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Structural stability of DHMAI antibacterial dental composite following in vitro biological aging

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1. Introduction

The development of new and improved dental composites entails assessing whether they present good properties and stability during the period of use without influencing their effectiveness [1]. Methacrylate-based composites are the most esthetic restorations currently used in direct techniques. The arrangement of the three-dimensional network of resin composites depends on the monomer structure that constitutes the resinous matrix [2]. The dense network undergoes structural changes during the photopolymerization process. The methacrylate monomer system is transformed from a liquid state to a rubbery state, and then to a solid glass state. In addition, the structure of the monomers as well as the incomplete polymerization can greatly impact the final properties of the material [3]. Incomplete polymerization may have potential disadvantages. The release of residual monomers from composite resins can stimulate the growth of cariogenic microorganisms around restoration [4, 5]. Furthermore, several components were shown to induce allergic reactions and/or are potentially cytotoxic, genotoxic or mutagenic [6]. Thus, knowledge concerning the structural properties and behavior of dental composites is necessary to improve their performance [3, 7, 8, 9, 10].

The stability of dental composites during function is defined by the extent to which they preserve the same characteristics and properties they had at the time of manufacture [1]. The polymeric structure of dental composites can be degraded in aqueous media, either by a hydrolysis mechanism or an enzymatic reaction [11]. A recent study has shown that the cariogenic *Streptococcus mutans* (*S. mutans*) bacteria has an esterase activity comparable to that of human saliva attaining levels that could degrade dental composites [12]. This is

particularly relevant since microorganisms have a high affinity for dental composites and can colonize their surfaces [13].

The development of antibacterial dental composites aims to fight secondary caries and increasing dental restoration longevity [13]. Among the strategies established to achieve this objective, is the current use of the Quaternary Ammonium Methacrylate (QAM) as an antibacterial monomer. This approach has great potential for decreasing biofilm formation around restoration. On dental composite surfaces, the immobilized QAM causes lysis of bacteria by contact of their positive charge with the negative charge of the bacterial wall [14, 15]. Studies have shown that QAMs with 16 carbons in their alkyl chain are the most effective. A number of mono- and di-methacrylate monomers have been developed including N-DiMethylAmino-HexaDecyl Methacrylate (DMAHDM) [16, 17], methacryloxyethylcetyl Dimethyl Ammonium Chloride (DMAE-CB) [18, 19] and 2-methacryloxyethylhexadecyl Methyl Ammonium Bromide (MAE-HB) [20]. The inhibitory force of these monomers depends on the halogen counter ion of QAM and the optimum amount which can be incorporated into the dental composite [21]. The Dimethyl Hexadecyl Methacryloxyethyl Ammonium Iodide (DHMAI) was recently incorporated in dental composite formulation (75/25, wt/wt %) Bisphenol A Glycidyl Methacrylate and Triethylene Glycol Dimethacrylate (Bis-GMA/TEGDMA)). Our previous study of series of concentrations showed that 7.5% DHMAI had good mechanical properties and strong antibacterial activity against the cariogenic bacteria strain *S. mutans* [22].

The aim of this *in vitro* study was to investigate the structural stability, thermal, and physicochemical properties of a 7.5% DHMAI experimental dental composite, after 3 months of aging, in the presence of *S. mutans*. Two hypotheses were suggested: (1) DHMAI would not affect the properties of the dental composite; (2) the 7.5% DHMAI composite would

remain stable after 3 months of aging in the presence of *S. mutans* and would preserve its structural and physicochemical properties.

2. Materials and methods

2.1. Synthesis procedure of quaternary ammonium salt monomers (DHMAI)

The DHMAI ($C_{24}H_{48}O_2N^+I^-$) was produced using the Menshutkin synthesis which converts a tertiary amine to quaternary ammonium salts by reaction with an alkyl halide: 0.06 mol of 2-DiMethylAmino Ethyl Methacrylate (DMAEMA) was added to 0.05 mol of Iodohexadecane and 0.05 % of hydroquinone. All reagents were purchased from (Sigma-Aldrich, Saint-Quentin Fallavier, Isère, France) and were used without purification. The mixture was agitated under inert atmosphere at 50 °C for 12 hrs. The formed paste was washed with diethyl ether and then dried. DHMAI was obtained as a white powder [22].

2.2. Preparation of resin composites

According to the results of our previous study [22], two formulations (table 1) were selected for thermal analysis. DHMAI was added into (75/25, wt/wt %) (Bis-GMA/TEGDMA) with mass ratios of 7.5 wt%. The initiator system (Comphoroquinone (CQ) and DMAEMA), was added at mass ratio of 1 wt%. All reagents were purchased from (Sigma-Aldrich, Saint-Quentin Fallavier, Isère, France). Then, resin was filled with 71 wt% of silanized glass particles 8235-SCHOTT® (SCHOTT AG, Landshut, Bavaria, Germany) consisting of 10 % Al_2O_3 , 10 % B_2O_3 , 30 % BaO, and 50 % SiO_2 , (mean size 0.7 μm). The compounds were well blended using SpeedMixer™ (Hauschild, Hamm, North Rhine-Westphalia, Germany) to

obtain homogeneous pastes, and then stored in sealed containers in darkness. Resin composite without DHMAI was prepared as a control.

