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Research Paper

Resin composite characterizations following a simplified protocol of accelerated aging as a function of the expiration date

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ABSTRACT

This study evaluated the mechanical, thermal, and morphological characteristics of different classifications of dental composites as a function of the material condition (new, aged and expired). Specimens were obtained according to these factors: Composites: Filtek P60, Filtek Z250, Filtek Z350XT, and Filtek Silorane; and Material conditions: new, aged, and expired. The syringe composites underwent an accelerated aging protocol (Arrhenius model). The flexural strength (*FS*) and flexural modulus (*E*) were obtained. The thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were also performed and the glass transition temperature (T_g) and the weight loss calculated. Topographic analysis of the composites was performed under SEM. The material conditions influenced the mechanical properties of the composites. The silorane composite exhibited a characteristic thermal behavior different from that of the methacrylates. In general, the T_g increased after the accelerated aging protocol and decreased for expired ones, compared to the new composites. A significant increase in *FS* of Filtek Z350XT after aging was accompanied by an increase in the T_g. The filler packings were in accordance with the manufacture's information. The topographic aspects of the composites were modified as a function of the material condition. The mechanical properties of the composites following a simplified protocol of accelerated aging varied as a function of the expiration date. The silorane composite presented a characteristic thermal behavior. Although the dental manufacturers may not be able to control variables as storage temperature and transportation conditions, these effects on the composite clinical performance can be minimized if properly considered.

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1. Introduction

The introduction of new or modified dental products on the market requires the assurance that these materials can be stored for an extended period without any decrease in their performance that may affect safety and efficacy when these products are applied (Hemmerich, 1998). Shelf life is defined as the term or period during which a commodity remains suitable for the intended used (Donohue and Apostolou, 1990). On the other hand, an expiration date is the termination of shelf life, after which a percentage of the product, i.e., medical devices, may no longer function as originally intended. In order to determine whether a certain product requires a shelf life and assign an expiration date, there are a number of different parameters that must be considered (Gillen et al., 1993). In this way, this product must be analyzed to determine if it is susceptible to degradation that would lead to functional failure and the level of risk that the failure would present (Donohue and Apostolou, 1990).

Stability has been defined as the extent at which a product retains, within specified limits, and throughout its period of storage and use, i.e., its shelf life, the same properties and characteristics that it possessed at the time of manufacture (Bajaj et al., 2012). In this way, sets of criteria for acceptable levels of stability for drug products have been proposed: chemical (i.e., degradation, manufacturing), physical (physical characteristics, i.e., viscosity, elasticity, tensile strength), microbiological (i.e., sterility, integrity), therapeutic, and toxicological (i.e., biocompatibility) (USP, 1990). Even though these criteria are specifically applied to evaluate the stability of drug products, it may be useful as a starting point to establish a set of criteria to evaluate the stability of dental products (Clark, 1991).

Accelerated aging protocols, in which certain products are subjected for a much shorter time to stresses that are more severe or more frequently applied than normal environmental or operational stresses, are generally applied to new medical devices to provide experimental data in order to guarantee their performance and the shelf-life claims (Hemmerich, 1998; Reich et al., 1988). Although full-period, ambient-aged specimens usually do not exist for new or modified products, it has been claimed that accelerated studies may not be appropriate for new products as there is no sufficient historical data available. In spite of these claims, these protocols are acceptable for some products to support tentative dates and storage conditions (i.e., at high temperature and humidity) (USP, 1990). Accelerated studies, combined with basic stability information on the components (functional chemical groups organized in diverse ways—crystalline or amorphous—along with additives such as antioxidants, inorganic fillers, plasticizers, colorants, and processing aids (Hemmerich, 1998), may be used to support tentative expiration dates (Clark, 1991). It is the sum of these variations combined with the variations in product use and storage environment that determines the degradation chemistry (Hemmerich, 1998). A tentative expiration date beyond a date supported by actual shelf life studies can somehow be projected according to these data.

There are numerous variables that affect the shelf life of a medical device. While developing these procedures for determining a shelf life of a device, the stability criteria and the product specific variables should be assessed (Clark, 1991). Although manufacturers may not be able to control all of the variables, their effect on device performance can be minimized if properly considered. These variables include the storage and the transportation conditions that can have an effect on the device in a manner which adversely affects device safety or performance (Kommanaboyina and Rhodes, 1999). Temperature variations, relative humidity, ventilation, visible light and other types of radiation are important factors to be considered (USP, 1990), especially when a dental product is shipped between various climatic zones (Lucas et al., 2004). On the other hand, the manufacturers determine the expiration date of the dental products consistent within known performance specifications and may not guarantee their performance beyond this time period (Reich et al., 1988). Although certain composite properties modified over a few years may not be necessarily clinically noticeable, this may impact the longevity of the bonded restoration (Hondrum, 1999).

This study evaluated the mechanical, thermal, and morphological characteristics of different classifications of composites indicated for posterior application as a function of the material condition (new, aged and expired). The following research hypotheses were tested: (I) the material conditions evaluated can influence the mechanical properties of the composites tested; (II) the thermal parameters of the aged and expired composites will be modified in comparison to that of new composites; (III) the morphological aspects of the composites will be modified as a function of the material condition.

2. Materials and methods

2.1. Experimental design

In this in vitro study, thermal, mechanical and morphological characterizations were performed according to these factors: (1) composites (3 M ESPE) at four levels: Filtek P60, Filtek Z250, Filtek Z350XT, and Filtek Silorane; (2) material conditions at three levels: control (new composites), expired composites, and aged composites; (3) energy dose at two levels: 24 J/cm^2 and 48 J/cm^2. The characteristics of the resin composites selected are described in the Table 1. The product among the combinations of the factors under study obtained twelve groups.

2.2. Accelerated aging protocol

The composites underwent a simplified protocol for accelerated aging, according to the Arrhenius model. The composite syringes were stored at 37 °C in an oven for 12 weeks. According to Clark (Clark, 1991) the accelerated aging protocol can be calculated by means of a mathematical formula, as follows:

$$r = Q_{10}^{((RT-ET)/10)} \quad (1)$$

where r is the accelerated aging rate; RT the room temperature (22 °C); ET the elevated temperature (37 °C) and Q_{10} the reaction rate coefficient (2).