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Effect of elastomeric monomers as polymeric matrix of experimental adhesive systems: degree of conversion and bond strength characterization

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Abstract

The aim of this study was to characterize the degree of conversion and the bond strength of experimental adhesive systems formulated with elastomeric monomers (Exothanes). Two-step self-etch adhesive systems were formulated, where the primer was constituted by HEMA, HEMA-P, water and ethanol, and the resin bonds were prepared mixing one type of Exothane (8, 9, 10, 24, or 32) (75 wt%) with TEGDMA (25 wt%). CQ, EDAB, and DPI were added as photo-initiation system. UDMA was used as control, so six different resin bonds were formulated at all. The adhesive system Clearfil SE Bond (CLSE) was used as a commercial control. The degree of conversion (DC) of each resin bond was evaluated in infrared spectroscopy (RT-FTIR, Shimadzu Prestige-21) using a diamond crystal ($n = 3$). The microshear bond strength (μ SBS) test was performed using a universal testing machine (EMIC DL-500). While the DC data was analyzed by one-way ANOVA and Tukey ($p < 0.05$), the μ SBS data was analyzed by Kruskal-Wallis test and Student-Newman-Keuls ($p < 0.05$). The resin bonds containing the Exothanes 8, 9, and 32 demonstrated DC values higher than 80%, differently to the other Exothane-based adhesives, which showed DC values close to 50%. UDMA and CLSE demonstrated lower DC than E₈, E₉, and E₃₂ ($p < 0.001$). The Exothane 24 resulted in the lowest DC value of the study ($p < 0.001$), although it was similar to the E₁₀ and UDMA adhesives ($p > 0.05$). Low μ SBS results were seen for the Exothane-based materials. CLSE demonstrated significantly higher bond strength than the other materials ($p < 0.001$). UDMA has also presented low μ SBS to the dentin substrate. It can be concluded that the Exothanes evaluated demonstrated satisfactory degree of conversion, with some of them reaching almost full conversion of monomers in polymer. However, considering the formulations investigated, they were not good bonding agents. So, they were not reliable options for composing the polymeric matrix of dental adhesive materials.

Keywords: Dental adhesives; Exothanes; Elastomeric monomers; Elastomers; Microshear bond strength; Degree of conversion; Polymerization reaction

Background

Dental adhesive systems are commonly used for bonding the restorative material to the tooth structure. The bonding mechanism is basically characterized by the substitution of superficial tooth minerals for resin monomers, which polymerize *in situ*, creating a micro-mechanical interlocking between the tooth and the bonding substance. This process is known as hybridization, where a hybrid layer is formed between the tooth

substrates and the restorative material [1]. The bonding mechanism starts with the application of an acidic substance, which enables the superficial tooth demineralization; then, another substance is used to prepare the demineralized substrate (primer), followed by the application of a resinous substance (resin bond), which links the tooth to the restorative material. Depending on the mode of using or combining these adhesive substances (acid, primer, and resin bond), different adhesive systems are present in the dentistry field.

The adhesive systems are polymer-based materials, and they are commonly constituted by methacrylate monomers, solvents, and a photo-initiation system, which makes them photo-curable [2]. The monomeric nature plays important roles for each bonding step, where the primer should be a polar substance (hydrophilic) as the tooth is a humid substrate, and the resin bond should be a more non-polar substance (hydrophobic) than the former as this characteristic increases its mechanical strength and reduces the polymer network degradation (hydrolysis). This dual characteristic (polarity and nonpolarity) are very important for acquiring high adhesion between the tooth and the restorative material. Even so, there are several *in vitro* studies which evaluated the bond strength stability between tooth substrates (enamel or dentin) and restorative materials and most of them have concluded that the adhesive interface degrades over time [2-5], mainly because of the monomers used in their composition. This fact may influence negatively with the performance of the restorative procedure. Consequently, materials showing higher strength and stability to wet environment are still needed in dentistry.