2.3. Specimen preparation and biological aging

Pellet samples (wt 80-90 mg) of each composite (table 1) were prepared in a Teflon mold of 6 mm diameter and 1 mm thickness. Samples were irradiated on both sides under LED lamp Elipar™ S10 (3M ESPE, Seefeld, Bavaria, Germany) for 40 s and then polished with 120-grit silicon carbide abrasive paper. The samples of each formulation were divided into two groups for before and after biological aging analysis.

For aging, *S.mutans* strain ATCC 25175 (Institut Pasteur, Paris, Île-de-France, France) were cultured in a brain heart infusion (BHI) medium. Pellet samples (n = 9) of each formulation (table 1) were UV sterilized (40 min) and placed in sterile flasks containing 50 ml BHI. Then, 10 ml of bacteria suspension at the optical density of $DO_{600} = (0.6 - 0.7)$ was added to each flask. The flasks were then incubated aerobically at 37 °C for 3 months. The culture media were renewed every 3 days and the purity of the bacteria was systematically checked by gram staining throughout the incubation period.

After 3 months of aging, the samples were collected and washed three times under sonication in sterile Dulbecco's Phosphate Buffered Saline (DPBS), to eliminate the dense biological deposits from the surface. The samples were then dried in air and protected from light.

2.4. Degree of conversion and residual functions rate of resin composites

DC measure [22] and the rate of RF were measured using FTIR spectroscopy IR 700 (Safas, Principauté, Monaco) with an attenuated total reflectance (ATR) sampling accessory. First,

the spectrum of uncured formulations (F1 control- F2 antibacterial) was obtained using 4 scans at 4 cm⁻¹ resolution. Then, the cured samples before and after aging ($n = 6$), prepared as described above, were analyzed. Fig. 1 shows the absorbance intensities of the aliphatic double bond (C=C) peak at 1638 cm⁻¹ and the internal aromatic (C=C) reference at 1607 cm⁻¹ before photopolymerization, after photo polymerization and after aging. The DC (%) and RF (%) were calculated according to the following equations:

$$DC_{\text{Before aging}} (\%) = [(P1/P2) - (P1'/P2')] / (P1/P2) \times 100$$

$$RF_{\text{Before aging}} (\%) = 100 - DC_{\text{Before aging}}$$

$$DC_{\text{After aging}} (\%) = [(P1/P2) - (P1''/P2'')] / (P1/P2) \times 100$$

$$RF_{\text{After aging}} (\%) = 100 - DC_{\text{After aging}}$$

Where $P1$, $P1'$ and $P1''$ are the absorbance peaks at 1638 cm⁻¹ of (C=C) aliphatic before photopolymerization, after photopolymerization, and after aging respectively. $P2$, $P2'$ and $P2''$ are the absorbance peaks at 1608 cm⁻¹ of (C=C) aromatic before photopolymerization, after photopolymerization and after aging respectively.

2.5. Thermal properties:

2.5.1. Characteristic properties assessed by DSC

DSC 820 (Mettler Toledo, Viroflay, Île-de-France, France) was used to identify the characteristic properties of experimental composites before and after aging. Samples (n=1) of 20-25 mg weight and 4 mg DHMAI particles were placed in aluminum crucibles and heated from 25 °C to 200 °C at a rate of 10 °C/min under 30 ml/min air flow.

2.5.2. Thermal expansion coefficient assessed by TMA

The CTE of experimental composites (n=3) was evaluated using a Thermo-Mechanical Analyzer TMA/SDTA 840 (Mettler Toledo, Viroflay, Île-de-France, France) before and after aging. The linear expansion (dimensional changes) was carried out by contact of a quartz probe with samples at 0.02 N under heat treatment ranging from 25-80 °C. Each sample was subjected to two heating runs at a 5 °C/min rate. After each measurement, the machine was cooled until it reached 25 °C over a period of 10 min; the CTE was calculated at the second heating run between 25 to 60 °C using internal software.

2.5.3. Thermal characterization, stability and degradation behavior assessed by TGA

Thermal characterization, stability and degradation behavior were studied using the TGA/DSC2 STAR[®] System (Mettler Toledo, Viroflay, Île-de-France, France). Samples of experimental composites before and after aging (n=1) were heated in an aluminum crucible (range 25-1000 °C), at a heating rate of 10 °C/min, under 50 ml/min air flow. The mass losses of sample composition were measured by the TG curves as a function of the temperature. The DTG curves were obtained by derivative TG curves to determine the most obvious temperatures of mass losses.

2.6. Statistical analysis:

Due to the small number of our samples, non-parametric tests were used; the Mann-Whitney Wilcoxon test for comparison between-group analyses (F1 group vs F2 group at $t = 0$ then at $t = 3$ months), and the Wilcoxon signed rank test for within-group analyses ($t = 0$ vs $t = 3$ months for F1 and F2 groups respectively). Statistical significance was accepted at $p < 0.05$.

3. Results

3.1. Degree of conversion and residual functions rate of resin composites

The DC and RF rates of experimental dental composites are shown in [Fig.2 A](#) and [B](#) respectively.

When compared to the control composite F1, DC% was significantly higher and RF% significantly lower in the antibacterial F2 composite, at $t = 0$ ($p = 0.02$). After aging, the RF% was not significantly different between F1 and F2 ($p=0.38$), but both composites showed a significant decrease in RF% when compared to their $t = 0$ values ($p = 0.03$); [Fig.2](#).

