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Review Article

Chemical analyses in dental adhesive technology

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Received 19 December 2011; received in revised form 25 March 2012; accepted 26 March 2012

KEYWORDS

Glass-ionomer;
Self-etch;
Polyalkenoic acid;
Functional monomer;
XPS;
XRD

Summary Adhesive technology has evolved rapidly since it was introduced more than fifty years ago. Today, decayed or fractured teeth can be reconstructed minimally invasively and nearly invisibly using adhesive technology. However, the clinical longevity is sometimes still too short. Although water sorption is thought to be the main destabilizer of the biomaterial–tooth bond, the actual interfacial degradation mechanisms are far from understood. The fundamental mechanism of bonding to enamel and dentin is essentially based on an exchange process, in which minerals removed from the dental hard tissues are replaced by resin monomers that upon polymerization become micro-mechanically interlocked in the created porosities. In addition to micro-mechanical interlocking through hybridization, the potential benefit of additional chemical interaction between functional monomers/polymers and tooth substrate components has recently regained attention. In this review paper, we focus on how chemical interaction at the biomaterial–hard tissue interface can improve bond durability.

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1. Introduction

The use of dental amalgam to restore teeth has been forbidden in Norway since 2008 [1], and in Sweden since June 1 2009 [2], mainly because of environmental issues and potential health risks related to its mercury content. Other countries may soon follow [3], and thus the use of tooth-colored materials to directly restore teeth is on the rise. Although decayed/fractured teeth can be reconstructed minimally invasively and nearly invisibly using contemporary adhesive technology, the clinical longevity of direct filling restorations is sometimes still too short [4,5].

In today's dental practice, tooth reconstruction can be performed using dental adhesive technology following either an "etch-and-rinse," "self-etch" or "glass-ionomer" approach [6]. Although the three adhesive approaches are totally different and are achieved through different bonding mechanisms, the success of each approach depends to a large extent on the properties of the resultant biomaterial–tooth tissue interface [7–9]. In other words, the longevity of the adhesive tooth restorations is highly dependent on the quality of the formed hybrid layer. Theoretically, a better hybrid-layer quality can be achieved through a more intense, chemical interaction of the adhesive materials with the different tooth-tissue components available at the interface.

Bonding to tooth tissue occurs mainly by micro-mechanical interlocking, through the formation of a hybrid layer or hybridization. Therefore, adhesive–hard tissue interfaces (hybrid layers) have been thoroughly studied ultra-morphologically using diverse techniques such as scanning electron microscopy (SEM) [10–13], transmission electron microscopy (TEM) [14–19], confocal microscopy [20,21] and even atomic force microscopy [7,22]; however, the complex molecular interactions at the interface have hardly been investigated and are far from understood. This also leads to a delay in the development of theoretically designed materials with long-lasting adhesive potential.

In this paper, we aimed to provide additional insight into the hybridization mechanisms at tooth–biomaterial interfaces, particularly those involving chemical analytical techniques.

2. Glass-ionomers

2.1. General characteristics of glass-ionomers

Glass-ionomers or glass-polyalkenoate cements are known to possess an auto-adhesive capability without requiring any kind of surface pre-treatment. Glass-polyalkenoate cements are typically separate powder and liquid formulations, which harden after mixing through an acid–base reaction between an ion-leachable fluoro-alumino-silicate glass and an aqueous polyalkenoic acid. These self-adhering cements offer the additional important clinical advantage of releasing fluoride

into the adjacent tooth structure, and thus have an inherent cariostatic potential [23]. In an attempt to combine the advantageous properties of glass-polyalkenoate cements and resin composites, a hybrid biomaterial has been introduced in which water-soluble polymerizable monomers were added to the original formulation of conventional glass-polyalkenoate cements. These resin-modified glass-polyalkenoate cements have been reported to possess improved properties and to adhere more strongly to hard tissue [24].

2.2. Analysis of chemical bonding of glass-ionomers to apatitic substrates

Although the unique property of self-adhesiveness of polyalkenoic acid-based materials was demonstrated *in vitro* and clinically many years ago [25,26], the inherent mechanism of the postulated chemical bonding was not fully demonstrated for many years. Amongst several chemical analytical tools, infra-red (IR) spectroscopy has most frequently been used in an attempt to demonstrate the chemical bonding process of glass-polyalkenoate cements [27–29]. However, IR cannot reveal indisputable evidence of chemical bonding. While the reaction of carboxyl groups with calcium can be detected using IR, it is not possible to distinguish between carboxyl groups of polyalkenoic acid that have chemically interacted with calcium at the hydroxyapatite (HAp) interface and those that merely participated in gelation through a reaction with calcium extracted from apatite. To detect true chemical bonding at the interface, chemical information must be gathered exclusively from the bonded layer within a few nm at the interface. Indeed, one of the most difficult problems in material science is to study the chemistry at interfaces.

X-ray photoelectron spectroscopy (XPS) is a highly selective and specific method of surface analysis [30]. The method allows the upper 1–10 atomic layers (0.5–5 nm) to be investigated with a detection limit of 0.1–1 at%. However, XPS is only capable of acquiring detailed chemical information of the interface between the two materials at an atomic scale under the condition that only an ultrathin film of the molecule with chemical bonding potential is present on top of the substrate (Fig. 1). Monolayers can be formed through the well-established Langmuir–Blodgett (LB) technique involving a controlled transfer of molecules from an air–water interface to a solid substrate [31]. However, not all molecules can be processed using this technique [32], and it is unsuitable for use with these polyalkenoic acids due to their high water solubility. Hence, instead of the controlled application of a monolayer on the substrate, it has proven necessary to remove any surplus poly-acid that did not directly participate in bonding to the substrate, followed by gathering of the chemical information of the surface treated with XPS [33,34].

XPS wide-scan spectra of untreated enamel and enamel treated with polyalkenoic acid are shown in Fig. 2. Although a C 1s peak was already present at the untreated enamel,

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