Recently, new types of monomers were brought up to the chemistry industry. The Exothane™ Elastomers are categorized as the most recent advance in Esstech's urethane chemistry [6], which could be used for a broad range of demanding formulations, including the adhesive ones. They present different physical characteristics when compared to the urethane dimethacrylate (UDMA), which is a common monomer found in dental materials composition [7,8], such as higher elongation and toughness properties. Moreover, these monomers have the potential of acquiring high conversion values, which is extremely desirable in adhesive materials, improving physical properties and maybe leading to low leachable activity as lower amount of residual monomers would be present at the hybrid layer, and as a consequence lower cytotoxicity [9]. In addition, considering that the Exothanes present elastomeric characteristics, they may imply mobility and capability to relax the polymer network, which is important for relieving the shrinkage stress phenomenon [10]. Nevertheless, there are several types of Exothanes and no existing studies investigating them.

Thus, the aim of the present study is to characterize the degree of conversion and the bond strength of experimental adhesive systems formulated with elastomeric monomers (Exothanes). The null hypothesis evaluated was that the experimental materials would not differ to the performance of a broadly used commercial adhesive system.

Methods

Formulation of the experimental adhesive systems

Two-step self-etch adhesive systems were prepared in the present study. The experimental self-etching primer was formulated by mixing the components described in Table 1, which was the unique primer used in the study. Methacryloyloxyethyl dihydrogen phosphate plus bis(methacryloyloxyethyl) hydrogen phosphate (HEMA-P) was synthesized as

Table 1 Experimental materials formulated in the study: a universal self-etch primer and different resin bonds

SELF-ETCH PRIMER	Components percentage (wt%)			
	HEMA-P	Distilled water	Ethanol	HEMA
$P_{\text{universal}}$	30	20	20	30
RESIN BONDS	Components percentage (wt%)			
	Variation monomer (75wt%)		TEGDMA	CQ + EDAB + DPI
E_8	Exothane 8		25	0.5/1/1
E_9	Exothane 9		25	0.5/1/1
E_{10}	Exothane 10		25	0.5/1/1
E_{24}	Exothane 24		25	0.5/1/1
E_{32}	Exothane 32		25	0.5/1/1
UDMA	UDMA		25	0.5/1/1

HEMA-P, Methacryloyloxyethyl dihydrogen phosphate plus bis(methacryloyloxyethyl) hydrogen phosphate; *HEMA*, hydroxyethyl methacrylate; *TEGDMA*, triethyleneglycol dimethacrylate; *CQ*, camphorquinone; *EDAB*, ethyl-4-dimethylaminobenzoate; *DPI*, diphenyliodonium hexafluorophosphate; *UDMA*, urethane dimethacrylate.

previously described [11]. It was mixed with distilled water, 2-hydroxyethyl methacrylate (HEMA) and ethanol. On the other hand, the resin bonds were formulated using the Exothane monomers, which were Exothane 8 (E_8), Exothane 9 (E_9), Exothane 10 (E_{10}), Exothane 24 (E_{24}), and Exothane 32 (E_{32}). They were all purchased from Esstech (Esstech Inc, Essington, PA, USA) and used without further processing. UDMA was used as control (Table 1). Also, the two-step self-etch adhesive system Clearfil SE Bond (Kuraray, Tokyo, Japan) was used as the commercial control. For the experimental resin bonds formulation, each resin matrix was constituted by one type of the Exothanes (75 wt%) and 25 wt% of triethyleneglycol dimethacrylate (TEGDMA); 0.5 wt% of camphorquinone (CQ), 1 wt% of ethyl-4-dimethylaminobenzoate (EDAB), and 1 wt% of diphenyliodonium hexafluorophosphate (DPIHFP) were added as photo-initiators (Table 1). All the primers and the resin bonds were ultrasonicated for 15 minutes.