3.2. Thermal behavior:

3.2.1. Characteristic properties assessed by DSC

The DSC curves of the F1 and F2 composites obtained at a $10\text{ }^{\circ}\text{C} / \text{min}$ heating rate at $t = 0$ are shown in [Fig. 3](#).

The DSC curves showed different thermal behaviors between the composites F1 and F2. Two glass transitions appeared in the composite F1 ($T_{g1} = 53\text{ }^{\circ}\text{C}$ - $T_{g2} = 145\text{ }^{\circ}\text{C}$) while F2 showed two glass transitions ($T_{g1} = 54\text{ }^{\circ}\text{C}$ - $T_{g2} = 173\text{ }^{\circ}\text{C}$) and an endothermic peak at $T = 85\text{ }^{\circ}\text{C}$. The temperatures for the F1 and F2 T_{g1} transitions were close while the F2 T_{g2} occurred at a higher temperature than that of F1 [Fig. 3](#). The endothermic reaction peak of the F2 composite was characteristic of the fusion of the antibacterial agent DHMAI.

The comparison of the F1 and F2 DSC curves before ($t = 0$) and after aging ($t = 3$ months) are represented in the [Fig. 4 A- and B](#), respectively. Concerning the thermal behaviors of each composite at $t = 0$ and after 3 months of aging, F1 showed differences over time while F2 remained stable [Fig. 4](#). At $t = 0$, the F1 composite showed two glass transitions ($T_{g1} = 53\text{ }^{\circ}\text{C}$, and $T_{g2} = 145\text{ }^{\circ}\text{C}$) whereas, after 3 months, three glass transitions occurred ($T_{g1} = 52\text{ }^{\circ}\text{C}$, $T_{g2} = 77\text{ }^{\circ}\text{C}$ and $T_{g3} = 132\text{ }^{\circ}\text{C}$). Moreover, the high temperature glass transition of F1 was lower at $t = 3$ months compared to $t = 0$ ($T_{g3} = 132\text{ }^{\circ}\text{C}$ vs $T_{g2} = 145\text{ }^{\circ}\text{C}$; [Fig. 4-A](#). After 3 months, the *SM* + BHI caused degradation of the dental composite.

Conversely, the F2 composite showed two glass transitions at both $t = 0$ ($T_{g1} = 54\text{ }^{\circ}\text{C}$, $T_{g2} = 173\text{ }^{\circ}\text{C}$) and $t = 3$ months ($T_{g1} = 50\text{ }^{\circ}\text{C}$, and $T_{g2} = 176\text{ }^{\circ}\text{C}$) which occurred at similar temperatures before and after aging [Fig. 4- B](#), indicating the effect of the antibacterial agent DHMAI insertion in the F2 composite.

3.2.2. Thermal expansion coefficient assessed by TMA

The comparison of thermal expansion curves before and after aging at the second thermal cycle (heating 2) is shown in [Fig. 5-A and B](#) for composites F1 and F2, respectively.

As shown by the thermograms obtained at the second heating run, the expansion curves of F1 and F2 were superimposable at $t = 0$. After 3 months of aging in *SM* + BHI, the curves

showed a slight decrease in the volumetric changes for both experimental composites but more marked for F1 [Fig. 5](#).

There was no significant difference between the CTE of F1 and F2 at $t = 0$ ($p=1$). CTE was lower for both composites after 3 months of aging in *SM + BHI*, but this decrease was not statistically significant. ($p = 0.25$ for F1 and $p=0.5$ for F2); [Fig. 6](#).

3.2.3. Thermal characterization, stability and degradation behavior assessed by TGA

The TG and DTG curves of experimental composites F1 and F2 before and after 3 months of aging in *SM + BHI* are shown in [Fig. 7](#). The mass loss values and the temperatures at degradation onset obtained from TG and DTG curves are reported in [Table 2](#).

TG curves revealed that the thermal degradation of all experimental composites occurred in 2 steps [Fig. 7-A](#). The DTG showed a different trend for F2 curve compared to F1, particularly during the first step (200-500 °C) where an overlap of two peaks appeared [Fig. 7-B](#). Moreover, at $t = 0$, degradation of F2 began before that of F1, with the onset temperature being 300 °C for F1 and 382 °C for F2 [Table 2](#).

After 3 months of aging in *SM + BHI*, the onset temperature for degradation of the F1 composite decreased by 26 °C (356 °C) compared to $t = 0$ (382 °C) while this temperature increased by 2 °C (300 °C vs 302 °C) for F2 [Table 2](#).

Prior to resin thermal degradation which occurs between 30 -200 °C, slight mass losses are observed for both composites [Table 2](#). After thermal degradation, total mass losses were not significantly different between $t=0$ and $t=3$ months for both composites. Mass losses are proportional with the organic phase ratio of the experimental formulations.

