

Acid-Base Resistant Zone in Teeth with the Direct Restoration Using Different Adhesive System Generations: A Systematic Review

Zurab Khabadze¹, Ekaterina Shilyaeva¹, Alexandra Kotelnikova¹, David Todua¹, Yusup Bakaev¹, Saida Abdulkerimova¹, Oleg Mordanov¹

¹Department of Therapeutic Dentistry, RUDN University, Moscow, Russia.

Correspondence: Oleg Mordanov, Department of Therapeutic Dentistry, RUDN University, Moscow, Russia. **E-mail:** mordanov19@gmail.com

Academic Editor: Myroslav Goncharuk-Khomyn

Received: 13 October 2021 / **Review:** 18 June 2022 / **Accepted:** 20 September 2022

How to cite: Khabadze Z, Shilyaeva E, Kotelnikova A, Todua D, Bakaev Y, Abdulkerimova S, et al. Acid-base resistant zone in teeth with the direct restoration using different adhesive system generations: a systematic review. *Pesqui Bras Odontopediatria Clín Integr.* 2023; 23:e210190. <https://doi.org/10.1590/pboci.2023.053>

ABSTRACT

Objective: To find out what the acid-base resistant zone (ABRZ) is and the mechanism of its formation.

Material and Methods: This systematic review was based on the search of laboratory studies in which self-etching adhesive systems were used. The electronic database PubMed was used for the search. The search began on August 2021 and ended on June 2022. We have analyzed the materials and methods of each research and entered them in the appropriate tables to give a clearer assessment of the obtained results. **Results:** This systematic review included 15 full-text articles published from 2011 to 2019. The ABRZ is formed on both dentine and enamel. On dentine, the ABRZ is formed only when using self-etching adhesive systems; on the enamel, on the contrary, the step of preliminary etch and rinse contributes to the formation of a thicker ABRZ. The functional monomer MDP and fluorine increase the thickness of the ABRZ and provide a hybrid layer /ABRZ boundary without defects and erosions. **Conclusion:** Self-etching adhesive systems ensure the creation of an ABRZ resistant to acid-base tests. This phenomenon can provide the resistance of tooth tissues to demineralization, and therefore increase their resistance to caries.

Keywords: Dental Bonding; Dentin; Dental Enamel; Acid Etching, Dental.

Introduction

In more than the last 20 years, adhesive dentistry has made a big step forward. Having achieved a high quality of adhesion, scientists began to develop materials that should facilitate the work of the doctor, reduce the number of stages performed and, consequently, minimize possible errors in each of them. One of the developments was the creation of self-etching adhesive systems (SEA) [1]

In comparison with etch and rinse adhesive systems, self-etching systems contain an acid component that plays an important role, acting as a gentle etching agent. Due to demineralization, resin monomers penetrate the matrix of dental tissues and chemically interact with hydroxyapatite (Hap) crystals [2-5].

In 2004, a zone located directly under the hybrid layer (HL) was described when investigating the dentin/SEA interface [1]. Further research works showed that this zone was characterized by its resistance against acids [1]; therefore, it was named the "acid-base resistant zone" (ABRZ) [1]. The authors suggested that this zone might play an important role in preventing caries formation [6]. In 2009, Waidyasekera et al. [7] used transmission electron microscopy (TEM) to study the structure of the ABRZ after an acid-base test; the results showed that ABRZ has a structure, which is more resistant to caries than ordinary dentin. It is believed that self-etching adhesive systems reduce the post-sealing sensitivity, most likely, this is due to the formation of this zone [8].

The source of ABRZ formation is the hybrid layer of dentin, which releases the functional monomers of the adhesive [9,10]; as a result, a stable, insoluble salt is formed [1]. Further studies revealed the presence of this zone in the enamel sections [2]. However, due to the structure that differs from dentin, as well as the absence of a hybrid layer, the mechanism of ABRZ formation in this case is different [1]. It is interesting to note that the etch and rinse technique excludes the formation of this zone in dentin [5], and on the contrary, increases its width in enamel [1].