Degree of conversion analysis

The degree of conversion (DC) of each resin bond was evaluated in triplicate using Fourier Transform mid-infrared spectroscopy (Prestige21, Shimadzu, Tokyo, Japan). A standard adhesive drop (3 μL) was dispensed over a diamond crystal (Smiths) where a preliminary reading (monomer) for the unpolymerized material was taken. Then, the composite was light-activated with a light-emitting diode unit (LED Raddi, SDI, Bayswater, Australia) for 30 seconds (s) and another reading was carried out (polymer). The DC was calculated as previously described [12].

Microshear bond strength (μSBS) evaluation and Failure analysis

The μSBS test was conducted according to a previous study [13]. Twenty-eight freshly bovine incisors were obtained, cleaned, and stored in 0.5% chloramine T for seven days. Then, they were transferred to distilled water and kept frozen until their use. Each tooth was embedded in acrylic resin, followed by the grounding of its vestibular surface in wet 600-grit silicon carbide paper to expose the dentin substrate. After that, the experimental adhesive systems were actively applied: one coat of the universal self-etch primer ($P_{\text{universal}}$) for 20 s and gently air-dried for 10 s; and one coat of the experimental resin bonds, which were

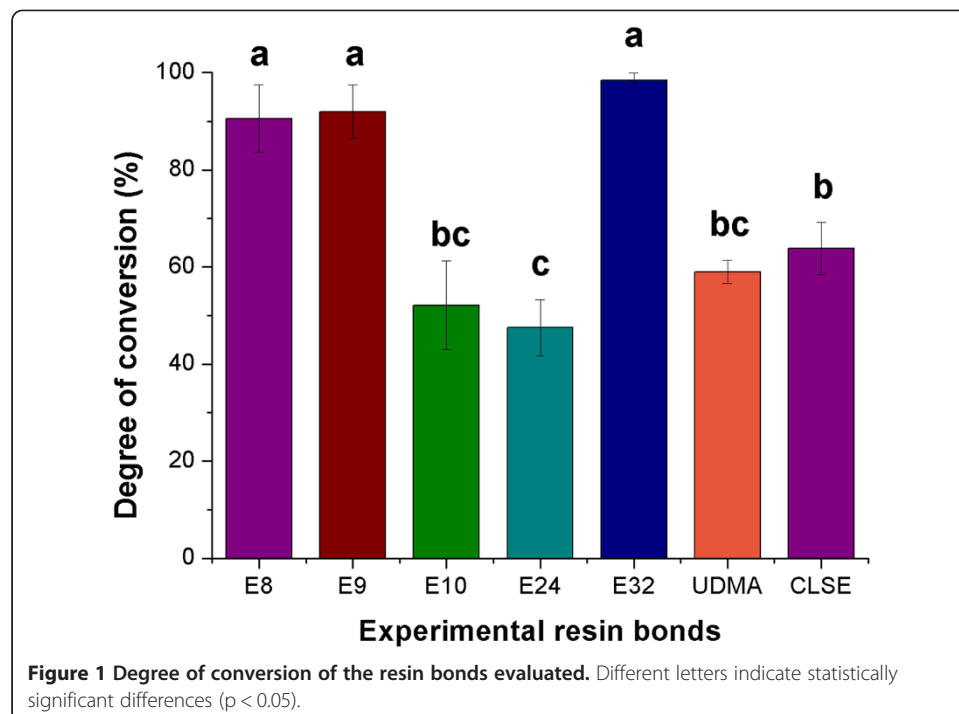
applied for 10 s. Clearfil SE Bond (Kuraray, Tokyo, Japan) was applied as the manufacturer instructions. To delimitate the bond testing area, a circular silicon matrix (array) with three orifices of 1.5 mm in diameter was positioned over the dentin surface, and the light-activation for 20 s was performed on each orifice with the same light-curing unit previously described. Then, an increment of composite resin (Filtek Z-250, 3M ESPE, St. Paul, USA) was inserted into the orifices and light-activated for 40 s. Then, the matrix was gently removed, resulting in specimens with three cylindrical restorations at the dentin surface, which were stored in distilled water at 37°C. After 24 hours, the specimens were positioned in a universal testing machine (DL-500, EMIC, São José dos Pinhais, Brazil), where a thin wire was looped around the composite cylinder restoration. The specimens were submitted to microshear bond strength μ SBS test at a crosshead speed of 1 mm/min and the results were expressed in MPa. After the test execution, the dentin surfaces were examined with a stereomicroscope at a magnification of 40x in an attempt of determining the failure pattern, which was classified as adhesive (on the adhesive interface), cohesive in dentin, cohesive in adhesive resin, or mixed.

