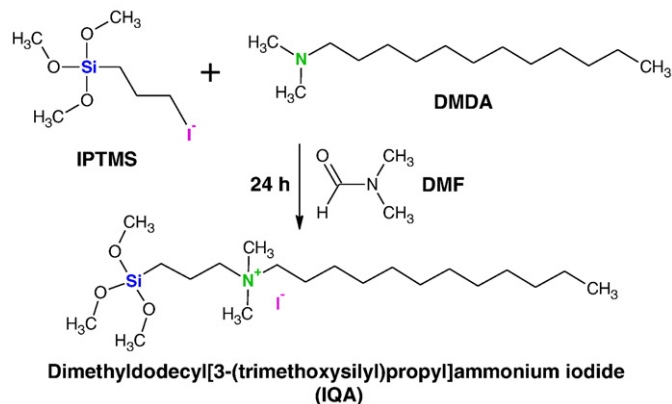


Fig. 1. Schematic representation of step 1.

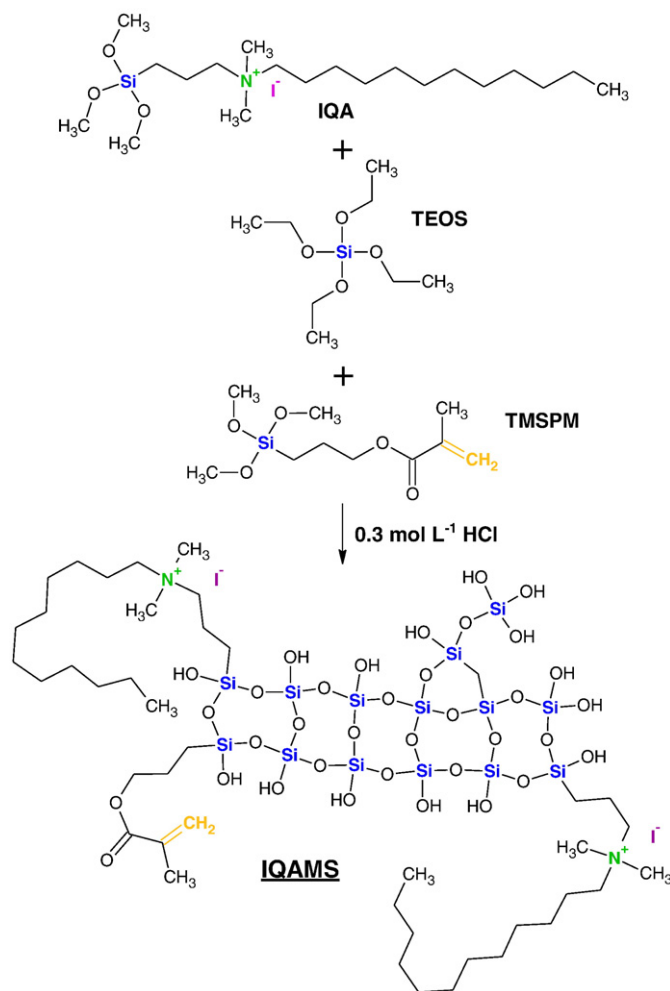


Fig. 2. Schematic representation of step 2.

For the incorporation, the IQAMS, obtained in the form of a viscous gel, was dispersed in 2 mL of ethanol and mixed to 11 g of Transbond XT composite resin (3 M ESPE) using the Speed Mixer™ Laboratory Mixer System (Synergy Devices Limited). Two different volumes of solution were used and the groups obtained are presented in Table 1.

A fourth group named TC, where C stands for coating, was obtained from application of 2 layers of IQAMS on the resin using disposable applicators (Microbrush).

### 2.3. Characterization techniques

#### 2.3.1. Chemical structure

Fourier transform infrared spectroscopy in transmission mode (T-FTIR) was performed in a SHIMADZU IRA-affinity-1 spectrometer in the range of 4000–650  $\text{cm}^{-1}$  with a spectral resolution of 4  $\text{cm}^{-1}$ . The samples were dispersed into potassium bromide (KBr) and pressed as pellets. Neat KBr pellet was used as background. FTIR in Attenuated Total

**Table 1**  
Groups and correspondent percentage and volume of IQAMS used.

| Group | wt% of IQAMS in the composite resin | Volume of IQAMS solution ( $\mu\text{L}$ ) |
|-------|-------------------------------------|--|
| T     | 0                                   | 0  |
| T10   | 10                                  | 735.7                                      |
| T15   | 15                                  | 1103.6                                     |

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