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The effect of functional monomer chain spacer length on the bond strength of an experimental dental adhesive

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ABSTRACT

The study evaluated the effect of the carbon-chain spacer of monomers on the bonding effectiveness of two-step self-etching adhesive systems (SES). We investigated three acrylamidophosphonic acid monomers which vary only by the length of the carbon chain, 2-(*N*-methylmethacrylamido)ethylphosphonic acid, 6-(*N*-methylmethacrylamido)hexylphosphonic acid and 10-(*N*-methylmethacrylamido) decylphosphonic acid, on their effect in bonding performance while formulated in a self-etching primer with dentin and enamel. The results correlated well with those obtained from Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM).

It is concluded that there are no significant differences in terms of immediate Shear Bond Strength (SBS) on either enamel or dentin by using experimental self-etching primer (SEP) of different spacer chain length.

1. Introduction

During the past years, adhesive technology has quickly evolved from etch-and-rinse (ER) systems to more user-friendly. Amongst the currently available adhesive systems, two-steps self-etching adhesive systems (SES) combine etching and priming into one step and do not require rinsing. The apparent user-friendliness of these materials has greatly improved their development and popularity [1]. Contrary to ER systems, SES do not require a separate etching step. Etching is done by acidic monomers contained in the primer or in the bonding resin in the case of all-in-one systems allowing to simultaneously condition and prime the dental substrate [2].

The simultaneous demineralization and infiltration of the tooth surface by SES is partially dependent on the pH of the self-etching solution whether it is a primer or an all-in-one system; the lower the pH, the greater depth penetration is. Self-etch adhesive (SEA) systems can be classified according to their pH as strong ($pH < 1$), mild ($1 < pH < 2.5$) and ultra-mild ($pH > 2.5$) [1,3].

Commonly used acidic monomers are usually constituted in an A-S-P motif, with A being an acidic group, P a polymerizable group and S a spacer like aliphatic chain. Acidic monomers are usually long molecules, for instance, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), 11-methacryloyloxy-1,1′-undecanedicarboxylic acid (MAC-10). Long spacers are used to avoid steric hindrance during polymerization and to enhance mechanical properties [4,5].

There still is a scarcity of data in the literature on the influence of the spacer chain length of acidic functional monomers used in SES on bond strength. By contrast, products of different compositions were compared but not allowing for a conclusion on the length of the chain effect.

The aim of this study was to assess the effect on adhesion of the spacer between the acidic group and the polymerizable one. We chose to use acrylamidophosphonic acid monomers in conjunction with a bisacrylamide co-monomer because those moieties display near-perfect stability towards hydrolysis [6–11]. Degradation of the monomers would have biased our results by introducing unwanted variable.

In this context, we developed three two-step SEA systems composed of experimental acrylamidophosphonic acid-based self-etching primer (SEP) with different aliphatic chain spacer used jointly with a commercially available bonding resin. The variation of the spacer length allowed for a variation of pH. Bonding performance of these SES was investigated along with morphological studies using AFM and SEM.

The null hypothesis was that the functional monomer spacing chain length of the experimental SEP does not affect immediate bond strength.

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0143-7496/

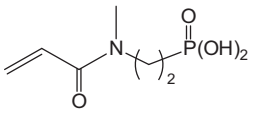
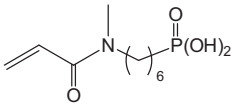
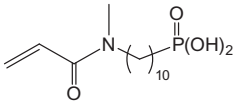
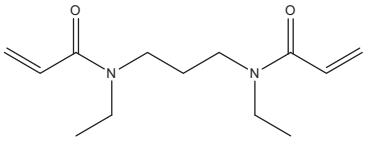
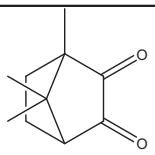
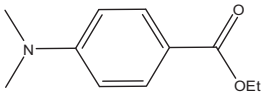
Abbreviated name	Formula
5C2	 2-(*N*-methylmethacrylamido)ethylphosphonic acid,
5C6	 6-(*N*-methylmethacrylamido)hexylphosphonic acid
5C10	 10-(*N*-methylmethacrylamido)decylphosphonic acid
DEBAAP	 *N,N'*-diethyl-1,3-bis(acrylamido)propane
CQ	 camphorquinone
EDAB	 ethyl 4-(dimethylamino)benzoate

Fig. 1. Self-etching primer (SEP) components.

2. Materials and methods

2.1. Syntheses

2-(*N*-methylacrylamido)ethylphosphonic acid (**5C2**), 6-(*N*-methylacrylamido)hexylphosphonic acid (**5C6**), 10-(*N*-methylacrylamido) decylphosphonic acid (**5C10**), presented in Fig. 2 were synthesized according to the literature [6,12].

Triethylamine was distilled over calcium hydride prior to use. Unless stated otherwise, all reagents were purchased from Sigma–Aldrich (Sigma–Aldrich SARL, Lyon, France) and were used without further purification. Dichloromethane was purified with a Puresolv™ apparatus (Innovative Technology, Newburyport, MA, USA). Column chromatography was performed on silica gel Si 60 (40–63 μm). Thin layer chromatography (TLC) was performed on silica gel 60 F_{254} plates (Merck Chemicals, Darmstadt, Germany). All reactions were