Rheology for a calcium phosphate ceramic in endodontics

J. Bosco , A. Fatimi, B.Enkel, A.Jean, P. Weiss
INSERM, U791, Laboratoire d'Ingénierie Ostéo-Articulaire et Dentaire, LIOAD, 1 Pl A. Ricordeau, F- 44042 Nantes cedex 1, France
Univ Nantes, UFR d'odontologie, 1 Pl A. Ricordeau, F- 44042 Nantes cedex 1, France.

Introduction

The apex remains the key to endodontic success, and therapeutic action still focuses on this biological repair unit. Current filling materials are toxic to periapical tissues and calcium phosphate materials, due to their biocompatibility and bioactive properties, may be viewed as possible alternatives.
The aim of this study was first to determine whether an injectable bone substitute could be used to obtain further healing of apical tissue by the neoformation of a mineralized barrier. In the next step, the work focused on rheological measurements as a tool for physical characterisation and on improvement of the injection technique.

Materials and methods

Injectable bone substitute

The ceramic was a biphasic calcium phosphate (BCP, Biomatlante, Vigneux de Bretagne, France) with a 60/40 HA/ß TCP weight ratio. A cellulose-derived polymer (Methyl-Hydroxyl-Propyl-Cellulose HPMC) was used because of its biocompatibility and rheological properties, which confer injectability on the final composite. Briefly, a solution of 2 % or 3% of HPMC was prepared by dissolving dry raw HPMC powder (E4M Premium EP, Colorcon, Bougival, France) in bidistilled water with stirring for 48 hours. The composite biomaterial was obtained by mixing a 2% or 3% HPMC solution with the 40-to 80-µm BCP granules in different weight ratios. The resulting composite was sterilized at 121°C for 20 minutes.

Animal experiments:

Six adult female sheep, with a mean weight of 64Kg, were used for endodontic treatment (n=8). Single-root teeth were prepared using the Hero system, the haemorrhage was controlled, and the sterilized materials to be tested were injected with a "lentulo". The animals were sacrificed at 12 weeks.
All the teeth were then embedded in methyl methacrylate and cut into parallel sections (100µm) with a diamond saw (Leitz 1600).
Qualitative observations were performed by light microscopy on the stained sections (Goldner trichromatic system). Other Sections were observed by Scanning Electron Microscopy (SEM). Energy dispersive X-ray (EDX) microanalysis was performed with an EDAX.

Rheological study

Rheology concerns the flow and deformation of a suspension and, in particular, its behaviour in the transient area between solid and fluid.
Rheological measurements were performed using the RheoStress300 rheometer (ThermoHaake®, Germany), using a plane/plane geometry with a cone diameter of 60mm. The rheometer was equipped with a circulating water bath to control temperature.
Zero-shear viscosity was determined from the flow curve of viscosity against shear rate. The experiment was carried out in rate-controlled mode at 25°C.
Dynamic oscillatory measurements were carried out to determine the elastic (G') and viscous (G") moduli as a function of frequency (*f*) at a fixed stress (τ) and fixed temperature. The experiment was carried out in stress-controlled mode at 25°C.

Results and Discussion

The animal experiment results obtained were grouped as "success", "uncertain" or "failure" depending on the amount of calcium phosphate granules at apical level: complete filling, partial filling and absence. The *in vivo* experimentation showed radiological apical sealing in 5 samples (arrows). SEM revealed mineral formation at the apex level with mineral tissue conduction between the BCP granules for 6 teeth but, for 5 samples, the apical amount of BCP granules could be classified as "partial filling". Only one tooth showed good apical sealing with an amount of BCP classified as "good filling". The BSE image in SEM showed a homogeneous level of mineralization between and around the granules and in close contact with them. Its density was the same as that of the dentin walls.

To improve the injectable properties, rheological measurements were performed. An increase in the amount of BCP led to an increase in the elastic (G') and viscous (G") moduli. For suspensions in 48 and 50% by weight, G" was higher than G', which implied a liquid behaviour of the suspension at a frequency of 1 Hz and a stress of 1Pa. For a given frequency, G' became higher than G", which implied that the suspension presented a solid behaviour. For higher concentrations, G' was higher than G", which implied a solid behaviour.
The crossline occurred at a lower frequency with a larger amount of BCP granules. A first step of correlation between these results and injection experiments was performed to model the rheological properties of this suspension.

Conclusion

Calcium phosphate materials make it possible to create tight bonding with mineralized tissues, and the formation of a mineralized barrier could ensure apical hermeticity. There are several tests for characterizing materials rheologically and the selection of such tests will depend on both the application and the injectability in this type of work.

References

[1] P. Weiss and coll. , Key Engineering Materials **284** (2004), 1053-1056.
[2] P. Weiss *et al.*, Bone **25** Suppl. 2 (1999), 67S-70S.