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Failure mechanisms in denture adhesives

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ABSTRACT

Objective. The mechanical properties of bio adhesives in oral care application are expected to be critical in defining the stability and release of devices such as dentures from the oral tissue. A multiscale experimental mechanical approach is used to evaluate the performance of denture adhesive materials.

Methods. The inherent mechanical behavior of denture fixatives was examined by separating adhesive material from a representative polymethyl methacrylate (PMMA) surface using atomic force microscopy (AFM) approaches and compared to macroscopic mechanical testing.

Results. Failure of denture adhesive material was found to be critically dependent on the formation of fibrillar structures within the adhesive. Small scale mechanical testing provided evidence for the mechanical properties of the fibrillar structures formed within the adhesive in macroscopic mechanical testing and indicated the importance of the forces required to fail the adhesive at these small length scales in controlling both the maximum forces sustained by the bulk material as well as the ease of separating the adhesive from PMMA surfaces.

Significance. Our results are important in defining the performance of denture fixative materials and their control of adhesive behavior, allowing the potential to tune properties required in the adhesion and removal of dentures.

1. Introduction

Complex processes regulate the adhesion of biomaterials to tissues and other interfaces [1–5], with the magnitude of such interactions defining the overall performance of implants. In particular, understanding the mechanical properties of the adhesive at the interface with the device and tissue are required for evaluation of resultant adhesive performance [5–7]. Adhesives for dentures are particularly demanding and need to provide fixation of the denture within the aggressive environment of the oral cavity but allow relatively effective removal on demand [8–13]. The adhesion of dentures is almost contradictory as both high adhesion for fixing and low adhesion for ease of removal are required. The potential sensitivity of adhesion in controlling denture fixing and removal motivates the need for techniques that are able to comprehensively evaluate the adhesion process. The relationship

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between molecular interactions at interfaces and in the bulk of the adhesive involves evaluation of stress transfer and failure mechanisms that are currently poorly defined. Methods that quantify structure–property relationships controlling the behavior of such interfaces are important in understanding and designing implants and adhesives in the biomedical field, including for oral care applications. From fundamental considerations, electrostatic interactions and hydrogen bonding are known to significantly contribute to the bulk mechanical and rheological properties of biomaterials used for dental adhesive applications [14]. However, the role of these interactions, together with other hydrophobic interactions occurring at the surface of implants and denture, on the failure of adhesive remains unclear. Chemical design of biomaterials is therefore important in controlling the failure of the adhesive, specifically at interfaces or within the bulk, and enables tailoring of the mechanical properties of the adhesive to function. The location of failure occurs either at an interface (adhesive failure) or in the bulk of the adhesive (cohesive failure) and has been shown to be particularly important in defining resultant adhesive performance [15,16]. Suitable experimental techniques are required to both measure the mechanical properties of the dental adhesive directly and relate to the chemistry of the adhesive. Microscale mechanical testing using atomic force microscopy (AFM) is often employed to understand these mechanical properties directly and, as the size considered is relatively small, geometric considerations that dominate at larger lengths can be ignored so that the inherent material chemistry is probed [17–21]. Extension of small-scale mechanical testing has incorporated in situ imaging using scanning electron microscopy (SEM) that allows correlation between the mechanical response of a biomaterial and the observed deformation or failure event, the latter being important in defining either adhesive or cohesive failure [17–20]. The powerful combination of small-scale mechanical testing and in situ imaging is therefore applicable to denture adhesives to provide quantitative evidence of the influence of chemistry on failure mechanisms. In this work, the mechanical properties of a dental adhesive in contact with poly(methyl methacrylate) (PMMA) dentures was examined. Our approaches aim to correlate the larger macroscopic length scale to more fundamental microscale behavior for comprehensive structure–property relationships.

2. Materials and methods

2.1. Overview

Commercially available dental adhesives (GSK, UK), Poligrip®, Ultra Wernets®, Denture Fixative Powder (PDFP) and Poligrip®, Ooze-Control Tip® Denture Adhesive Cream (PDAC), were used in this study. PDFP is composed of poly(methylvinylether/maleic acid) sodium–calcium mixed partial salt, cellulose gum and aroma while PDAC is composed of poly(methylvinylether/maleic acid) sodium–magnesium–zinc mixed partial salt, petrolatum, cellulose gum, mineral oil, silica, poly(methylvinylether/maleic acid), flavor, Red 30 aluminum lake and Red 7 calcium lake. PDAC contains more hydrophobic compounds such as hydrocarbon vehicles (mineral oil and petrolautum), in addition to MVE/MA copolymer. These compounds may affect the hydration of the polymers and gel formation resulting in different adhesion behavior. Both materials were applied as adhesives to investigate their adhesion behavior with a PMMA substrate representing a standard denture material. Both PDFP and PDAC were wet by mixing with distilled water at the ratio of 1:1 in a petri dish before mechanical testing. This approach was considered to represent the hydration state of the adhesives in typical usage conditions in the oral cavity for fixing dentures [22]. Adhesion behavior of PDFP and PDAC with PMMA was investigated at both the macroscale and microscale to fully characterize their adhesion mechanics, as shown schematically in Fig. 1.

2.2. Macroscopic testing

Macroscopic testing was performed by detaching two adhesive-bonded PMMA plates and examining their adhesion properties. Commercial PMMA plates were cut using a circular saw (Struers, Germany) into dimensions of 3 mm × 6 mm × 15 mm with the cross section area of 3 mm × 6 mm for adhesive attachment. Two PMMA plates were bonded with a small amount (weighted approximately 0.15 ± 0.5 g) of wet adhesive sufficient to fully cover the cross section area and held together by hand for 1 min before mounting the two adhesive-bonded PMMA plates on a commercial microtester (Deben, 200 N tensile stage, U.K., as shown in Fig. 1. The microtester was mounted onto a scanning electron microscope (SEM) sample stage within the SEM chamber (Quanta 3D FEG, FEI, EU/USA) so that mechanical testing was observed using the SEM. The opposite ends of each PMMA plate were clamped tightly by the sample grip of the microtester, leaving the two adhesive-bonded cross section surfaces in the middle of the gauge. Initial distance between the two sample grips was calibrated to 10.09 mm. Uniaxial tensile test was performed by translating one of the grips away from the other at a constant rate of 0.5 mm min^{-1}, causing the two adhesive-bonded cross section surfaces to detach. The force and extension applied to the sample was recorded using the microtester while SEM allowed physical deformation to be related to the mechanical information.

2.3. Nanoscale AFM testing

2.3.1. Sample preparation

Adhesion behavior between PDFP/PDAC and PMMA was further investigated at smaller length scales to evaluate the relationship between mechanical properties and interfacial chemistry in a geometrically simple setup. Spherical microscale PMMA-coated silica beads were used to study the PDFP/PDAC-PMMA adhesion at the microscale. These microscale experiments were important and allowed comparison with macroscopic testing that incorporates chemistry as well as potentially larger structural features, such as voids, that may dominate adhesion behavior. The microbeads were prepared by coating commercial 3.43 μm diameter silica beads (Bangs Laboratories Inc., USA) with PMMA polymer brushes using protocols adapted from the literature [23,24]. 1 ml of toluene kept under nitrogen was added to 50 mg silica beads and sonicated for 10 min until the suspension was cloudy. The