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Effect of nanoclay addition on physical, chemical, optical and biological properties of experimental dental resin composites

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ABSTRACT

Objective. To prepare organically modified montmorillonite (OM MMT) and assess mechanical, physical, chemical and biological effects of its introduction into resin-composites.

Methods. Natural MMT clay was modified by a methacrylate functionalized quaternary ammonium intercalating agent. Interlayer distance was measured by X-ray diffraction. Dental composites were then prepared with $x=0, 1, 2.5, 5$ or 7.5 wt.% of OM MMT, $(75-x)$ wt.% of silanated barium glass and 25 wt.% of methacrylate based matrix). Relative weight loss was measured and the effect of the substitution on mechanical properties was studied by dynamic mechanical analysis and hardness tests. Properties of resin composites were evaluated in terms of water sorption, light transmittance, biological tests and by high-performance liquid chromatography (HPLC).

Results. Resin based composites with well-dispersed organically modified MMT were successfully prepared. There were no significant weight loss differences shown by TGA within all samples. The DMA analysis showed that the introduction of clays have a beneficial effect in increasing the storage and elastic modulus of composites. Clay presence was shown to interfere with the blue light transmittance, affecting Vickers hardness and water sorption levels. The amount of released monomers measured from extracts was below expected levels for this type of materials and biological tests show satisfactory cell compatibility.

Significance. This paper reports the successful functionalization of MMT by a methacrylate group and further incorporation in experimental dental composites. Physical and biological results show a potential interest to the application of nanoclays into dental resin composites.

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1. Introduction

Dental caries and its clinical management is still a major public health concern in dentistry all over the world. Recent publications [1] have even reported an increase in global prevalence, resulting in a great demand for direct restorative materials. Mainly because of their aesthetic properties, dental composites are gradually becoming the preferred restorative solution adopted by the dentists. They are typically a mixture of methacrylate resin-based matrix, a photo initiator, and a high percentage of inorganic fillers that confer mechanical, optimal and radiopacity properties.

In spite of many improvements on dental composites since the early 60s, concerns still exist about composites longevity especially when materials are placed in high load bearing sites [2,3]. To overcome this limitation, expectations for innovations lay on the development of composite systems with improved properties such as higher mechanical strength [4–7], fracture resistance [8] and biocompatibility [9,10].

Nanoclay fillers are being commonly added to engineering composites as reinforcing phase to afford great properties enhancements in comparison with conventional micrometric counterparts [11]. Notably, clay-based composites are of great interest for use in food packaging, protective coatings, and other applications where efficient barriers are needed [12] but have not been widely studied in the dental restorative materials field.

These peculiar properties come from the natural clays inner structure composed of stack of 1 nm thick, 100 nm wide layered crystalline aluminosilicate sheets or platelets. Once unstacked and dispersed in a polymeric matrix the sheets can improve mechanical and barrier properties of the resulting nanocomposite [11,13].

Several steps are necessary to achieve clay platelets dispersion. Initially in natural clay, hydrated alkali or earth-alkali cations such as Na^+ or Ca^{2+} are present in interlayer galleries to charge balance the negatively charged sheets [13]. These sheets are linked together by means of weak polar and Van der Waals forces and are not easily exfoliated inside polymeric matrices. One of the first steps to incorporate clays into a polymeric matrix is to increase the inter platelet distance by ionically exchanging the cations by bulkier cationic surfactants such as ammonium salt solutions [11,13]. The increased interlayer distance allows for an easier invasion of the clay galleries by the matrix polymeric chains. Furthermore, the ammonium salts can possess chemical functional groups that improve the clay compatibility with the matrix.

Montmorillonite is one of the most commonly used clay to produce polymer layered silicate nanocomposites and presents general formula $(\text{Al}_2\text{Mg}_3)\text{Si}_4\text{O}_{10}(\text{OH})_2\text{M}^+$ where M^+ is the monovalent cation. Few studies show the effect of clays on dental materials, but some promising yet preliminary results are reported in the current literature. The incorporation of ion exchanged and vinyl modified montmorillonite into dental monomers was successfully achieved even if the authors could not avoid the formation of agglomerates and did not obtain well distributed platelets into the composite bulk [14]. PMMA modified montmorillonite has proven to be an effective reinforcing agent to dental adhesive monomers and results

in significant improvements on material mechanical properties [15]. However, for the production of experimental dental composites, the use of clay treated with ammonium salt did not improve the studied properties of the experimental materials [16]. The step of replacing interlayer cations by organic molecules, producing an organically modified clay material, is not only interesting in terms of compatibilization of clay polarity with that of polymers but also to contribute to expand interlayer distance [11].

Clays are not easily exfoliated into individual layers and the successful exfoliation of layered silicates into a polymeric matrix remains a current scientific and technical challenge in material science [17]. Recent publications on polymer-layered silicate nanocomposites suggest that controlling the exfoliation process is crucial to control the microstructure and the properties of the produced composites [18,19].

One of the most promising reactions to create clay mineral-polymer nanocomposites is the in situ polymerization of the corresponding monomers within the interlayer space. The monomers size allows for easier infiltration in clay interlayers galleries. Interlayer polymerization reactions were studied using 4-vinyl pyridine [20], acrylates [21], acrylonitrile [22], and others. In this study, an aqueous solution of methacrylate-terminated ammonium salt (2-(methacryloyloxy)ethyl trimethylammonium chloride) solution was used to treat natural clay. This agent is expected to organically modify the clay, achieving a notably higher compatibility with the methacrylate monomers present in the matrix. The conditions are such that the polymerization is performed within the interlayer space, contributing to clay exfoliation. New organically modified montmorillonite clays and experimental resin based dental composites were then prepared and characterized. Microstructure of clays by X-ray diffraction (XRD) was investigated and the effect of organically modified clay content on physical, chemical, biological and optical properties of nanocomposites was evaluated. Our hypothesis is that the incorporation of organically modified montmorillonite (OM MMT) improves mechanical, physical, chemical and biological properties of resin-composites. The null hypothesis tested is that there is no difference in mechanical, physical, chemical and biological properties of resin-composites with the incorporation of organically modified montmorillonite (OM MMT).

2. Materials and methods

Organically modified clay was prepared by cation exchange of natural montmorillonite (MMT) clay (Cloisite[®] Na+, BYK Additives Inc.) with 2-(methacryloyloxy)ethyl trimethylammonium chloride solution (TCI Europe). Natural clay was dispersed in water using magnet stirrer at ambient temperature for 30 min and with the application of ultrasound (UP400S, Hielscher) at ambient temperature for another 30 min with 3 mm sonotrode at 100% amplitude. An excess of salt solution was calculated from CEC 0.92 meq/g and added little by little under ultrasound agitation. The resulting treated clay was washed 5 times with distilled water to remove salt excess, dried at 65 °C, ground and sieved below 63 μm.

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