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ORIGINAL RESEARCH

Physicochemical analysis of MTA Angelus® and Biodentine® conducted with X ray difraction, dispersive energy spectrometry, X ray fluorescence, scanning electron microscope and infra red spectroscopy

Análisis fisicoquímico del MTA Angelus® y Biodentine® mediante difracción de rayos X, espectrometría de energía dispersiva, fluorescencia de rayos X, microscopio electrónico de barrido y espectroscopía de rayos infrarrojos

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ABSTRACT

The aim of the present study was to characterize components of commercial cements used in dentistry MTA Angelus® White (Angelus Lodrina, Parana Brazil) and Biodentine™ (Septodont, Saint-Maur-des Fosses, France). Techniques used for said characterization were Scanning Electron Microscope, X-Ray Diffraction, X Ray Fluorescence, Electron Dispersion Spectrometry, and Infrared Spectroscopy. Both cements were mixed according to manufacturer's instructions. A study of surface texture was conducted with Scanning Electron Microscope (SEM), and X Ray Diffraction (XRD) analysis, and X Ray fluorescence analysis (XRF), an analysis of Dispersive Energy Spectrometry (DES), as well as an Infra Red Spectroscopy (IRS) in order to determine functional groups. **Results:** In XRD analysis, a difference was found: Biodentine exhibited Na_2O and ZrO_2. These elements were absent in MTA. MTA presented Cr_2O_3 and BiO_2 which in turn were absent in Biodentine. EDS analysis revealed that differences were found in the radio-opacifying agent, and that Biodentine presented $CaCl_2$ differing in this from MTA. Statistical analysis conducted revealed statistically significant percentages in contents, even though components were found to be practically the same. SEM analysis revealed marked differences: MTA presented irregular and porous surface whereas Biodentine exhibited irregular and filament form. **Conclusion:** There is a great similarity in the chemical components of MTA Angelus and Biodentine, with the exception of chemical components providing radio-opacity, the size and form of the grain, and, in Biodentine presence of calcium chloride.

RESUMEN

El propósito de este estudio fue caracterizar los componentes de los cementos comerciales para uso en odontología MTA Angelus® Blanco (Angelus, Lodrina, Paraná Brasil) y de Biodentine™ (Septodont, Saint-Maur-des Fosses, Francia) mediante Microscopia Electrónica de Barrido, difracción de rayos X, fluorescencia de rayos X, espectrometría de dispersión de electrones y espectroscopia infrarroja. Los dos cementos se mezclaron según las indicaciones del fabricante. Se les practicó un estudio de textura de superficie mediante el microscopio electrónico de barrido (MEB), un análisis de difracción de rayos X (DRX), un análisis de fluorescencia de rayos X (FRX), un análisis de espectrometría de energía dispersiva (EDS) y un análisis de espectroscopia infrarroja (IR), para determinar los grupos funcionales. **Resultados:** Se presentó una diferencia en el análisis XRD entre Biodentine presentó Na_2O y ZrO_2 mientras que están ausentes en el MTA. El MTA presentó Cr_2O_3 y BiO_2 ausentes en el Biodentine. En el análisis EDS las diferencias fueron en el agente radiopacador y que el Biodentine presentó Cl a diferencia del MTA y en el análisis estadístico realizado a pesar de que prácticamente se presentaron los mismos componentes los porcentajes en los contenidos de éstos fueron estadísticamente significativos. En el análisis de MEB hay una gran diferencia, el MTA presenta una superficie porosa e irregular, el Biodentine una forma fibrilar e irregular. **Conclusión:** Existe una gran similitud en los componentes químicos entre el MTA Angelus y Biodentine con excepción de los componentes químicos para proporcionarles radioopacidad, el tamaño y la forma del grano y en el caso del Biodentine el cloruro de calcio.

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Key words: MTA Angelus®, Biodentine™, DES, SEM, XRD, XRF, IRS.
Palabras clave: MTA Angelus®, Biodentine™, EDS, MEB, DRX, FRX, IR.

