

Efficacy of varying the NMEP concentrations in the NMGly–NMEP self-etching primer on the resin-tooth bonding

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Abstract

It is well understood that the application of a self-etching primer enhances the bonding at the resin-teeth interface. In this study, we designed a self-etching primer consisting of *N*-methacryloyl glycine (NMGly) and *N*-methacryloyl-2-aminoethyl phosphonic acid (NMEP). The demineralization effects on the hydroxyapatite or dentin by the carboxylic acid in the NMGly and by the phosphonic acid in the NMEP and their effects on the bond strength of the resin to the tooth were examined. The application of the NMGly–NMEP solution to the enamel resulted in an increase in the bond strength when additional amounts of NMEP were added to the NMGly aqueous solution. This increase was due to the phosphonic acid in the NMEP demineralizing the enamel. Conversely, the addition of the NMEP to the NMGly solution resulted in a decrease in the bond strength to the dentin. The optimal concentration of the NMEP in the NMGly–NMEP solution resulted in bond strengths of over 20 MPa for both the enamel and dentin.

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1. Introduction

Self-etching adhesives have been developed in an attempt to simplify the application of the tooth adhesive system. Most common total-etching adhesive systems consist of three steps. The first step is the application of a phosphoric acid to etch the tooth. The second step is the application of the primer after rinsing the etched-tooth surface with water and then air-drying. The third step is the application of the bonding agent. This can be

contrasted with most common self-etching adhesive systems that consist of two steps. The first step is the application of a self-etching primer to the tooth to condition the tooth. The second step is the application of the bonding agent onto the conditioned layer. Since the self-etching primer both etches and primes the tooth, the second step associated with the total-etching adhesive systems is eliminated. In addition, the bonding agent applied to the conditioned layer is done without any rinsing of water.

The application of a self-etching primer, consisting of an acidic and hydrophilic methacrylate monomer, allows for the acidic monomer to remove or condition the smear layer, and demineralizes the underlining intact tooth. However, the etching potential of the enamel or dentin by the self-etching primer is strongly dependent

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on the type of acidic group in the acidic monomer utilized in the self-etching primer. It is well understood that the acidic monomer, whose acidic group is of a phosphoric acid, has a higher conditioning potential than monomers whose acidic group is of a carboxylic acid [1].

However, the demineralization aspects of the tooth by the acidic monomer have not yet been identified. In addition, significant additional study needs to be undertaken about the molecular level interactions between the acidic group in the acidic monomer and the teeth components. Specifically, it is important to understand the amount of interacted acidic monomer with the teeth components when a self-etching primer has been applied. This would be especially helpful in preventing the over etching of the tooth by an acidic monomer. Studies on the interaction between the acidic monomer and the teeth components have been conducted by using electron spectroscopy for chemical analysis [2] and by using nuclear magnetic resonance (NMR) [3–5].

In order to understand the effects that the degree of demineralization of the tooth by the acidic monomer has on the bond strength of the resin to the tooth, we synthesized a series of three *N*-methacryloxy- ω -amino acids (NM ω As) with carboxylic acids of different pK_a values. These monomer's etching and priming efficacies for tooth bonding were examined [6]. When the NM ω A solution was applied to the dentin, the bond strength of the resin was over 20 MPa. The effect of the carboxylic acid's pK_a value in the NM ω A on the bond strength to the dentin was not observed, even though the demineralization amounts, due to the NM ω A having a priming efficacy on the dentinal collagen, were dependent on the pK_a values of the carboxylic acid. Conversely, for the *N*-methacryloyl glycine (NMGly) whose carboxylic acid's pK_a value was the lowest amongst the three NM ω A derivatives, the maximum mean value of the bond strength to the enamel was only 16 MPa. This value was due to the demineralization of the enamel being limited by the carboxylic acid in the NMGly adhesives.

In order to improve the enamel adhesion for the NM ω A adhesives, we designed the *N*-methacryloyl-2-aminoethyl phosphonic acid (NMEP) as an acidic monomer for the NM ω A adhesives. The NMEP was utilized since the hydroxy group, bonded to the phosphorus in the NMEP, is considered to be of a higher etching potential than the carboxylic acid in the NM ω A.

In this study, we designed a novel NMGly–NMEP self-etching adhesive. The effects of increasing the amounts of NMEP added to the NMGly–NMEP solution had on the demineralization of the tooth and its effects on the bond strength to the tooth were examined. The effect that the degree of demineralization had on the bond strength of the resin to the tooth

conditioned by the NMGly–NMEP adhesive was then discussed.

2. Materials and methods

2.1. Materials

NMGly and NMEP were synthesized by condensing methacrylic chloride to the amine of glycine and to the amine of 2-aminoethyl phosphonic acid [7].

Hydroxyapatite (HAP 200, Taihei Chemistry, Osaka, Japan) was used as a model for the enamel, since the HAP's Ca/P ratio and crystallinity are similar to the enamel's. The dentin particles were prepared as follows. A crown dentin was cut by using an air-turbine with a diamond bur under a stream of cooling water. The cooling water containing the cut dentin particles was collected in a beaker. The cut dentin particles were obtained by decantation and then rinsed three times with distilled water. After the dentin suspension was centrifuged, the dentin particles were transferred to a laboratory dish and then air-dried in a thermo-stabilized room at 20 °C for 1 day. The averaged particle size of the dentin was 14.6 μ m.

2.2. Determination of the pK_a value of the phosphonic acid in the NMEP

An NMEP of 1.5 mmol was dissolved into 2 ml of 20 mass% of deuterium oxide aqueous solution. Hydrochloric acid or sodium hydroxide was then added to the NMEP solution. After the NMEP solution's pH values were measured, the solution's ^{13}C NMR spectra were observed by using a JEOL EX 270 spectrometer (Tokyo, Japan). The chemical shift's resolution for the ^{13}C NMR peak was 0.009 ppm. The hexamethyl-di-siloxane (HMDSO) was used as an external reference. The pK_a values of the phosphonic acid's two hydroxy groups in the NMEP were determined from the titration curve of the chemical shift of the NMR peak attributed to the α -methylene carbon bonded to the phosphorus in the NMEP [7].

2.3. Interacted amounts of the carboxylic acid in the NMGly and of the hydroxy group in the NMEP on the teeth components

An NMGly of 5 mol% was dissolved into 20% deuterium oxide aqueous solution. The NMGly–NMEP solution was prepared by dissolving the NMEP into 1.00 g of the NMGly solution. The amounts of NMEP added to the NMGly solution were 0, 0.35, 0.70, and 1.40 mmol, respectively. The HAP or dentin of 60 mg was then suspended in 600 mg of these NMGly–NMEP solutions. After the suspensions were vibrated for

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