There is a wide range of SEA on the market, which differ in the composition of their monomers. Since the formation of ABRZ is determined by the chemical reaction between the monomer and hydroxyapatite crystals, the SEA composition also affects the characteristics of this zone [7,11-14]. Thus, it is necessary to understand the influence of various adhesive systems and techniques of their application on the formation of ABRZ in enamel and dentin.

The primary goal of this systematic review was to find out what the ABRZ is and the mechanism of its formation. Second, the review aimed at studying the influence of the composition of the adhesive system and the protocol of adhesive preparation on the ABRZ formation. Thirdly, it was necessary to study the features of the ABRZ on the enamel and the dentine.

Material and Methods

The concept of this review is based on the PRISMA (Preferred Reporting Items for Systematic Reviews and Meta-Analyses).

Register

The review protocol was registered in an international prospective register of systematic reviews (PROSPERO ID CRD42021284756) in which the methodology and inclusion and exclusion criteria were specified and documented. The research strategy of the present work was formulated according to PICO (Participant, Intervention, Comparison, Outcome), as seen in Table 1.

Table 1. Pico strategy.

P (Participants)	Self-etch and etch-and-rinse adhesive systems
I (Intervention)	Direct restorations with the self-etch and etch-and-rinse protocols
C (Comparison)	Resistance to acid and base of enamel and dentine in self-etch and etch-and-rinse protocols
O (Outcome)	An acid-base resistant zone (ABRZ). Formation of nano-layered structures. Adhesive stability.

Selection Criteria

Publications that met the following selection criteria were included: 1) Full-text articles in English, not older than 10 years; 2) The articles should contain detailed information about the results and parameters of the study (samples and tissues, adhesive system, surface treatment, ABRZ description); 3) The articles contain studies conducted in vitro on human teeth; and 4) The articles contain studies conducted using self-etch adhesive systems (SEA). Publications that were not related to the topic of the study, literature reviews, as well as articles that did not have sufficient and specific data for the analysis were excluded.

Information Sources

The electronic databases used for the search were PubMed and EMBASE. It was not necessary to contact the authors to access the articles. The search started in July 2021 and ended in June 2022.

Search and Selection of Studies

A search in English with no time limit was performed by three independent people. The following search query was used: (("acids"[MeSH Terms] OR "acids"[All Fields] OR "acid"[All Fields]) AND ("alkalies"[MeSH Terms] OR "alkalies"[All Fields] OR "base"[All Fields]) AND ("resist"[All Fields] OR "resistance"[All Fields] OR "resistances"[All Fields] OR "resistant"[All Fields] OR "resistants"[All Fields] OR "resisted"[All Fields] OR "resistence"[All Fields] OR "resistences"[All Fields] OR "resistent"[All Fields] OR "resistibility"[All Fields] OR "resisting"[All Fields] OR "resistive"[All Fields] OR "resistively"[All Fields] OR "resistivities"[All Fields] OR "resistivity"[All Fields] OR "resists"[All Fields]) AND "Zone"[All Fields]) AND (y_10[Filter]).

The studies were filtered and selected in several stages. Firstly, they were evaluated by titles. Secondly, individual documents at the first stage were additionally assessed by reading the abstracts and full-text articles. The difference in the choice was resolved through discussion among the readers.

Risk of Bias

Risk assessment of bias was undertaken during the data extraction process. For the included studies, it was conducted using the Cochrane Collaboration's ROBINS-I tool for assessing the risk of bias [15-17]. The overall risk of bias was then assigned to each trial, according to Higginset et al. [16]. The levels of bias were classified as follows: low risk, if all the criteria were met; moderate risk, when only one criterion was missing; high risk, if two or more criteria were missing; and unclear risk if there were very few details to make a judgment about a certain risk assessment.

Results

A total of 210 articles were identified by keywords and resumes. Duplicate studies were excluded. 34 articles were identified as potentially relevant articles by checking the titles and abstracts, then a full text of 30 articles analysis was carried out, including materials and methods, for compliance with the inclusion criteria.

Articles that didn't meet the inclusion criteria were excluded from this review. As a result, after applying the inclusion and exclusion criteria, 15 full-text articles published between 2011 and 2019 were included and analyzed in the systematic review. After evaluating the selection of articles in accordance with the inclusion criteria, a final analysis of individual studies was conducted. The process of sampling and analyzing studies is presented in the block schematic diagram (Figure 1).