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INTRODUCTION

Dental materials have been evolving alongside dentistry due to technological advances, which have assisted these materials to possess better physical, chemical and biological properties.

Retro-filling materials are commonly used in endodontic surgical procedures. An ideal endodontic reparation material should be radio-opaque, biocompatible, with anti-bacterial effect, dimensionally stable, easy to manipulate and not be contaminated or affected by blood. Other desirable characteristics for the selected material would include for it to be osteo-inductor, provide suitable sealing against bacteria and fluids as well as being able to avoid filtrations when placed in humid environment and possessing sufficient resistance to compression and hardness.[1]

Many materials have been used to perform retrograde filling. Among them we can count amalgam, zinc oxide-eugenol, polycarboxylate cements, glass ionomer cements, composite resin, epoxy-resin, gutta-percha and mineral trioxide aggregate (MTA) type cements based on Portland cement.

Main disadvantages of the aforementioned materials include micro-leakage, varied degrees of toxicity, as well as sensitivity to presence of humidity.[2,3] Among these MTA has been recognized as a bioactive material,[4] hard tissue conductor[5] hard tissue inductor as well as biocompatible.[6]

MTA is a material commonly used for retrograde filling procedures, apex formation and perforation repairs, nevertheless its handling is less than ideal due to its long setting time and difficulties in preserving mix consistency.[7]

Calcium silicate cements, especially those derived from Portland cement, such as mineral trioxide aggregate (MTA) and others have been designed and are used in clinical dental applications.

Self-adjusting properties of calcium silicate cements are due to the progressive hydration reaction of orthosilicate ions (SiO_4).

When calcium silicate particles react to water a hydrated calcium silicate nanoporous amorphous gel is formed (HCS gel) in the cement particles, while calcium hydroxide ($Ca(OH)_2$) (portlandite) forms nuclei and grows in available gaps and spaces of the pores. With time, HCS gel polymerizes and hardens, forming thus a solid net which is associated to greater mechanical resistance. HCS gel is soluble in $Ca(OH)_2$,released by the cement surface and increases alkalinity of surrounding environment.[8]

The purpose of the present study was to explore the components of MTA Angelus® White cement (Angelus, Lodrina, Paraná Brazil) and Biodentine™ (Septodont, Saint-Maur-des Fosses, France) by means of X-ray diffraction and electron dispersion spectrometry, X -ray fluorescence, as well as observing the surface with scanning electron microscope and infra red spectroscopy.

MATERIALS AND METHODS

Both cements used for the present project were divided into two groups:

- Group 1 MTA Angelus® White (Angelus, Lodrina, Parana, Brazil).
- Group 2: Biodentine™ (Septodent, Saint-Maur-des-Fosses, France).

One gram of the powder provided by the manufacturer was used for XRD and XRF analyses. For DES, SEM and IRS analyses all products were mixed using powder and liquid provided by the manufacturer. Manufacturer's instructions were strictly followed. One 8 mm diameter x 4 mm thickness sample was manufactured for each group. Five points were randomly taken for the analysis.

X ray diffraction analysis was conducted with a diffractometer Phillips Mod 1130/96 (generator) and pw1050/24 (goniometer) using CuKα at angular intervals ranging from 4° to 70°.

X ray fluorescence analysis (XRF): An X ray fluorescence quantitative chemical analysis was conducted with a Siemens SRS 3000 spectrometer, gauged with Geochemical Reference materials. This analysis was conducted with the sample in dry base, and loss by calcination (LBC) was determined by calcinating 1 g of the sample at 950 °C during one hour.

Dispersive energy spectrometry (DES) and scanning electron microscopy (SEM): Once hardened, the samples were placed on the sample holder with a carbon film to which they adhered. Observations were made with Scanning Electron Microscope (leol model 5900 LV, Tokio, Japan). Used magnifications were 500X, 1000X and 2000X.

For the dispersive energy spectrometry analysis (DES) an elemental chemical analysis was conducted with an Oxford device, ISIS model, with 133 eV resolution, with carbon to uranium element detection.

For the present study amplifications of 500X, 1000X and 2000X were used in all samples at four pre-determined points.

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