Statistical analysis

Data of the degree of conversion were submitted to one-way Analysis of Variance and the Tukey test at the 0.05 level of significance as a *post hoc* test. The microshear bond strength data were submitted to Kruskal-Wallis and Student-Newman-Keuls test as a *post hoc* test at the 0.05 level of significance.

Results

The degree of conversion (DC) obtained with each experimental resin bond formulated and the commercial control is shown in Figure 1. The resin bonds containing the

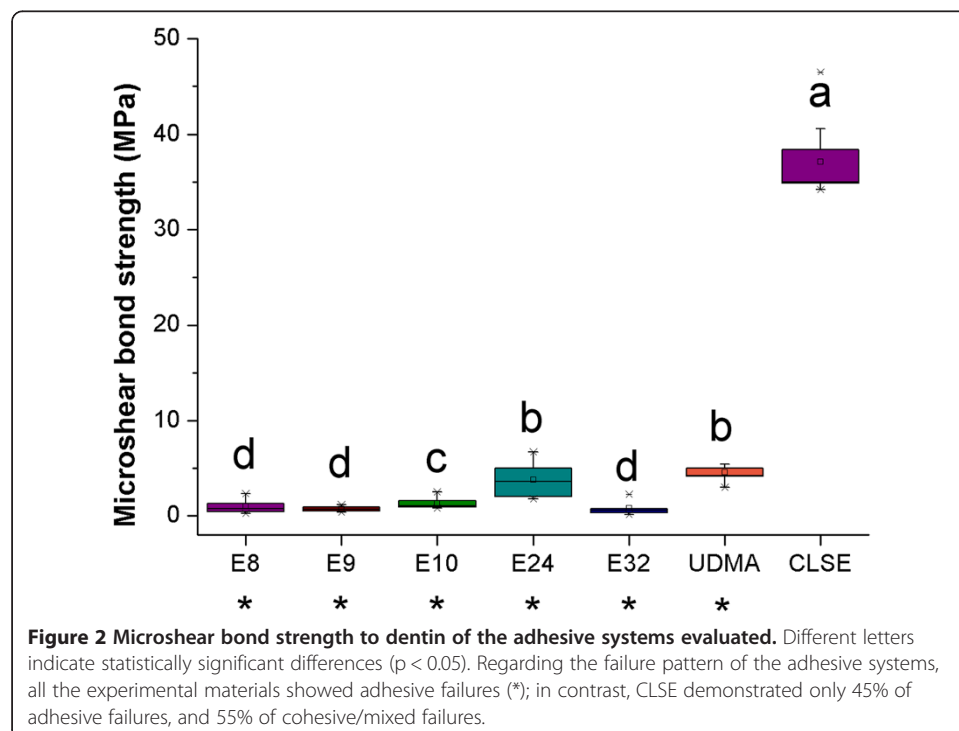


Exothanes 8, 9, and 32 demonstrated DC values higher than 90%, showing higher DC than the control groups and the other resin bonds (E₁₀ and E₂₄) ($p < 0.001$). The resin bonds containing the Exothanes 10 and 24 presented DC values near to 50%, which were similar to the experimental control group (59%) ($p = 0.769$ and $p = 0.257$, respectively). The commercial control group showed a DC value of 63.8%, which was similar to the E₁₀ ($p = 0.247$), but significantly different to the E₂₄ ($p = 0.048$).

