

# **Effect of dimethyl sulfoxide on dentin collagen**



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#### a r t i c l e i n f o

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### a b s t r a c t

*Objectives.* Infiltration of adhesive on dentin matrix depends on interaction of surface and adhesive. Interaction depends on dentin wettability, which can be enhanced either by increasing dentin surface energy or lowering the surface energy of adhesive. The objective was to examine the effect of dimethyl sulfoxide (DMSO) on demineralized dentin wettability and dentin organic matrix expansion.

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*Methods.* Acid-etched human dentin was used for sessile drop contact angle measurementto test surface wetting on 1–5% DMSO-treated demineralized dentin surface, and linear variable differential transformer (LVDT) to measure expansion/shrinkage of dentinal matrix. DMSO-water binary liquids were examined for surface tension changes through concentrations from 0 to 100% DMSO. Kruskal–Wallis and Mann–Whitney tests were used to test the differences in dentin wettability, expansion and shrinkage, and Spearman test to test the correlation between DMSO concentration and water surface tension. The level of significance was  $p < 0.05$ .

*Results.* Pretreatment with 1–5% DMSO caused statistically significant concentrationdependent increase in wetting: the immediate contact angles decreased by 11.8% and 46.6% and 60s contact angles by 9.5% and 47.4% with 1% and 5% DMSO, respectively. DMSOwater mixtures concentration-dependently expanded demineralized dentin samples less than pure water, except with high  $(\geq 80\%)$  DMSO concentrations which expanded demineralized dentin more than water. Drying times of LVDT samples increased significantly with the use of DMSO.

*Significance.* Increased dentin wettability may explain the previously demonstrated increase in adhesive penetration with DMSO-treated dentin, and together with the expansion of collagen matrix after drying may also explain previously observed increase in dentin adhesive bonding.

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## **1. Introduction**

Ideally, in dentin the adhesive associates intimately with surface and creates a layer which can withstand the mechanical and chemical stress imposed upon it in the oral environment. The hybrid layer where collagen and adhesive intermingle is also considered the weak link, prone to degradation over time by host-derived proteinases and hydrolysis [\[1,2\].](#page--1-0) Failure of fillings to perform is often due to failure in this interface [\[3\].](#page--1-0)

The wetting ability of the adhesive is essential to the formation of a quality bond [\[4\].](#page--1-0) The difference in the free energies of the adhesive and solid surface dictates the manner of interaction. High energy surface is easily wetted, while low energy surface causes the adhesive to bead up on the surface. For optimal wetting solid surface energy should be maximized by surface treatments, which are usually able to expose charged groups or other high energy structures on the surface being treated [\[5\].](#page--1-0)

In both self-etch and etch-and-rinse techniques, dentin surface is modified to enhance adhesive performance. The acid-treated dentin surface includes both crystalline apatite and collagen type I. It is a heterogeneous surface with the ability to absorb liquid and change in dimension when treated with different solvents  $[6]$ . The etched dentin presents a unique challenge to adhesive to interact with crystalline structures and organic components. Expansion of the collagen network is essential for allowing resin infiltration within and among collagen fibrils and for the formation of a good quality hybrid layer [\[7\].](#page--1-0)

Manufacturers have tried to compose their adhesives in such a manner that their wetting ability on dentin surface is maximized. This has led to adhesives that are very hydrophilic [\[8\].](#page--1-0) Etched dentin modification with 2- Hydroxyethyl Methacrylate (HEMA) increases wettability [\[9\].](#page--1-0) However, increase of hydrophilic monomer concentration leads to a hybrid layer with high water sorption [\[10\]](#page--1-0) which is prone to hydrolytic degradation  $[1,3]$ .

