Journal of Dentistry (2005) **33**, 731–739

ELSEVIER

Journal of Dentistry

www.intl.elsevierhealth.com/journals/jden

SEM-EDX study of prepared human dentine surfaces exposed to gingival retraction fluids

Olalekan A. Ayo-Yusuf[a,*], Cornel H. Driessen[b], Andre J. Botha[c]

[a]*Department of Community Dentistry, School of Dentistry, Room 6.43-2, Oral and Dental Hospital, University of Pretoria, P.O. box 1266, Pretoria 0001, South Africa*
[b]*Department of Restorative Dentistry, School of Dentistry, University of Pretoria, Pretoria, South Africa*
[c]*Laboratory for Microscopy and Microanalysis, University of Pretoria, Pretoria 0002, South Africa*

Received 4 November 2004; received in revised form 1 February 2005; accepted 3 February 2005

KEYWORDS
Gingival retraction fluids;
Impression;
Dentine;
Smear layer;
Acid etching;
Permeability;
Bonding

Summary *Objectives*: To evaluate the effects of gingival retraction fluids (GRF) on prepared dentine surfaces, and to test the null-hypothesis that prior exposure of dentine surfaces for prolonged period to any of the fluids evaluated does not influence acid-etching of the exposed surfaces.
Methods: The investigation was carried out using SEM and energy-dispersive X-ray analysis (EDX). The GRF studied were Hemodent™ (HMDT), Astringedent® (AST) and Ultradent® buffered 25% Aluminium Chloride (ULTB). Longitudinal sections of 220-grit ground dentine surfaces were exposed to GRF for 0.5-, 1-, 2- and 5-min ($n=4$ each). Another group of samples was produced by 20 s application of 35% phosphoric acid (PA) on GRF-pretreated dentine. Control samples were not exposed to GRF. Differences in etching effect—a function of the Ca-contents detected by EDX, were analysed using Friedman's and Wilcoxon's rank test ($P<0.05$).
Results: The SEM demonstrated the presence of a relatively non-porous amorphous dentine matrix, but many of the dentine tubule orifices remained occluded. Granular precipitates, which remained even after acid-etching, were noted on surfaces exposed to Hemodent™ for 5 min. Characteristic crystal growth was observed on surfaces exposed to Astringedent® for 1- or 2-min prior to acid-etching. The EDX data demonstrated that there were differences in resulting Ca-content; ULTB > AST > HMDT > ULTB + PA > HMDT + PA > AST + PA > PA, but AST and HMDT were not significantly different.
Conclusions: The exposure of dentine prepared surfaces to these three GRF altered its morphology and reduced the dentine's susceptibility to acid-etching, thus the null-hypothesis is rejected.

Introduction

The application of retraction cord saturated with gingival retraction fluid before impression-taking is

* Corresponding author. Tel.: +27 12 3192514/83 4421970; fax: +27 12 3237616.
E-mail address: lekan.ayoyusuf@up.ac.za (O.A. Ayo-Yusuf).

0300-5712/$ - see front matter © 2005 Elsevier Ltd. All rights reserved.
doi:10.1016/j.jdent.2005.02.002

a routine clinical procedure, particularly when the finish-lines extend intrasulcularly.[1] Occasionally, gingival retraction is required in order to permit the completion of tooth preparation or to allow cementation of laboratory-manufactured restorations. Recent emphasis on dentine bonding, particularly in cervical restorations, has drawn increased attention to how critical gingival retraction is.

Ample information is available on the local effects of gingival retraction fluids on surrounding soft tissue, but little is known about the effects of these fluids on adjoining tooth substrate. According to Land et al.,[2] gingival retraction fluids may inadvertently contaminate prepared dentine surfaces. The duration of contact in a clinical situation varies, probably depending on the dentist's preference and the specific treatment to be provided. Intrasulcular placement for 1–10 min and longer has been advocated.[3] Recent studies have shown that most of the commonly used fluids are acidic, with pH values ranging from 0.8 to 3.[2,4] In an SEM study, Land et al.[2] reported that the prepared dentine surfaces appeared etched to an advanced stage after a 5-min exposure to 15.5% ferric sulfate (Astringedent®). Bertolotti[5] suggested that Astringedent® may be responsible for certain clinical cases of debonding of bonded restorations, but offered to direct physical evidence.

Many current approaches to dentine bonding rely on acidic treatment of the dentine surfaces to remove or alter the smear layer and establish a microporous surface.[6] 35% phosphoric acid, a widely used acid etchant, is capable of removing the smear layer completely and can enlarge the dentine tubules. Bonding of resinous materials to acid-etched dentine has been explained in terms of resin tag formation within dentine tubules,[7] and by the formation of a hybrid layer at superficial demineralised dentine, where hydrophilic monomers penetrate the microporous intertubular dentine before polymerization.[8]

It is often necessary to use several methods to provide a comprehensive understanding of the dentine surface, because the information provided by the different methods is often complementary. According to Gwinnett,[9] the introduction of SEM and EDX marked a technological advance in instrumentation. He also reported that the relatively high resolution of microscopic detail coupled with a large depth of field makes SEM and EDX ideally suitable to detailing surface morphology and identifying surface composition.

The objectives of this investigation were (1) to evaluate the effects of three gingival retraction fluids, namely Hemodent™ (HMDT), Astringedent® (AST), and Ultradent® Buffered 25% Aluminium Chloride (ULTB), on prepared dentine surfaces after various exposure times, and (2) to test the null hypothesis that the prior exposure of prepared dentine surfaces for prolonged period to any of the fluids evaluated, would not have any influence on acid-etching of the exposed dentine surfaces. The investigation was carried out using SEM and EDX.

Materials and methods

pH measurements

A method previously described by Land et al.[2] was used to determine the pH. Briefly, a Beckman 10Φ pH meter equipped with a Beckman combination electrode was used for the measurements. pH reference solutions at pH 7 and pH 4 were used to standardise the electrode. All measurements were conducted in triplicate using separate samples from the same bottle.

Specimen preparation for SEM/EDX

Fifty-five freshly surgically removed unerupted human third molar teeth were used. The teeth were stored at 4 °C in isotonic saline containing 0.001% sodium azide, in order to inhibit microbial growth. The roots were sectioned at about 1 mm below the cementoenamel junction (CEJ) using an Isomet low-speed diamond circular saw (Buehler Ltd, Evanston, IL). The pulpal soft tissue was carefully removed with cotton forceps and then rinsed clear. The buccal and lingual surface of each tooth was abraded wet with 220-grit silicon carbide paper for 30 s at 600 rpm, in order to flatly eliminate surface enamel and produce a standardised dentine smear layer. The specimen teeth were then sectioned mesio-distally to obtain 2 mm thick longitudinal flat sections of the lingual and buccal surfaces of each tooth. The prepared buccal or lingual dentine sections from each specimen tooth, while moist was exposed by drop contact, to the respective gingival retraction fluids namely HMDT, AST and ULTB (Table 1). Exposure was carried out sequentially for various periods, namely 30 s, 1, 2 and 5 min. Following the exposure period, the surfaces were rinsed with firm air–water spray for 10 s and dried with a gentle air stream for 5 s without desiccating the dentine.

In another group, similar treatment was carried out on the second of the paired specimen obtained from each tooth sample. However, each of the specimen surfaces were further treated with a 20 s application of 35% phosphoric acid gel (PA)