The microshear bond strength (μ SBS) results are presented in Figure 2. All the experimental materials (Exothane-based and UDMA-based adhesive systems) resulted in low μ SBS values. In contrast, the commercial CLSE adhesive resulted in 35.6 MPa of bond strength, which was the highest μ SBS median value of the study. CLSE showed higher bond strength than UDMA and the other adhesives ($p < 0.05$). UDMA was as strong as the E₂₄ adhesive system ($p > 0.05$), which demonstrated higher μ SBS values than the other Exothane-based adhesives ($p < 0.05$). With regard to the failure pattern distribution, the Exothane-based and UDMA-based adhesive systems generated only adhesive failure modes, differently to the control CLSE, which demonstrated equilibrium of adhesive (45%) and cohesive/mixed (55%) failures (see in Figure 2, image and legends).

Discussion

The aim of this study was to investigate about the reliability of using the Exothane monomers as polymeric matrix of dental adhesive materials. So, experimental adhesive systems containing these monomers were prepared. It is already known that a satisfactory performance of polymeric dental materials is directly correlated to a proper polymerization reaction of monomers in polymer [7,12,14]. Interestingly, within the degree of conversion results obtained from each resin bond formulated it is possible to verify that some



Exothanes acquired excellent conversion values (Figure 1). In fact, adhesives containing the Exothanes 8, 9, and 32 showed almost full polymerization, and according to Ferracane (2006) [7] how greater the degree of conversion, higher the material's stability to the hydrolysis degradation phenomena. This will probably generate a long-durable material with improved physic-mechanical properties [15]. Nevertheless, not by all the experimental Exothane-based resin bonds resulted in excellent conversion values, as the Exothanes 10 and 24 showed degree of conversion near to 50% (Figure 1).

The conversion of monomers into a polymer network is influenced by intrinsic factors, such as the monomer's degree of functionality (number of double bond terminations per molecule) [16], chain extender length [17], viscosity [18], and reactivity [19]. According to the Esstech site, no data of molecular formula or weight are supplied for the Exothanes 8, 10, 24, and 32. Consequently, their degree of functionality, chain extender length, and reactivity are unknown by the scientific community. In contrast, the Exothane 9, which is not new in the chemistry field, has been already commercialized with another product name (PEG 400 Extended Urethane Dimethacrylate, Item # X-726-0000 from the Esstech catalog) [6]. It presents a functionality 2 and a high molecular weight (1139.4 g/mol), but a low viscosity (1.855 PaS), fact that has probably contributed to its high degree of conversion (Figure 1), as low viscosities facilitate the molecule mobility, increasing its conversion [18]. Unfortunately, the absence of knowledge about the molecular structure and weight of the other Exothanes limits the discussion and understanding of why they react so differently, leading to polymers with almost full conversion (those containing the Exothanes 8 and 32) and others with only half-conversion values (those containing the Exothanes 10 and 24) (Figure 1).

When the Exothane-based resin bonds are compared to the control materials, it can be observed that the presence of the Exothanes 8, 9, and 32 has improved the degree of conversion, differently to the other two Exothanes (10 and 24), which contributed to convert similarly to the UDMA-based resin bond. Considering this result, it can be inferred that these latter Exothanes present a similar molecular structure of UDMA, as they are also urethane-based monomers. With regard to the degree of conversion obtained with the CLSE resin bond, it is in accordance with previous studies [20,21]. In addition, the 64% of monomeric conversion achieved with this adhesive was similar to the UDMA and E₁₀ groups (Figure 1), but different to the E₂₄ resin bond, which showed a statistically lower degree of conversion, probably because the Exothane 24 monomer may present low mobility ability, limiting the polymerization reaction [17].

With regard to the bond strength results, it is possible to verify that the Exothanes are not good options for the development of dental adhesive systems, at least using the formulations evaluated in this study. When only the degree of conversion results are observed, it can be expected a satisfactory adhesive capacity for all the Exothanes, as conversion values higher than 50% are enough for bonding to tooth substrates [22]. However, the bond strength results obtained in this study showed low adhesive ability for all the Exothane-based adhesive systems (Figure 2).

