Electron spin resonance, a useful analytical technique for studying the kinetic decay of free radicals trapped in dental resin

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INTRODUCTION Electron spin resonance (ESR) spectroscopy is used to detect and characterise free radicals present in solid or liquid phases such as organic radicalar species (allylic and propagating radicals) trapped in dental resins [1]. The aim of this presentation is to show how this analysis tool can be useful to follow the time evolution of the concentration of free radicals when dental resins are photopolymerised in various conditions and stored in various environments.

METHODS Experimental unfilled resin composites were prepared using a Bis-GMA/TEGDMA 70/30 mixture (wt%/wt%). A photoinitiator consisting of a camphorquinone/amine mixture (50/50 wt%) was added to the composites in the proportion of 1%.

Immediately after photopolymerisation in suitable moulds, we followed the kinetics of the EPR peak variation intensity for each radical for six hours, at ambient temperature. After the end of each kinetic follow-up, the resin bar was immersed in distilled water at 25 or 37°C. For some experiments, the storage environment was different (air, oxygen, nitrogen at controlled temperature). The peak intensities were followed over time (24 hours, 1, 2, 3 and 4 weeks) after the photopolymerisation. The samples were stored in the different environments between each spectroscopic examination.

RESULTS All the results showed decreasing free radical concentration kinetics that comprised two domains (*Fig 1*) :

-a first, between 0 and 24 hours, where the decay rate measured was rapid (200 u.a.day^{-1}), constant, and independent of the dental resin storage conditions. One interpretation of this observation involves the relaxation of the free volumes existing in the vitrified resin [2].

- a second, extending from 24 hours to one month and more, where the rate measured was weaker (3 to 100 u.a.day^{-1}), and decreased progressively or stayed constant depending on the storage conditions. A first analysis of this second domain suggested a free radical oxidation phenomenon.

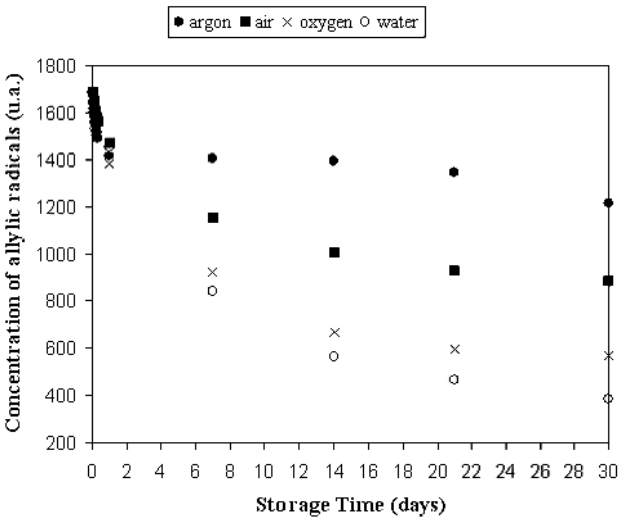

Fig. 1: Concentration decay of the allylic free radicals with time in various storage conditions of the resins.

PERSPECTIVES These results show how ESR spectroscopy can provide new information which may lead to a better understanding of the phenomena existing in dental resins. We plan to pursue and refine these experiments by using specific EPR methods (spin-trap for example) and to compare present and future results with those published in the literature concerned with the leaching in physiological media of compounds present in dental resins.

REFERENCES [1] D. Truffier-Boutry, X. A. Gallez, S. Demoustier-Champagne, J. Devaux, M. Mestdagh, B. Champagne, G. Leloup., *J. Polym. Sci. Part A : Polym Chem*, 2003, 41(11), 1691-1699 ; [2] D. Truffier-Boutry, S. Demoustier- Champagne, J. Devaux, J-J. Biebuyck, M. Mestdagh, P. Larbanois, G. Leloup., *Dental Materials*, 2006, (22)5, 405-412.