Photoacoustic analysis of dental resin polymerization

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Abstract: In this work, we use the photoacoustic technique to monitor the curing process of diverse dental materials, as the resins chemically activated (RCA). The results obtained reveal that the composition of a determined RCA significantly alters its activation kinetics. Photoacoustic data also show that temperature is a significant parameter in the activation kinetics of resins. The photoacoustic technique was also applied to evaluate the polymerization kinetics of photoinactivated resins. Such resins are photoinactivated by incidence of continuous light from a photodiode. This leads to the polymerization of the resin, modifying its thermal properties and, consequently, the level of the photoacoustic signal. Measurements show that the polymerization of the resin changes the photoacoustic signal amplitude, indicating that photoacoustic measurements can be utilized to monitor the polymerization kinetics and the degree of polymerization of photoinactivated dental resins.

1. INTRODUCTION

The photoacoustic (PA) technique has been used for more than 20 years as a work tool in diverse fields of biological and biomedical sciences, including odontology [1-5]. Through PA measurements, it is possible to obtain information on the characteristic times involved in photoinitiated processes, as the photopolymerization of composite resins. In this work, we use the PA technique to monitor the curing process of diverse dental materials, as the resins chemically activated (RCA). In our study, resin samples with diameter 0.5cm were analyzed to evaluate the polymerization of RCA for different monomer/polymer ratios and submitted to different temperatures. The PA technique was also applied to evaluate the polymerization kinetics of photoinactivated resins. These resins are photoinactivated by incidence of continuous light (λ = 475 ± 15nm) from a photodiode. As a result of the photopolymerization process, the thermal properties of the resin are modified and, consequently, there is a change in the level of the photoacoustic signal.

1. METHODOLOGY

The experimental setup included a 250W tungsten lamp as the light source. Light was chopped with a mechanical chopper at 17Hz and directed to a photoacoustic cell closed in one side by a glass window and in the other side by an aluminium sheet. On the aluminium external face the resin sample was put to polymerize chemically or by photoactivation. The photoacoustic signal was recorded as a function of time.

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3. RESULTS AND DISCUSSION

3.1 Temperature dependence

Figure 1 shows the results obtained for chemically activated resins maintained at 22°C, 29°C and 37°C, following the ratio monomer/polymer indicated by the producer (0.2g monomer:0,5g polymer).

![Figure 1: Time evolution of the PA signal for resins chemically activated (RCA) at different temperatures.](image)

Measurements performed at 22°C show the slowest polymerization process. These results confirm that, at higher temperatures, polymerization is faster — in clinical treatments, ambient temperature may affect the period available for adjustments of the resin during polymerization.

3.2 Monomer/polymer ratio

Figure 2 shows time evolution of the PA signal phase for chemically activated resins prepared with different monomer/polymer (m/p) ratios.

![Figure 2: Time evolution of the PA signal for RCA prepared with different monomer/polymer (m/p) ratios.](image)

In resins containing a higher m/p ratio, the initial polymerization phases are fast, because of the easy diffusion of the monomers among the polymer units; however, the final phase (t > 5 min) is slower. On the other hand, resins with less monomers show a delay in the beginning of the polymerization, but a fast evolution in the final phase. The PA curve that corresponds to the proportion indicated by the producer presents an intermediate behavior in both phases.
3.3 Photopolymerized resins

Figure 3 presents the time evolution of the PA signal amplitude during polymerization of a photoactivated resin. The PA signal amplitude increases promptly upon incidence of the photodiode light, indicating the beginning of the polymerization process. It can be noticed that a large transition in the PA signal amplitude takes place in a few seconds for photoactivated resins under polymerization.

![Graph showing PA signal amplitude vs. time](image)

Figure 3. Time evolution of the PA signal amplitude during the photopolymerization process. The photodiode was switched on at $t = 50$ s (off at 70 s).

For the different resins studied, the PA amplitude generally increases with polymerization, corresponding to a reduction of the thermal effusivity of the sample. This could be explained by the diminution of the specific heat, which occurs for materials that suffer some phase transition from a less organized state to a more organized structure.

4. CONCLUSIONS

The PA technique can be employed in the study of the polymerization kinetics of both chemically activated resins and photoactivated resins. The results obtained reveal that the composition of a determined RCA affects its activation kinetics. In this way, the PA technique can be employed to determine the best monomer/polymer ratio for a given RCA. Photometric data also show that temperature is a significant parameter in the activation kinetics of resins. Finally, measurements indicate that the photopolymerization of a resin alters the corresponding PA signal amplitude. As the experimental configuration utilized consists of an open system, it is possible to investigate the effect of different light sources in the photopolymerization process. Other perspective of work involves a systematic study of the relation between PA signal amplitude and the polymerization level.

References