4. Discussion

The viscosity of the resinous matrix is one of the factors influencing the photopolymerization and the DC of methacrylate-based dental restorations [23, 24]. The decrease of the viscous monomer "BisGMA" ratio, induced by DHMAI incorporation, led to a better mobility of reagents during network formation. DC was thus improved, implying a decrease in RF compared to the control. Results showed a significant increase in DC and therefore decrease in RF levels after 3 months of incubation. In terms of the structural stability, RF is more explicit than DC to reveal monomer release following the bacterial action. Of note, although F2 showed lower RF than F1 at $t = 0$, both composites displayed a similar level of RF after 3 months, as F2 tended to release less residual monomers (release rate of 5.78%) than F1 (release rate of 7.43%). A previous study [25] showed that the experimental bacterial medium "*S.mutans*" accelerated the release of trapped monomers compared to a simple aqueous medium. Moreover, Bourbia et al [12] showed that *S. mutans* has esterase activity that degrades dental composites. Therefore, the difference in the release rate between F1 and F2 can be attributed to the antibacterial activity present on the F2 surface, which could prevent the bacterial effect on the monomer release and thus enhanced the dental composite stability. We hypothesized that slow and gradual release over time could reduce the risk of cytotoxicity.

For the DSC, F1 showed a second glass transition $T_{g2} F1 = 145$ °C which could be considered the highest glass transition temperature after which the dental resin would be completely polymerized. F2 showed a second glass transition at a temperature considerably higher than the T_{g2} of F₁, as well as a melting peak characteristic of the antibacterial agent DHMAI, at $T = 85$ °C as shown in Fig. 3. The absence of a distinct glass transition temperature of DHMAI reflected its conversion in the synthesis of the dental resin. Thus, the antibacterial

agent co-polymerized well with the other monomers [26]. This characteristic was important for assuming the branching of the antibacterial agent within the network. The glass transitions correlate with the crosslinking density (DC) and are related to the mobility of monomers trapped in the dental resins, which explains the difference in intensity of the tg peaks between the composites [8, 27, 28]. The higher temperature for F2 Tg₂ compared to F1 Tg₂ could be explained by chain mobility restriction which would delay post-polymerization [28]. DSC thus confirmed that DHMAI contributed to improving monomer conversion during photopolymerization. The present results are coherent with a number of other studies using DSC [8, 27, 28].

After 3 months of aging in the biological medium (SM+ BHI), the structural stability of F1 was affected. A unique vitreous transition, at low temperature, appeared at t = 0 (Tg₁=53 °C), while two glass transitions, at low temperature, occurred after aging (Tg₁ = 52 °C, Tg₂ = 77 °C) Fig. 4-A. The second transition could be attributed to the existence of a secondary network nested in the initial network. The latter was revealed after 3 months of aging in the bacterial biological medium by relaxation and swelling. Therefore, the difference in thermal behavior at t = 0 and after aging could be explained by the degradation effect caused by direct contact with bacteria. Furthermore, since the glass transition at high temperature after 3 months of aging (Tg₃ = 132 °C) was lower than at t = 0 (Tg₂ = 145 °C), it is likely that the culture medium had plasticized the network and thus decreased the thermopolymerization temperature. This was not the case for F2, indicating that DHMAI co-polymerization contributed to inhibiting dental composite biodegradation. Thus, the 7.5% DHMAI formulation provided significant modifications to the thermal behavior of the composite resins without affecting its TG after 3 months of aging in the presence of *S. mutans*. The DSC confirmed our hypothesis that DHMAI enhanced structural stability and inhibits biodegradation.

CTE measurements were performed on the thermal expansion curves at the second heating (curves not shown). We considered that a first heating before analysis was necessary to calibrate the dental composites, to erase their thermal past, and to ensure reproducibility and reliability of the CTE values [29]. At $t=0$, results showed that incorporation of 7.5% DHMAI into the (75/25) (BisGMA / TEGDMA) composite F2, did not affect the CTE of the dental composite [10]. F1 and F2 showed tendency towards a decreased CTE after 3 months of aging but was not statistically significant. In terms of volumetric variations, our hypothesis that the incorporation of 7.5% of DHMAI does not affect the CTE thermo-mechanical property of the antibacterial composite F2 was confirmed.

Based on the TGA and DTG results, the influence of DHMAI can be seen in the degradation progression which started earlier compared to composite control and in the change of the first decomposition shape. In fact, the BisGMA/TEGDMA (75/25 % wt/wt) co-polymerization of control composite F1 showed thermal stability. The first mass loss between 380 and 570°C occurred in a one-step process reflecting network homogeneity[30]. While 7.5 % DHMAI composite showed an overlap of two peaks between 290 and 580 °C. This could be attributed to the heterogeneity of the matrix structure [31] due to the incorporation of DHMAI and could explain why the degradation started earlier than the control composite. After 3 months of *S.mutans* aging, F1 showed a difference in its thermal behavior compared to $t = 0$. DTG illustrated similar decomposition peaks. Mass losses between $t = 0$ and $t = 3$ months were similar. But, the temperature at the onset of degradation decreased by 26 °C when compared to $t = 0$. This indicates that the stability limit of the F1 composite was affected by the biological medium. However, F2 showed a thermal behavior similar to that of $t = 0$. The temperature for onset degradation, the decomposition peaks, and the mass losses were similar at $t = 0$ and $t = 3$ months. F2 was thus more thermally stable than F1.

