DENTAL MATERIALS 31 (2015) 855–863

Available online at www.sciencedirect.com

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A systematic approach to standardize artificial aging of resin composite cements

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ARTICLE INFO

Article history:
Received 1 February 2015
Received in revised form
20 March 2015
Accepted 27 April 2015

Keywords:
Resin composite cement
Compressive strength
Indirect tensile strength
Aging
Thermal cycling
International standardization

ABSTRACT

Objective. The aim of the investigation was to contribute to the ongoing discussion at the international standardization committee on how to artificially age dental resin composite cements.

Methods. Indirect tensile strength ($n = 30$) of a dual-cured resin composite cement (Panavia F2.0) was measured to evaluate the effect of water storage at 37 °C or thermal cycling (5 °C/55 °C/1 min) for up to 64 days. The influence of water temperature (5–65 °C) after 16 days and the effect of 1 day water storage at 37 °C prior to aging were assessed. Storage in air at 37 °C served as control.

Results. Thermal cycling affected the indirect tensile strength most, followed by water storage at 55 °C, whereas water storage at 37 °C had only little influence. Major deterioration occurred before day 4 (≈6000 cycles). A 1-day pre-treatment by water storage at 37 °C prior to thermal cycling attenuated the effect of aging.

Significance. For the material investigated, thermal cycling for 4 days is the most efficient aging procedure. A 1-day water storage at 37 °C prior to thermal cycling is recommended to allow complete polymerization. A 4-day water storage at 55 °C may be considered as a viable alternative to thermal cycling.

1. Introduction

With the increasing number of ceramic restorations in dentistry, the use of adhesive resin composite cements has steadily increased. Compared with conventional cements based on acid–base reactions, they have better mechanical properties, higher bond strength, reduced solubility and improved esthetics [1,2]. In general, these materials consist of three components: the polymer matrix, the fillers and the silanes as binder between the organic and the inorganic phase [3,4]. The properties of resin composite cements such as elasticity, plasticity, hardness, strength and thermal as well as chemical stability are determined by the properties of

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http://dx.doi.org/10.1016/j.dental.2015.04.015

these single components and the respective microstructure [4–6].

Polymerization is catalyzed either by autocatalysis ("self-curing") or photo-initiation ("light-curing"). The autocatalytic polymerization has the disadvantage that the reaction starts immediately after mixing the catalyst (initiator) with the polymerizable component. Thus, the processing time is limited. In contrast, the photo-initiation allows determining the start of the polymerization. However, areas that are not reached by the light will not polymerize adequately. To avoid the disadvantages of each, cements were developed comprising both initiator systems ("dual-curing"). The idea was to provide a material of which the processing time is adapted to the clinical requirements and, independent of the influence of light, a high degree of conversion will be achieved [3,7].

The conversion level is an important factor for the mechanical strength of luting cements because composites with a high degree of conversion also have better mechanical properties [3,7]. Most cements show a higher degree of conversion by dual-curing compared with self-curing alone [8–10]. Additional catalysts are beneficial to improve polymerization. For instance, the application of ED Primer (Kuraray-Noritake, Kurashiki, Japan) increases the degree of conversion of Panavia F2.0 (Kuraray-Noritake) by 15% after self-curing and by 24% after dual-curing [11,12]. The conversion degree in self-curing is influenced not only by the concentration of monomer and catalyst, but also by the ambient temperature [13–15]. An increase in temperature from 3 to 60 °C resulted in an increase in the degree of conversion of 36% [16].