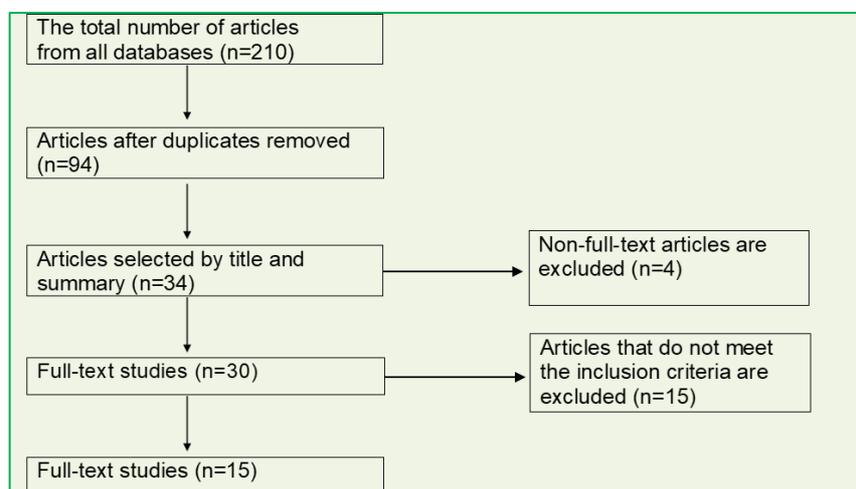


Figure 1. Research selection process.

The studies included in the systematic review evaluated the structure of the acid-base resistant zone (ABRZ) as in dentin [4,10,13-18,20-22,24], so it is in enamel [8,16,19,23] (Table 1).

Table 2. Characteristics of the studies included in this analysis.

Author	Year	Number of Specimens	Research Material	Examined Tooth Tissue	Observation Method
Sato et al. [4]	2019	-	non-carious human third molars and human premolars	Dentin	SEM
Li et al. [8]	2013	50	noncarious human third molars (N=20) and human premolars (N=30)	Enamel	SEM
Kirihara et al. [10]	2013	21	non-carious human molars	Dentin	SEM
Aung et al. [13]	2019	30	non-carious human premolars	Dentin	SEM
Nikaido et al. [14]	2015	9	non-carious human third molars	Dentin	TEM
Guan et al. [15]	2016	45	noncarious human third molars	Dentin	SEM / TEM
Nikaido et al. [16]	2011	15	non-carious human molars	Enamel and Dentin	SEM
Ko et al. [17]	2020	48	non-carious human third molars	Dentin	SEM
Nurrohman et al. [18]	2012	12	non-carious human third molars	Dentin	TEM
Sato et al. [19]	2018	-	non-carious human third molars and human premolars	Enamel	SEM
Vicheva et al. [20]	2021	20	non-carious human molars	Dentin	SEM
Ochiai et al. [21]	2019	-	non-carious human third molars	Dentin	SEM
Matsui et al. [22]	2015	36	non-carious human third molars	Dentin	SEM / TEM
Kakiuchi et al. [23]	2018	-	non-carious human third molars	Enamel	SEM
Nurrohman et al. [24]	2012	-	non-carious human third molars	Dentin	SEM

SEM: Scanning Electron Microscopy; TEM: Transmission Electron Microscopy.

Various adhesive systems and etching protocols were used in experimental tests, which are presented in Tables 3 and 4.

Table 3. Materials used in the study.