Following the first thermal degradation, all tested composites showed another mass loss in the range of 540-700 °C. This mass loss was attributed to the decomposition of the matrix attached to the condensed silanes coupling agents at the filler surface [31, 32]. Also, in our study, a minor loss of mass occurred before the beginning of the thermal degradation of F1 and F2, likely due to humidity and solvent evaporation [33]. Mass losses were analogue for all composites at t=0 and t=3 months and coherent with the organic phase ratio $\approx 29\%$ of the experimental formulations. The structural composition of all composites was not affected after 3 months of the *in vitro* biological aging, but the F1 control exhibited different thermal behavior. This evolution was different from the F2 evolution and could indicate signs of biodegradation. However, we confirmed once again that 7.5 % DHMAI provide significant modifications to the thermal behavior of dental composites and contribute to the antibacterial composite structural stability enhancement after 3 months of the *in vitro* aging in (SM + BHI).

Despite the limitations of the present *in vitro* study (experimental *in vitro* model, experimental aging with one bacterial strain, relative short aging time...), according to the FTIR-ATR, DSC, TMA, and TGA analyses, the initial hypotheses were confirmed: incorporation of 7.5 % DHMAI did not affect the initial composite properties, enhanced the DC, and led to different thermal behaviors. Moreover, the hypothesis concerning the stability of 7.5 % DHMAI composite in the current experimental conditions (the biological medium SM + BHI) was also validated. DHMAI created an antibacterial protective barrier for the dental composite and preserved its structural and physicochemical properties after 3 months of *in vitro* aging. This behavior was confirmed by the biodegradation of the control composite that did not contain the antibacterial agent. Thereafter, it would be necessary to evaluate the long-term efficiency, cytocompatibility, and *in vivo* behavior of the 7.5 % DHMAI composite.

5. Conclusion

This study highlighted the contribution of DHMAI in enhancing the structural stability of a dental composite after 3 months of *in vitro* biological aging. This stability was demonstrated by:

- A lower residual functions rate and gradual release over time compared to the control composite which could reduce the risk of cytotoxicity induced by monomers release and enhance restoration longevity.
- Unchanged thermal behavior, demonstrating the non-degradation of the dental composite after *in vitro* biological aging and the preservation of its structural stability over time.

In addition to the strong antibacterial effect of 7.5% DHMAI composite, the latter has shown an ability to protect the dental composite against biodegradation. The DHMAI monomer shows promising results and its use could be extended to other dental bioactive materials

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Figure captions

Fig. 1: Variation of the vibration bands at 1638 cm^{-1} of aliphatic (C=C) functions compared to vibration bands at 1607 cm^{-1} of aromatic (C = C) functions.

Fig. 2: A) Degree of conversion of dental composites. B) Residual functions rate of dental composites. Error bars represent standard deviation ($n = 6$). Groups sharing same label letters (a, b, c) had statistically similar values ($p > 0.05$).

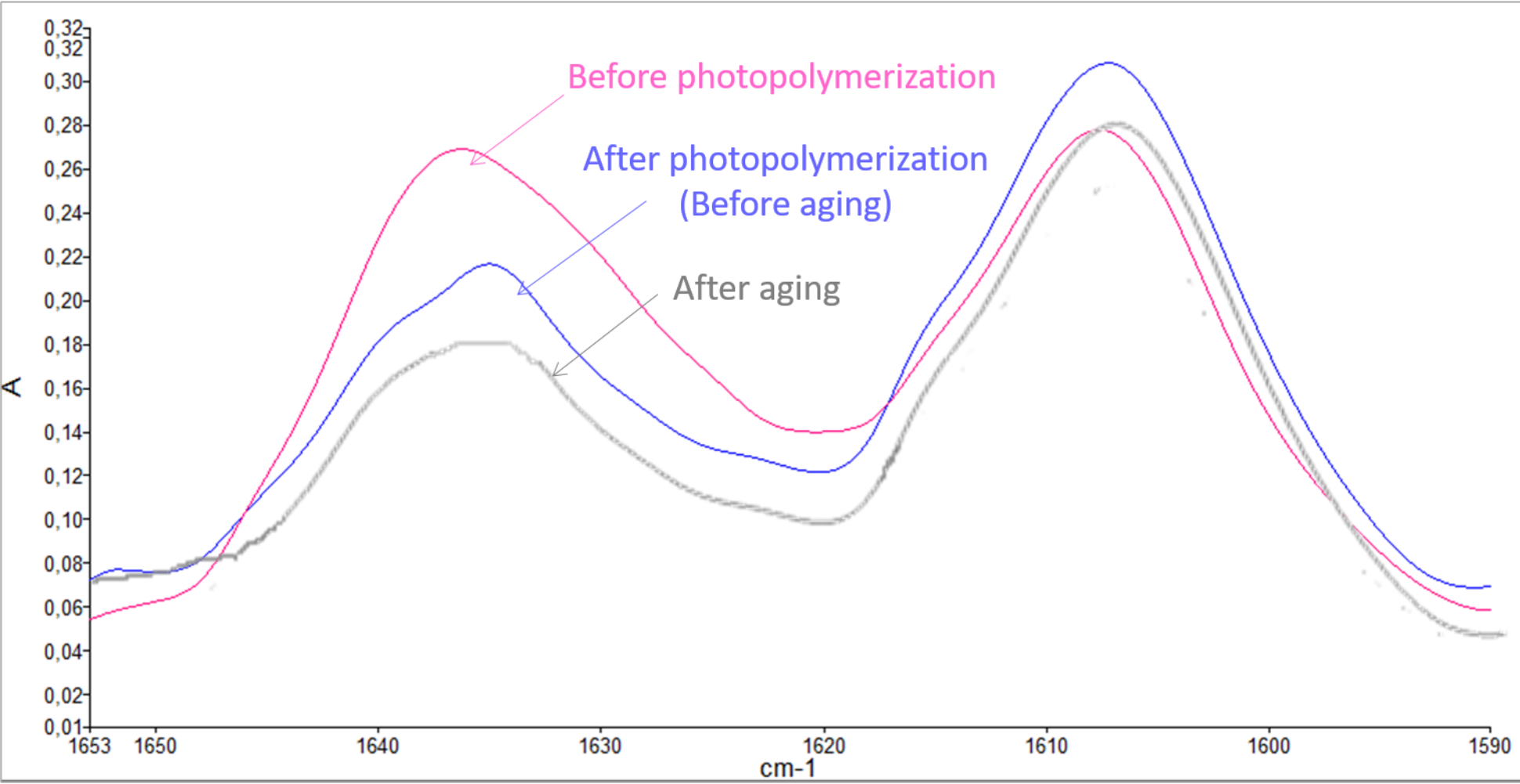
Fig. 3: Differential scanning calorimetry thermograms of composites F1 control and F2 antibacterial.

