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Mechanical viscoelastic behavior of dental adhesives

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ABSTRACT

Objectives. The purpose of the study was to evaluate the mechanical properties of dental adhesive materials at different testing temperatures after dry and wet storage.
Methods. Specimens ($d = 1$ mm, $l = 18$ mm) from six materials were tested: Silorane Adhesive System (SL), Heliobond (HE), One-Step Plus (OS), Optibond Solo Plus (OP), cmf Adhesive System (CF) and Protobond (PR). Static and creep testing was performed by applying a constant torque below the proportional limit of the materials, while dynamic testing consisted of dynamic torsional loading. Experiments were performed after 24 h of dry and wet storage under temperatures from 21 °C to 50 °C and various viscoelastic parameters were calculated.
Results. Shear modulus ranged from 0.19 to 1.99 GPa, while flexural modulus from 0.67 to 5.69 GPa. Most of the materials were affected by the presence of water and increase of temperature. OP showed the highest recovery after creep, while SL exhibited the highest permanent deformation.
Significance. Contact with water after polymerization and increase of temperature resulted in a decline of the mechanical properties, especially for the HEMA-containing adhesives.

1. Introduction

The need for restorative materials that could replace amalgam led to the introduction of composite resins that are now widely used by dentists. One of the most important steps that consequently led to the popularity of these materials was the breakthrough in adhesive technology. The finding that acid etching enamel led to higher bond strength between resin and enamel resulted in further research and the understanding of hybrid layer and dentin etching. Dental adhesives have evolved since then and many commercial products are available with different compositions and different approach to the way they deal with tooth tissue [1]. Their aim is to achieve strong bonding between dental tissues and restorative materials which will provide clinical longevity to the restoration.

Despite the advancements in bonding, the bonded interface of composite restorations is still the weakest area of the restoration and the main reason for failures such as marginal discoloration and poor marginal adaptation which may later lead to loss of retention [2]. This is apparent in the fact that various strategies and types of adhesives are used in order to achieve the most satisfactory bonding performance. Without regard to the steps required, the approaches contemporary adhesives use are two: etch-and-rinse and self-etch. In the former approach the tooth substrate is first etched and rinsed

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(conditioning stage), followed by a priming stage and the application of the bonding resin [3]. On the other hand, self-etch approach does not require a separate etch-and-rinse stage and uses acidic monomers that simultaneously condition and prime dentin and enamel [4].

The resulting composite restorations are a complex system that consists of different substrates and interfaces. The long-term performance of these restorations is the result of the behavior of the various components and their ability to withstand stress and deformation. Testing separately the various components of the composite restoration can help to identify which is the least stable under different conditions [5]. While the viscoelastic behavior of the restorative composite resins is often studied, adhesive resins are not commonly examined regarding their mechanical behavior. This phenomenon may be attributed to the fact that research is focused mainly on their bonding properties and that some techniques cannot be applied directly to materials that are used in thin layers [6]. The technique being used in the current study was developed in order to test small specimens appropriate for dental materials, as most instruments used for this type of testing are optimized for much larger specimens. It has been previously used for the determination of the mechanical properties of other dental materials as composite resins [7], resin cements [8] impression materials [9] and fiber-reinforced posts [10]. The advantages of this method is that it can be used both for static and dynamic testing under different conditions that can be controlled by the operator and can provide measurements for various viscoelastic parameters.

The aim of this study was to evaluate the mechanical properties of various commercial dental adhesives under different conditions both under static and dynamic testing. The null hypothesis was that the materials will not present differences in their properties and will not be affected by storage and testing conditions.

2. Materials and methods

Six commercially available materials were tested and are shown in Table 1. In the case the material consisted of more than one component only the bonding component was tested. Cylindrical specimens (diameter $d = 1$ mm, length $L = 18$ mm) from each material were made with the use of glass capillary tubes. Each material was poured to a plastic funnel to be gently dried and then let to flow into the transparent glass tube. Due to the specimens' length, each one was light-cured in consecutive sections along its axis in order to achieve thorough polymerization and for a time according to the manufacturers' instructions (600 mW/cm^2, Coltulux 4 light, Coltene Whaledent, Altstätten Switzerland).

The materials were tested under four different conditions ($n = 4$ for each condition):

i) Tested dry at 21 °C, after 24 h of storage in room temperature of 21 °C.
ii) Tested wet at 21 °C, after 24 h of storage in distilled water at 21 °C.
iii) Tested wet at 37 °C, after 24 h of storage in distilled water at 37 °C.
iv) Tested wet at 50 °C, after 24 h of storage in distilled water at 50 °C.

The specimens were mounted using a jig for centering between a Plexiglas disc (0.5 mm thick) and a rod. The experiments were performed using an apparatus (Fig. 1), previously described by Lakes [11], that is capable of testing cylindrical

Table 1 – The materials used in the study.

Material	Composition	Type
Silorane adhesive system (SL) 3M ESPE - Seefeld, Germany	Hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, initiators, stabilizers Fillers: Silane treated silica	Two-step self-etch: self-etch primer and bond
Heliobond (HE) Ivoclar Vivadent - Liechtenstein	Bis-GMA, TEGDMA, initiators, stabilizers	Unfilled enamel bonding agent
One-Step Plus (OS) Bisco - Schaumburg, IL, USA	Bis-GMA, HEMA, BPDM, acetone Fillers: 8.5% wt. glass ionomer	Two-step etch and rinse: etching gel and bond
OptiBond Solo Plus (OP) Kerr - Orange, CA, USA	Bis-GMA, HEMA, GDM, GPDM, ethanol Fillers: 15% wt. 0.4 μm barium glass, fumed silica, sodium hexafluorosilicate	Two-step etch-and-rinse: etching gel and bond
cmf adhesive system (CF) Saremco - St Gallen, Switzerland	Bis-GMA, Bis-EMA Fillers: silanized barium glass	Three-step etch-and-rinse: etching gel, primer and bond
Protobond (PR) Dental Co-operative - Thessaloniki, Greece	Bis-GMA, Bis-EMA, HPMA, initiators, stabilizers	Two-step etch-and-rinse: etching gel and bond

Bis-GMA: bisphenol A glycidyl methacrylate, TEGDMA: triethylene glycol dimethacrylate, HEMA: 2-Hydroxyethylmethacrylate, BPDM: biphenyl dimethacrylate, GDM: glycerol dimethacrylate, GPDM: glycerol phosphate dimethacrylate, Bis-EMA: bisphenol A ethoxylate dimethacrylate, HPMA: hydroxypropyl methacrylate.