Tooth Tissue	Author	Brand Name	Adhesive System		Presence of Components			Surface Conditioning
			Kind		MDP	Fluoride	Calcium	
Dentin	Aung et al. [13]	1. Tooth Primer (TP)	1-SEA	+	-	-	TP was applied to the dentin for 10 s SBU was applied to dentin for 10 s BL was applied to the dentin for 10 s	
		2. Scotchbond Universal adhesive (SCU)	1-SEA	+	-	-		
		3. Bondmer Lightless (BL)	1-SEA	-	-	-		
Dentin	Nikaido et al. [14]	Clearfil SE Bond (SEB)	2-SEA	+	-	-	Adhesives were applied on the ground dentin surface	
		Clearfil Bond SE One (SEO)	1-SEA	+	+	-		
		G-Bond Plus (GBP)	1-SEA	-	-	-		
Enamel	Li et al. [8]	Single Bond (SB)	3-step etch and rinse	-	-	-	Apply etchant for 15 s; water rinse, air dry; apply two coats of adhesive; blow for 5 s. Apply primer for 20 s; air dry for 10 s; apply bond for 10 s; air blow for 10 s. Combinations: Etch with 35% PA for 15 s, water rinse, air dry; apply primer for 20 s, air dry for 10 s; apply adhesive for 10 s, air blow for 10 s. Etch with 35% PA for 15 s, water rinse, air dry; apply adhesive for 10 s, air blow for 10 s.	
		Clearfil SE Bond (SE)	2-SEA	+	-	-		
		PA etching/Clearfil SE Bond primer/Clearfil SE Bond adhesive (PA/SEp+a)		+	-	-		
		PA etching/Clearfil SE Bond adhesive (PA/SEa)		+	-	-		
Dentin	Guan et al. [15]	Clearfil SE Bond (SE2)	2-SEA	+	-	-	Apply primer for 20 s; air dry for 10 s; apply bond for 10 s; air blow for 10 s. Apply primer for 15 s; air dry for 5 s; apply bond for 15 s; air blow for 15 s. Apply the adhesive for 20 s; air dry for 5 s. Combinations: Dry surface (SBD): apply etchant for 15 s; rinse for 10 s; air dry for 5 s; apply adhesive Moist surface (SBM): apply etchant for 15 s; rinse for 10 s; partially dry leaving a visibly moist surface; apply adhesive Apply adhesive for 20 s; air dry for 5 s	
		Optibond XTR (XTR)	2-SEA	-	+	-		
		Scotchbond Universal adhesive (SBS)	1-SEA	+	-	-		
		K-etchant gel/Scotchbond Universal adhesive (SBD and SBM)	PA + 1-SEA	+	-	-		
Enamel and Dentin	Nikaido et al. [16]	Experimental all-in-one adhesive (MDP)	1-SEA	+	-	-	Apply primer for 20 s; air blow; apply bonding agent	
		Experimental all-in-one adhesive (3D-SR)	1-SEA	-	-	-		
		Experimental all-in-one adhesive (4-META)	1-SEA	-	-	-		
Dentin	Kirihara et al. [10]	Experimental two-step self-etch adhesive (different fluoride concentrations)	2-SEA	+	+	-	Apply primer for 20 s; air blow; apply bonding agent	
Dentin	Ko et al. [17]	Clearfil SE Bond (SEB)	2-SEA	+	G1: - G2: + G3: +	-	Pre-conditioning treatment for 3 min: Group 1 – in sterile water (control), Group 2 – in 3.8%SDF, Group 3 – in 38%SDF. Apply primer for 20 s; air dry; apply bond	
Dentin	Nurrohman et al. [18]	Scotchbond multi-purpose (SMP)	3-step etch-and-rinse	-	-	-	Etch with 37% PA for 15 s, water rinse, and air dry; apply primer, air dry; apply adhesive s, air blow Etch with 37% PA for 15 s, water rinse, air dry; apply adhesive s, air blow Adhesives were applied on the ground dentin surface Adhesives were applied on the ground dentin surface	
		Clearfil photo bond (CPB)	2-step etch-and-rinse	+	-	-		
		Clearfil SE Bond (CSE)	2-SEA	+	-	-		
		Adper easy bond (AEB)	1-SEA	-	-	-		
Enamel	Sato et al. [19]	Clearfil Universal Bond Quick (UBQ)	1-SEA	+	-	-	For both 1-SEA:	