Fig. 4: Differential scanning calorimetry thermograms at $t=0$ and after 3 months of aging in (*S. mutans* and BHI). A) Control composite F1. B) Antibacterial composite F2.

Fig. 5: Thermo-mechanical analyses curves of the dimensional change of composites before and after 3 months of aging in *S. mutans* obtained in run 2. A) Control composite F1. B) Antibacterial composite F2.

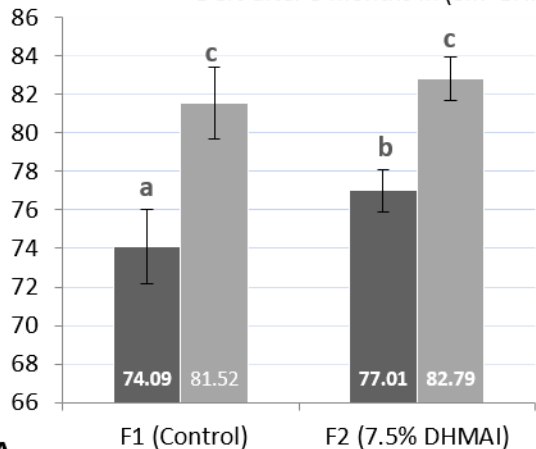
Fig. 6: Variation of thermal expansion coefficient of composites F1 and F2 before and after 3 months aging in *S. mutans*. Groups sharing same label letter (a) had statistically similar values ($p > 0.05$).

Fig. 7: Thermal degradation behavior of composites F1 and F2 before and after 3 months of aging in *S. mutans*. A) Thermo-gravimetric curves B) Differential Thermo-gravimetric curves.



Degree of conversion(%)

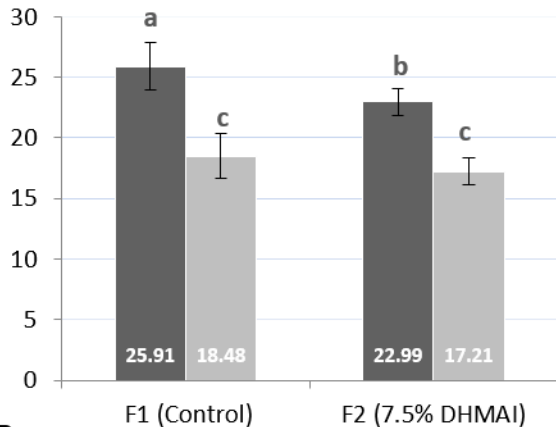
■ DC% t0
■ DC% after 3 months in (Sm+BHI)



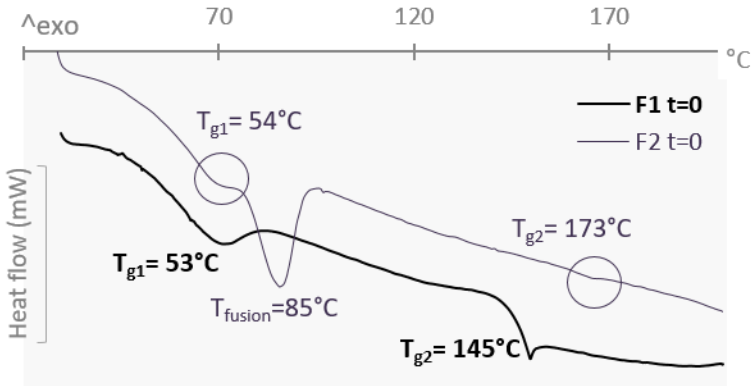
A

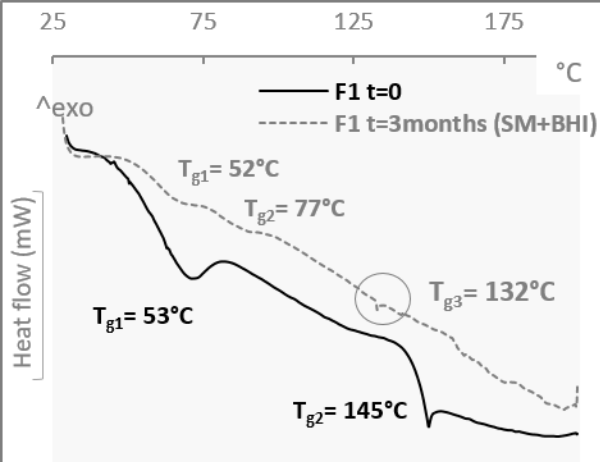
Rate of residual functions (%)