Cements in an aqueous medium such as saliva are exposed to a long-term aging process, which might significantly reduce the mechanical properties [17]. The effects are wide-ranging, but typically include the leaching of unreacted components and the degradation of the polymer network [17,18]. At present, no standardized procedure for artificial aging of resin composite cements is available. Thermal cycling simulates the effect of varying temperatures in the oral cavity, which might range from 0 °C (melting ice) to 60 °C (hot beverages). For that reason, thermal cycling is usually performed between 5 and 55 °C with cycle times of 1 min [19–21]. The suggested duration of thermal cycling differs from 3000 to 100,000 cycles [22–26]. It is proposed that 10,000 cycles might represent 1 year of service [27,28]. After the placement of the restoration, however, the cement is setting at 37 °C and polymerizes for another 24 h [29]. During this time, thermal stress is rare. Thus, to mimic the clinical situation, the effect of a pre-treatment of the cement at 37 °C prior to artificial aging should be analyzed.

Composite resin cements are brittle materials and therefore tolerant to compressive stress but susceptible to tensile loading [30]. Hence, a three-point bending test for the characterization of the strength of luting materials is recommended in the respective standard ISO 4049. A 1-day water storage at 37 °C prior to the test is specified to allow a complete polymerization. However, the preparation of specimens for bending tests is demanding and time-consuming. Especially during development of a new product and with the increasing number of products available, the quantity of such tests will increase. Therefore, simplification of these tests would be attractive. Compressive or indirect tensile strength tests provide reliable information on the mechanical strength of the investigated material with the advantage of easy handling [31,32]. To the knowledge of the authors, an impact of aging on indirect tensile strength or compressive strength of resin composite cements is not yet investigated and therefore worth to be analyzed. Further, water storage instead of thermal cycling could be an option to economize the test procedure, because thermal cycling requires a special device with a limited capacity.

Table 1 – Composite resin cement and primer used in the study (material compositions according to the manufacturer's product specification).

Material	Composition
Panavia F 2.0	*Base paste*: hydrophobic aromatic dimethacrylate, hydrophilic and hydrophobic aliphatic dimethacrylate, sodium aromatic sulphinate, silanated barium glass filler, surface-treated sodium fluoride, catalysts, accelerators, pigments *Catalyst paste*: 10-methacryloyloxydecyl dihydrogen phosphate (MDP), hydrophobic aromatic dimethacrylate, hydrophilic and hydrophobic aliphatic dimethacrylate, silanated silica filler and colloidal silica, dl-camphorquinone, catalysts, initiators
ED Primer	*Liquid A*: 2-hydroxyethyl methacrylate (HEMA), MDP, N-methacryloyl-5-aminosalicylic acid (5-NMSA), water, accelerators *Liquid B*: N-methacryloyl-5-aminosalicylic acid (5-NMSA), catalysts, accelerators, water

Against that background, the appropriate international standardizing committee (ISO/TC 106/SC 1/WG 9) is currently in the process of evaluating an adequate test design for assessing the effect of aging on the mechanical strength of resin composite cements. The aim of the present study was to contribute to the discussion by a systematic analysis of the effect of different aging procedures on composite resin cements by compressive and indirect tensile strength tests.

2. Materials and methods

In the present study, Panavia F2.0 (Kuraray-Noritake) was selected as test material (Table 1). The specimens were produced in a customized Teflon mold 3 mm in height with five cylindrical holes each 3 mm in diameter. Depending on the curing mode, an opaque Teflon plate (self-curing) or a transparent glass plate covered with a Mylar strip (Hawe Transparent Strips, KerrHawe, Bioggio, Switzerland) (dual-curing) was fixed at the bottom of the Teflon plate. All surfaces of the mold coming in contact with the specimens were first coated with ED Primer (Kuraray-Noritake). One drop each of primer liquids A and B was mixed for 20 s, applied to the mold and gently dried for 5 s with air as recommended by the manufacturer. Base and catalyst pastes of the cement were mixed according to the manufacturer's instructions in a 1:1 ratio for 20 s with a plastic spatula on a mixing tray and at the latest 2 min after start of mixing filled into the molds with slight excess. For *self-curing*, the top of the Teflon mold was also covered with an opaque Teflon plate and left for 20 min under a load of 1 kg. For "dual-curing," the top of the Teflon mold was