The adhesion process of the tooth substrates is a complex mechanism which involves the demineralization of the surface, micro-porosities formation, resin monomers infiltration, and a proper polymerization. Several factors may hamper one or more of these factors, such as the acidic substance concentration used for etching the substrate [23], the polarity of the adhesive substances applied over the demineralized tooth [24,25],

the complete or incomplete volatilization of any solvent retained in the adhesive layer [26], or the light source and intensity used for photo-activate the adhesive [12]. Considering each of these aforementioned factors, the acidic substance used for the experimental materials formulation was the HEMA-P, which has demonstrated satisfactory demineralization ability in a previous study; [11] after the primer's application, the solvent was volatilized by air stream for 10 s, leading to the formation of a shiny layer over the substrate (a solvent-free pellicle was visually formed); lastly, the light source used was the same for all the materials evaluated. Nevertheless, the polarity of each resin blend formulated is unknown, and this fact may have probably caused the different bond strength results obtained in the study.

The polarity of a monomer is a characteristic that shows its hydrophilicity/hydrophobicity ratio [7]. Generally, the primer substance is applied over the dentin substrate, which is a humid structure that is chemically compatible with hydrophilic molecules; so, the primer should be hydrophilic, enabling the resin monomers infiltration into the demineralized dentin, leading to their entanglement with the exposed collagen fibrils [27]. Nevertheless, hydrophilic substances tend to suffer faster degradation phenomena due to the hydrolysis processes that occur in the oral environment [7,24]. In contrast, the resin bond substance, which is applied over the primer, should be a hydrophobic material, as this characteristic implies strength and more stability for the adhesive to resist the degradation phenomena [28]. Even so, the resin bond should be only moderately hydrophobic, as a poor interaction between the primer and the resin bond may occur (phase separation). Considering that the same primer formulation was used in the current study, the Exothane-based adhesives (experimental resin bonds) have probably an extremely hydrophobic nature, as very low bond strength results were obtained (Figure 2), whit complete adhesive failure modes. This characteristic shows that a proper interaction between the primer and the experimental resin bonds have not occurred, including for the control UDMA-based resin bond. This result may suggest that the urethane derivatives investigated in this study are hydrophobic substances, even when they are mixed with TEGDMA (a more hydrophilic monomer). On the other hand, the control CLSE showed 55% of cohesive/mixed failure modes, indicating that a strong hybrid layer was created between the dentin substrate and the adhesive components. This is already understood because the CLSE is constituted by 10-methacryloyloxydecyl dihydrogen phosphate (MDP), which enables a chemical and a micro-mechanical bonding mechanisms [2], improving the bond strength results, as confirmed by the present study.

Considering the results obtained in this study, the null hypothesis evaluated that the experimental adhesive systems would not differ to a broadly used commercial adhesive system can be partially accepted, as regarding the degree of conversion analysis, some of the materials (E₁₀ and UDMA) demonstrated similar conversion when compared to the control Clearfil SE Bond. With regard to the bond strength performance, all the experimental adhesive systems presented lower bonding ability than the commercial material used. Therefore, further studies evaluating the ultimate microtensile bond strength of Exothane-based adhesives are necessary, mainly investigating different formulations. Moreover, sorption and solubility tests are interesting for clarifying the hydrophilicity or hydrophobicity of the Exothanes, although these monomers should be full-characterized about their physic-mechanical properties, as very few information are available.

Conclusion

Despite of the limitations of this *in vitro* study, it can be concluded that the Exothanes evaluated demonstrated satisfactory degree of conversion, with some of them reaching almost full conversion of monomers in polymer. However, considering the formulations investigated, they were not good bonding agents. So, they were not reliable options for composing the polymeric matrix of dental adhesive materials.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

EAM and LLV participated in the supervision of the experiments and in the writing of the manuscript. GDB and LSA participated in the formulation of the materials and in performing the experiments. EP participated in the design of the study and performed the statistical analysis. SSC and FAO conceived of the study, participating in its design and coordination, helping to drafting the manuscript. All the authors have read and approved the final manuscript.

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