■ RF% t0
■ RF% after 3 months in (Sm+BHI)

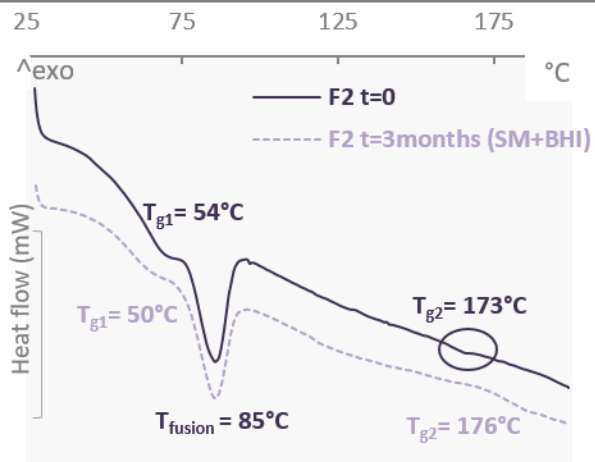


B



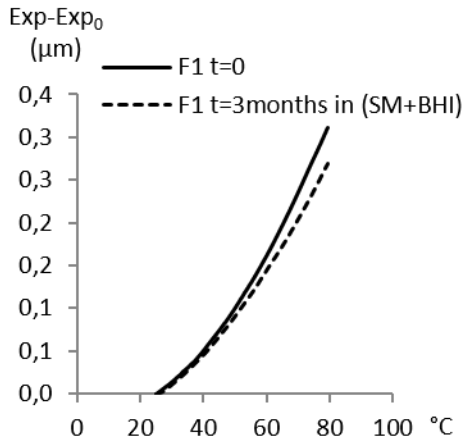


A



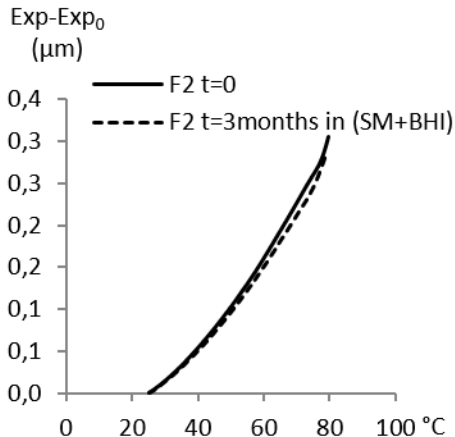
B

Control composite



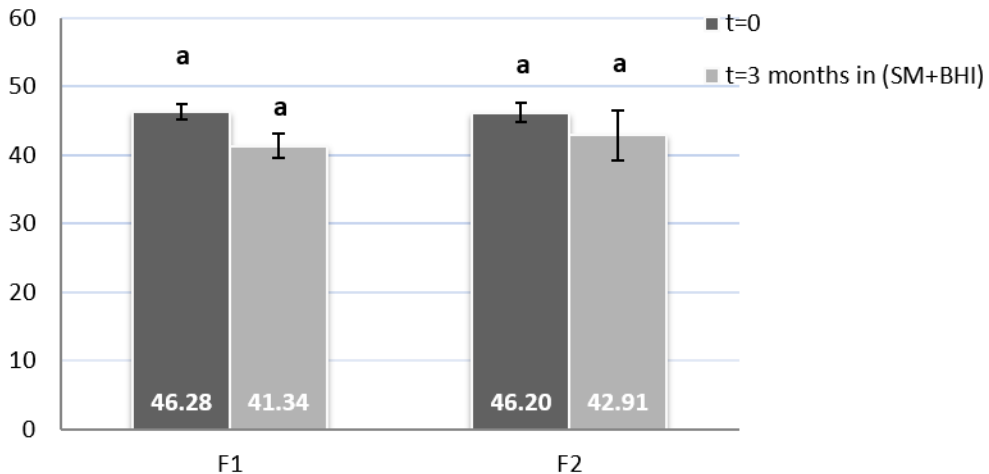
A

Antibacterial composite

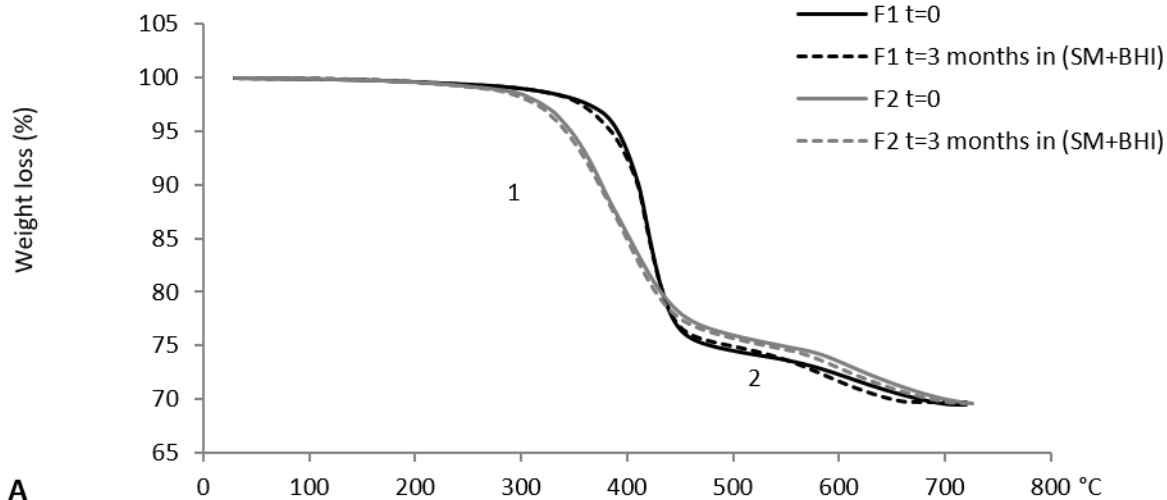


B

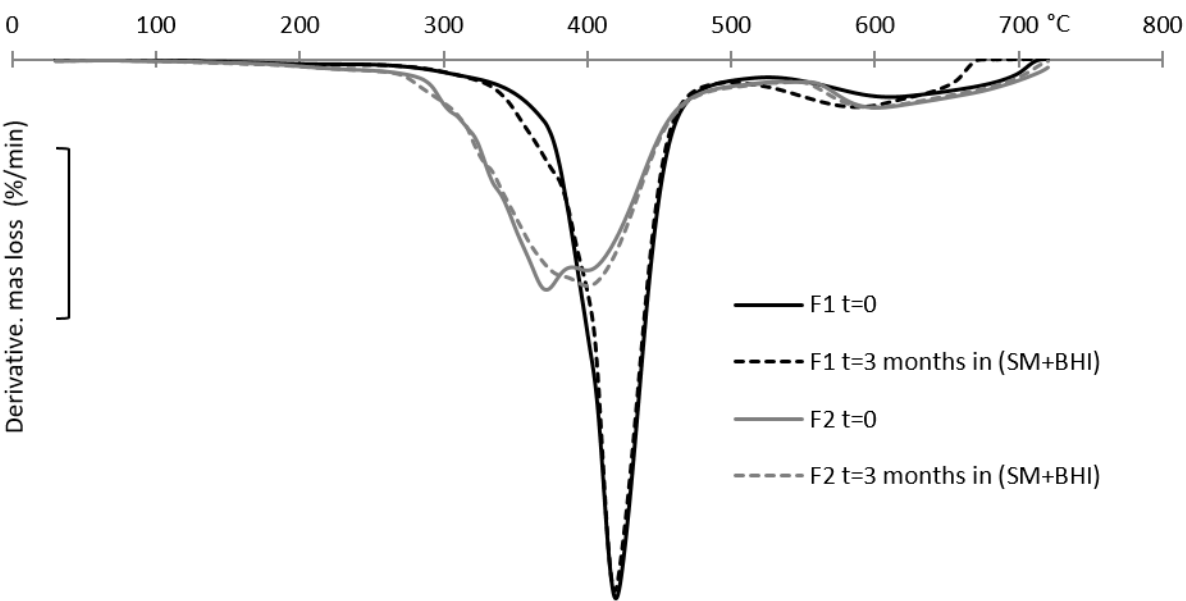
Variation of thermal expansion coefficients ($10^{-6}/C^{\circ}$)



Δ exo



A



B

Table 1: Composition of the organic phase of the experimental composites

Table 1- Composition of the organic phase of the experimental composites						
Formulations		Organic phase 29 wt%				
		BT (75/25) wt%	MPC wt%	DHMAI wt%	CQ wt%	DMAEMA wt%
F1	Control	28.42	0	0	0.29	0.29
F2	Antibacterial 7.5 % DHMAI	26.29	0	2.13	0.29	0.29

BT = Bisphenol A GlycidylMethacrylate (BisGMA) mixed with Triethylene Glycol Dimethacrylate (TEGDMA); MPC = MethacryloyloxyethylPhosphorylCholine; DHMAI = DimethylHexadecylMethacryloxyethyl Ammonium Iodide; CQ = Comphoroquinone; DMAEMA = DimethylaminoEthylMethacrylate.

Table 2: Data of experimental composites obtained by Thermo Gravimetric Analysis and its derivative curves.

Table 2: Data of experimental composites obtained by Thermo Gravimetric Analysis and its Derivative curves.				
Thermal degradation reaction steps	F1 t0	F2 t0	F1 3 months (SM+BHI)	F2 3 months (SM+BHI)
Loss before degradation (%)	0.6	0.26	0.62	0.32
Mass loss 1 (%)	25.26	24.62	24.47	24.66
Onset (°C)	382	300	356	302
Mass loss 2 (%)	4.63	5.51	5.18	5.45
Onset (°C)	577	580	541	576
Total mass loss 1+2 (%)	29.89	30.12	29.68	30.12

F1: Control composite, F2: Antibacterial composite, SM: *S. mutans*, BHI: brain heart infusion medium.