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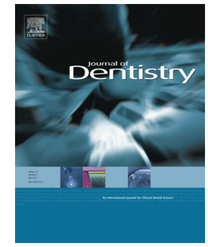

Effects of different artificial ageing methods on the degradation of adhesive–dentine interfaces

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ABSTRACT

Objectives: To compare the effects of four commonly used artificial ageing methods (water storage, thermocycling, NaOCl storage and pH cycling) on the degradation of adhesive–dentine interfaces.

Methods: Fifty molars were sectioned parallel to the occlusal plane, polished and randomly divided into two adhesive groups: An etch-and-rinse adhesive Adper SingleBond 2 and a self-etch adhesive G-Bond. After the composite built up, the specimens from each adhesive group were sectioned into beams, which were then assigned to one of the following groups: Group 1 (control), 24 h of water storage; Group 2, 6 months of water storage; Group 3, 10,000 runs of thermocycling; Group 4, 1 h of 10% NaOCl storage; and Group 5, 15 runs of pH cycling. The microtensile bond strengths were then tested. The failure modes were classified with a stereomicroscope and representative interface was analyzed with a field-emission scanning electron microscopy (FESEM). Nanoleakage expression was evaluated through FESEM in the backscattered mode.

Results: The four artificial ageing methods decreased the bonding strength to nearly 50% and increased the nanoleakage expression of both adhesive systems compared with the control treatment. Adhesive failures were the predominant fracture modes in all groups. However, differences in detailed morphology were observed among the different groups.

Conclusions: Water storage, thermocycling, NaOCl storage and pH cycling could obtain similar degradation effectiveness through appropriate parameter selection. Each *in vitro* artificial ageing method had its own mechanisms, characteristics and application scope for degrading the adhesive-dentin interfaces.

Clinical significance: Water storage is simple, low-cost but time-consuming; thermocycling lacks of a standard agreement; NaOCl storage is time-saving but mainly degrades the organic phase; pH cycling can resemble cariogenic condition but needs further studies. Researchers focusing on bonding durability studies should be deliberate in selecting an appropriate ageing model based on the differences of test material, purpose and time.

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1. Introduction

The emergence and development of adhesive systems have allowed the direct adhesion of restorative materials to tooth structures. As the basis of aesthetic tooth-coloured adhesive restoration, dentine adhesive systems have been developed from the first to the seventh generation in the last decades. Contemporary dentine adhesives present favourable sealing and bonding capabilities in a short period.[1] Despite these improvements, the durability and stability of adhesive–dentine bonds remain limited.[2] Recent studies have found no one-to-one relationship between immediate bonding strength and long-term bonding values because of the rapid degradation of dentine-bonded interfaces.[3] Degradation can weaken adhesion and produce marginal deterioration, which reduce service life and lead to undesirable clinical effects, such as marginal staining, post-operative sensitivity and secondary caries.[4] The reported mean service life of tooth-coloured restorations is merely 5.7 years.[5] Therefore, the degradation of adhesive–dentine interfaces emerged as a hot research topic among dentists and researchers.

Patients' mouths are the ultimate testing environment in predicting and evaluating the bonding durability of adhesive restorations. However, ensuring smooth *in vivo* testing runs and rendering appropriate standardisation are difficult because of several factors, including inter-operator variability, subject diversity, patient willingness and poor feedback.[6] In addition, clinical trials are time consuming and expensive.[7] The durability and stability of adhesive–dentine interfaces are individually or collectively affected by occlusal forces, eating habits and intraoral temperature and humidity changes.[8] Such complex intraoral conditions complicate the process of distinguishing the specific factors related to the degradation of adhesive–dentine interfaces.[9] Thus, an easy, rapid and realistic artificial ageing model should be established to help predict the behaviour and lifespan of adhesive restoration during clinical ageing.

Adhesive restorations are often situated in damp environments surrounded by saliva, and water is crucial in the deterioration of bonding interfaces.[10] Therefore, many previous studies have immersed samples into distilled water to assess bonding durability.[11,12] Intraoral temperature varies depending on eating, drinking and breathing habits. Rapid temperature changes inevitably affect the stability of adhesive restoration.[7] Thus, thermocycling is commonly used to assess bonding durability by simulating the changes in intraoral temperature.[13–15] NaOCl is a popular non-specific proteolytic agent that is widely used in various dental procedures. Recent studies have immersed samples into 10% NaOCl solution, which can dissolve collagen fibrils, to evaluate bonding durability.[16,17] By contrast, little is known about the effects of pH cycling on the degradation of adhesive–dentine interfaces. Intraoral pH varies depending on the acidic component in dentinal fluid, bacterial metabolism, saliva and eating and drinking habits. pH changes trigger acid attack, which affects adhesive–dentine interfaces.[18,19] Thus, establishing a pH cycling model for bonding evaluation is necessary. Different *in vitro* artificial ageing methods, such as water storage, thermocycling, NaOCl storage and pH cycling, may have different effects on the degradation of adhesive–dentine interfaces. That is, each artificial ageing method may have its own mechanism, characteristics and application scope. To date, comparative studies on these *in vitro* models remain lacking.

Microtensile bond strength (MTBS), fracture evaluation and nanoleakage are the most widely used assessment indices for the degradation of adhesive–dentine interfaces. Certain differences exist between etch-and-rinse (E&R) adhesive and self-etch adhesive regardless of which *in vitro* ageing method or assessment index is used.

Therefore, the present study aims to compare the effects of different *in vitro* artificial ageing approaches (water storage, thermocycling, NaOCl storage and pH cycling) on the degradation of E&R and self-etch adhesive–dentine interfaces. The results of this study describe the degrading characteristics of different ageing methods and provide theoretical and experimental bases for their applications. The null hypothesis is that MTBS, fracture modes and nanoleakage do not significantly differ among the different artificial ageing methods irrespective of the adhesive used (E&R or self-etch).

2. Materials and methods

The diagram of the test design used in the study is shown in Fig. 1.

2.1. Specimen preparation

Fifty extracted, noncarious human third molars were gathered after acquiring the donors' informed consents as approved by the Ethics Committee of the School and Hospital of Stomatology, Wuhan University, China. The teeth were scaled, cleaned and maintained in 1% chloramine at 4 °C for no more than one month until their usage. The mid-coronal dentine was exposed by utilising a low-speed water-cooled diamond saw (Isomet; Buehler, Evanston, IL, USA). A standardised smear

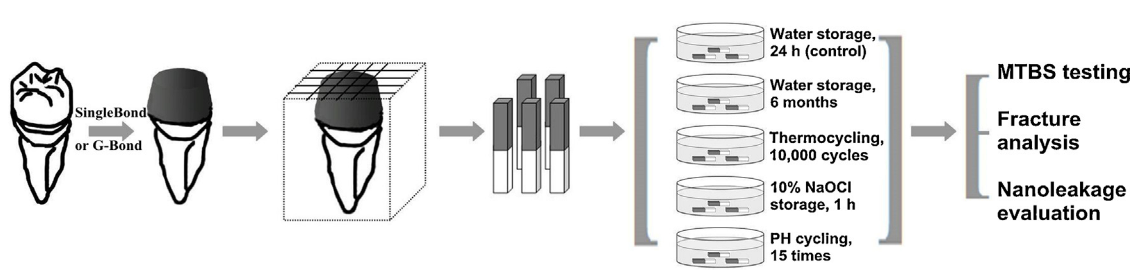

Fig. 1 – Diagrammatic representation of experimental grouping, ageing procedures and test methods used in the study.