THESE de DOCTORAT DE L'UNIVERSITE DE LYON

opérée au sein de

l'Université Claude Bernard Lyon 1

Soutenance publique le 3 mai 2018 à 09H30
salle des Thèses de la faculté d'odontologie, par :

Delihta Fernando

Novel Mesoporous Bioactive Glasses (MBGs) as fillers in dental adhesives "Synthesis, Physico-chemical and biological evaluation".

Devant le jury composé de :

Pr Hill Robert	Queens Mary University	Rapporteur
Pr Turkun Sebnem	Ege University	Rapporteure
Pr Berdal Ariane	Université Paris Diderot	Rapporteure
Dr Jackson Phil	LUCIDEON Ltd	Examinateur
Pr Colon Pierre	Université Paris Diderot	Directeur de thèse
Dr Attik Nina	Université Lyon 1	Co-directrice de thèse
Dr Pradelle-Plasse Nelly	Université Paris Diderot	Co-directrice de thèse
Pr Grosgogeat Brigitte	Université Lyon 1	Examinatrice

Abstract

Improving the stability of adhesive dentin interface is crucial to extend the longevity of composite restorations. Remineralization through use of ion releasing materials is a promising approach to protect the hybrid layer from hydrolytic and enzymatic degradation. Mesoporous bioactive glasses (MBGs) offer attractive surface features (enhanced surface area and porosity) to use them as fillers in dental adhesives to promote remineralization through ions release. Moreover, the functionalization of pores with antibacterial drugs is a good way to combat secondary caries.

The present work focused on the synthesis and evaluation of novel MBGs suitable to be used as fillers in dental adhesives. The MBGs were prepared in an acetate based sol-gel system with industrially safe and non-toxic precursors. MBGs prepared in large scale (50g) offered enhanced surface characteristics in comparison to small scale (10g) MBGs. The investigation on the influence of network modifiers (CaO:Na_2O) on the surface characteristics of MBGs revealed that the porosity was driven by CaO content in the composition. Notable, very high surface area (535m^2g^{-1}) and pore volume (0.33cm^3g^{-1}) was attained in the MBG with highest CaO content.

Next, the order of precursor addition effect on the surface characteristics of MBGs has been studied. By Keeping the composition fixed and varying the order of precursor addition during sol-gel synthesis doubling of surface area, 1.5 times increase in pore volume and 1.2 times decrease in mean pore size was obtained. The demonstrated method is a simple and straightforward route to improve the porosity and homogeneity of MBGs. Furthermore, modulation of mean pore size for a fixed composition is also useful to tailor the pores of the fillers for drug delivery application.

With regards to bioactivity, the MBG fillers with highest CaO content had increased calcium phosphate precipitate in SBF after 7 days as opposed to MBG with high Na_2O content. Furthermore, all tested samples were non-cytotoxic to Human Gingival Fibroblasts (HGFs) in vitro. Positively, MBGs treated at lower temperature significantly enhanced the metabolic activity of HGFs.

Ball milling was employed to reduce the primary particle size of MBG to less than 3µm. Milling seemingly had an adverse effect on the porosity of the MBG filler. Nevertheless, some porosity remained. The commercial adhesive was mixed with 3, 10, 20 and 30 weight percentage of MBG filler. MBG filled adhesive up to10 weight percent filler content had flowable viscosity suitable for adhesive application.

The developed MBG with high porosity and CaO content appears as a new step in the development of dental adhesives and also other bioactive dental materials.