		G-Premio Bond (GPB)	1-SEA	+	-	-	1. Apply adhesive and immediately air-dried (UBQ-0, GPB-0), 2. Apply adhesive for 10 s, air-dried (UBQ-10, GPB-10), 3. Etch with 37% PA for 10 s, apply adhesive and immediately air-dried (UBQ-PA, GPB-PA).
Dentin	Sato et al. [4]	Adhesive Universal (AU)	1-SEA	+	-	-	Combinations: 1. AU was applied to the dentin for 20 s; 2. ME was applied for 30 s, water rinse, air dry; AU was applied for 20 s; 3. EC was applied for 10 s, water rinse, and air dry; AU was applied for 20 s; 4. KE was applied for 15 s, water rinse, and air dry; AU was applied for 20 s
Dentin	Vicheva et al. [20]	Clearfil SE Bond (SE2)	2-SEA	+	-	-	Combinations: SE2: Apply SE2 primer for 20 s, air dry, apply SE2 adhesive for 20 s; KE: Apply KE for 10 s, water rinse, and air dry; apply SE2 primer for 20 s, air dry, and apply SE2 c; AP: Apply Alloy Primer for 5 s, apply SE2 primer for 20 s, air dry, and apply SE2 adhesive for 20 s; PB: Mix and apply Porcelain Bond Activator with a drop of SE2 primer for 20 s, air dry, apply SE2 apply SE2 primer for 20 s, air dry, and apply SE2 adhesive for 20 s
Dentin	Ochiai et al. [21]	Combinations: Clearfil SE Bond primer + Clearfil SE Bond (SEP-SEB) Clearfil SE Bond primer + Clearfil Protect Bond (SEP-PBB) Experimental calcium-containing primer + Clearfil SE Bond (CaP-SEB) Experimental calcium-containing primer + Clearfil Protect Bond (CaP-PBB)	2-SEA 2-SEA 2-SEA 2-SEA	+	- + - +	- - + +	For each group: Apply primer for 20 s, air dry, and apply adhesive for 20 s;
Dentin	Matsui et al. [22]	Clearfil SE Bond (SEB) Experimental adhesive (EA)	2-SEA 2-SEA	+	- -	- -	Apply primer for 20 s, air dry, and apply adhesive
Enamel	Kakiuchi et al. [23]	Combinations: Experimental self-etching primer Compositions of experimental adhesives: 00 M0 0F MF	2-SEA	+	- - - +	- - - -	For each group: Apply primer for 20 s, air dry, and apply adhesive
Dentin	Nurrohman et al. [24]	M-Bond (MB) M-Bond II (MB2)	1-SEA 1-SEA	- -	- -	- -	Mix equal amounts of Primer A and B for 10 s, apply to dentin for 20 s, and air dry for 10 s Apply primer for 20 s and air dry for 10 s

AS: Adhesive System; EC: "Enamel Conditioner" (Shofu), which contains 40% organic acid; KE: K-etchant GEL (Kuraray Noritake Dental), which contains 40% phosphoric acid; MDP: Methacryloxydecyl Dihydrogen Phosphate; ME: "Multi Etchant" (Yamakin), which contains methacryloyl oxytetra ethylene glycol dihydrogen phosphate, M-TEG-P; PA: Phosphoric Acid; 1-SEA: One-Step Self-Etch Adhesive; 2-SEA: Two-Step Self-Etch Adhesive; 38%SDF: "Saforide" 38% Ag(NH₃)₂F; 3,8%SDF:"Saforide" 38% Ag(NH₃)₂F

Table 4. Results of studies.