Dimethyl sulfoxide (DMSO) possesses a high dielectric constant combined with low surface energy and a capacity to solvate polymers [\[11\]](#page--1-0) and adhesives [\[12\].](#page--1-0) DMSO is an aprotic solvent which possesses the polarity needed to break down water's self-associative tendencies and to form stable complexes with water, especially at 25–35 vol% concentration, resulting with e.g. the lowest freezing point and viscosity [\[13,14\].](#page--1-0) DMSO is an ideal solvent to facilitate radical polymerization reactions [\[15\]](#page--1-0) such as are used in dental adhesion. Pretreatment of acid-etched dentin with DMSO may improve long-term bond preservation [\[16,17\]](#page--1-0) by inhibition of collagenolytic enzymes [\[16\].](#page--1-0) It may even increase the immediate bond strength with etch-and-rinse adhesive which may be due to improved penetration of adhesive into the exposed collagen matrix [\[18\]](#page--1-0) related to the DMSO-induced changes in dentin surface energy. To date, DMSO pretreatment has been shown to have positive effects on dentin bonds strength and its durability with very low (0.004%) [\[16\]](#page--1-0) and relatively high (50%) [\[17\]](#page--1-0) concentrations. Using all possible DMSO concentrations with conventional dentin bond testing methods, such as microtensile bond strength, would be extremely laborious and time-consuming. Therefore, we aimed to look at the effect

of different DMSO concentrations on demineralized dentin behavior in search for the potential explanations that hopefully would guide the further adhesive bonding studies. The null hypotheses tested were that (1) DMSO has no effect on demineralized dentin surface energy, and that (2) DMSO has no effect on the expansion of demineralized dentin.

# **2. Materials and methods**

# *2.1. Specimen preparation for contact angle measurements*

Six intact human third molars extracted as part of patients' routine dental treatment were used in this study. The use of human tissue was approved by Ethical Committee of the Northern Ostrobothnia Hospital District. The teeth were stored in 0.02%  $NaN_3$  at 4 °C until preparation. The teeth were cut with a low-speed saw (Buehler Ltd, Lake Bluff, IL, USA), exposing the superficial dentin and checked to ensure the absence of enamel.

The samples were prepared using the following series of procedures: (1) dentin surface was sanded with 500 grit abrasive paper using circular motions to achieve comparable surface roughness and smear layer; (2) dentin was etched with phosphoric acid etchant (3M ESPE, St. Paul, MN, USA) for 15 s, with gentle agitation to achieve a uniform etched dentin surface; (3) rinsed with deionized water for 15 s; (4) samples were set into individually prefabricated polysiloxane molds (Coltène/Whaledent AG, Altstätten, Switzerland) for stability during contact angle measurements; (5) dentin surface was treated with one of the DMSO (Sigma-Aldrich, St. Louis, MO, USA) deionized water solutions, (0%, 1%, 2%, 3%, 4% or 5-vol% DMSO) for 60 s; (6) surface was dried with filtered, pressurized air for 60 s from approximately 1 cm distance; (7) after drying, contact angle measurements were conducted using deionized water.

# *2.2. Contact angle measurements*

KSV Ltd. CAM 200 Surface energy analyzer (KSV Instruments Ltd, Helsinki, Finland) was used for sessile drop contact angle measurements. A manual liquid dispenser Hamilton Threaded Plunger Syringe (Hamilton Bonaduz AG, Bonaduz, Switzerland) was used for liquid disposal on dentin surface. The Young & Laplace equation which determines the contact angle as a result of energies contributing into static system was used to determine sessile drop contact angles:

# $\sigma$  SV –  $\sigma$  SL =  $\sigma$  LV cos  $\theta$

where  $\sigma$  SV is energy in solid-vapor interface,  $\sigma$  SL is energy in solid-liquid interface,  $\sigma$  LV is energy in liquid vapor surface and  $\cos \theta$  is equilibrium contact angle. The measurement time of 61 s was used to allow equilibrium state to develop, but minimize the effect of evaporation on contact angles. Images were captured at 1 s frequency for 61 s using an automated trigger. For each dentin sample 10 consecutive measurements with the same pretreatment solution were performed. In between measurements, the dentin surface was rinsed with de-ionized water for 15 s and lightly air dried before being exposed to

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