Tooth Tissue	Author	Outer Lesion (OL)	Presence of ABRZ		Morphology of ABRZ	Thickness of ABRZ	Joint between ABRZ and Adhesive
			+	-			
Dentin	Sato et al. [4]	Presence: in all groups Depth: from 10 to 15 µm	1-SEA, ME+1-SEA, EC+1-SEA	KE+1-SEA	1-SEA, ME+1-SEA, EC+1-SEA: relatively regular edge KE+1-SEA: thick hybrid layer	-	1-SEA, ME+1-SEA, EC+1-SEA: funnel-shaped erosion; KE+1-SEA: no erosion
Enamel	Li et al. [8]	Presence: in all groups Depth: from 10 to 15 µm	2-step etch and rinse; 2-SEA; PA+2-SEA; PA+2-SEA (without primer)	-	SE: relatively regular edge, presence of densely arranged grainlike crystals; SB, PA/ SEp+a, and PA/SEa: irregular and wavelike shape edge, presence of densely arranged crystals;	0.5 µm (SE), 3 µm (SB), 5 µm (PA/ SEp+a), 5 µm (PA/SEa)	SB, PA/ SEp+a, and PA/SEa: no erosion SE: extremely small funnel-shaped erosion
Dentin	Kirihara et al. [10]	Presence: in all groups Depth: from 10 to 15 µm	2-SEA	-	0-75% fluoride: relatively uneven edge; 90-100% fluoride: relatively regular edge	ABRZ tended to increase with increasing concentration of NaF in the adhesive	0-20% fluoride: butt joint; 50-75% fluoride: slight slope; 90-100% fluoride: clear, round slope
Dentin	Aung et al. [13]	Presence: in all groups Depth: 20 µm	1-SEA	-	-	-	TP: butt-joint SBU: funnel-shaped erosion BL: slight erosion
Dentin	Nikaido et al. [14]	Presence: in all groups	1-SEA; 2-SEA	-	Thinner at the top and much thicker at the bottom of the OL	0.51 µm (SEB), 0.46 µm (SEO), 0.34 µm (GBP).	SEB: no erosion SEO: funnel-shaped erosion GBP: funnel-shaped erosion
Dentin	Guan et al. [15]	Presence: in all groups Depth: from 10 to 15 µm	1-SEA; 2-SEA; Only in the self-etch groups	PA+1-SEA	electron-dense zone with densely arranged crystallites	0.5 µm (SE2), 0.1 µm (XTR), 0.5 µm (SBS)	XTR: slope at the bottom SBS: funnel-shaped erosion
Enamel and Dentin	Nikaido et al. [16]	Presence: in all groups Depth in dentin: from 10 to 15 µm Depth in enamel: from 10 to 20 µm	1-SEA	-	relatively regular edge in all groups	Dentin: 0.8–1.0 µm (MDP) 0.7–1.0 µm (3D-SR), 0.6–0.7 µm (4-META) Enamel: > 0.5 µm in each adhesive, in MDP thicker than in 3D-SR and 4-META	Enamel: 4-META: funnel-shaped erosion

Dentin	Ko et al. [17]	Presence: in all groups Depth: from 10 to 15 µm	2-SEA	-	No gap or erosive lesion formation in all specimens	from 0.5 to 1 µm	Group 1: butt joint Group 2, 3: slope
Dentin	Nurrohman et al. [18]	Presence: in all groups Depth: from 15 to 20 µm	2-SEA, 1-SEA	3-step etch-and-rinse, 2-step etch-and-rinse (sparsely packed crystals were observed below the HL)	- SPB: sparsely packed crystals were observed below the HL, - SCE: electron-dense zone with densely arranged crystallites - AEB: electron-dense zone with densely arranged crystallites	0.5 µm (SCE), 0.27 µm (AEB)	SMP: funnel-shaped erosion SPB: funnel-shaped erosion SCE: butt joint AEB: funnel-shaped erosion
Enamel	Sato et al. [19]	Presence: in all groups Depth: from 15 to 20 µm	1-SEA, PA+1-SEA	-	-	PA+1-SEA > 1-SEA	1-SEA (UBQ-0, GPB-0, UBQ-10, GPB-10): funnel-shaped erosion PA+1-SEA UBQ-PA, GPB-PA): no gap or defect
Dentin	Vicheva et al. [20]	Presence: in all groups Depth: from 10 to 15 µm	2-SEA	-	SE2, AP, PB: relatively regular edge KE: uneven edge with protrusions	Comparatively similar among the groups (SE2, KE, AP) except for PB, where it was noticeably thinner	SE2: slight slope KE: funnel-shaped erosion AP, PB: no erosion
Dentin	Ochiai et al. [21]	Presence: in all groups	2-SEA	-	In all groups relatively regular edge	0.25 µm (SEP-SEB) - the thinnest, >1.0 µm (SEP-PBB), >0.5 µm (CaP-SEB), >1.0 µm (CaP-PBB)	SEP-PBB and CaP-PBB (in the fluoride bond groups): slope SEB-SEP and CaP-SEB: funnel-shaped erosion
Dentin	Matsui et al. [22]	Presence: in all groups Depth: from 15 to 20 µm	2-SEA	-	SEB and EA: similar thickness of ABRZ. SEB: In the forefront of ABRZ, a high electron density area with a sharp edge; EA: In the forefront of ABRZ, a lower electron density while the forefront	1.0 µm (SEB), 1.0 µm (EA)	SEB: butt joint, EA: funnel-shaped erosion
Enamel	Kakiuchi et al. [23]	Presence: in all groups Depth: from 15 to 20 µm	2-SEA	-	-	0.5 µm (00), 0.5 µm (M0), 1.0 µm (0F), 1.0 µm (MF)	00: funnel-shaped erosion M0: funnel-shaped erosion 0F: no erosion MF: slope
Dentin	Nurrohman et al. [24]	Presence: in all groups	1-SEA	-	-	0.5 µm (MB), 0.5 µm (MB2)	MB: funnel-shaped erosion MB2: funnel-shaped erosion

Two-Step Self-Etch Adhesive on Dentine

The studies selected for the review examined the effect of the following 2-SEA on the formation of ABRZ: Clearfil SE Bond [8,14,15,17,18,20-22], Optibond XTR [15] and three other studies used experimental 2-SEA [10,21,23]. In all samples of 2-SEA groups, the ABRZ was formed on the dentin and enamel. In all studies where Clearfil SE Bond was used, the adhesion between ABRZ and HL was without erosion and formed a butt joint; sometimes, a slope from the top to the bottom of the outer lesion could be observed. Preliminary dentin etching with orthophosphoric acid led to the absence of ABRZ.

One-Step Self-Etch Adhesive on Dentine

The studies included in the review examined the effect of the following 1-SEA on the formation of ABRZ: Scotchbond Universal adhesive [4,13,15], Clearfil Bond SE One [14], Adper easy bond [18] and M-Bond (II) [24]. All the studied samples had KORZ. In all samples with the use of 1-SEA, a funnel-shaped erosion was formed at the ABRZ/HL border. The thickness of the ABRZ ranged from 0.27 to 0.5 microns. Accordingly, preliminary dentin etching with orthophosphoric acid also led to the absence of ABRZ.

Two-Step Self-Etch Adhesive on Enamel

The studies included in the review examined the effect of the following 2-SEA on the formation of ABRZ in the enamel: Clearfil SE Bond [3] and experimental adhesive [23]. In the enamel samples after application of 2-SEA, there was an ABRZ with a thickness of 0.5-3 μm ; however, when performing the stage of etch and rinse with orthophosphoric acid, the thickness was increased and was about 5 μm . Moreover, the stage of etch and rinse before applying 2-SEA contributed to the formation of a butt joint, while its absence led to extremely small funnel-shaped erosion.

One-Step Self-Etch Adhesive on Enamel

The studies included in the review examined the effect of the following 1-SEA on the formation of ABRZ in enamel: Clearfil Universal Bond Quick [19], G-Premio Bond [19], and experimental adhesive [16]. When using 1-SEA, the ABRZ was formed on the enamel with the funnel-shaped erosion at the joint with HL; its thickness was from 0.5 to 1.0 depending on the type of adhesive; however, as in the case of 2-SEA, with the preliminary etching stage with orthophosphoric acid, there was no gaps or defects at the ABRZ/HL border.

Comparison of 1-SEA and 2-SEA

A comparative assessment of 1-SEA and 2-SEA was carried out in three studies included in our review; all tests were performed on dentin [14,15,18]. The formation of ABRZ was observed in all experimental groups. 2-SEA showed the widest crust, the fit of which to the hybrid layer had no erosion. There was funnel-shaped erosion in the 1-SEA samples at the HL/ ABRZ boundary.

Methacryloxydecyl Dihydrogen Phosphate

Among the studies included in the review, the molecule of MDP is contained in the 1-SEA [4,13-15,16,19] and the 2-SEA [8,10,14,15,17,18,20-23]. The MDP-containing adhesives formed thicker ABRZ, both on enamel and on dentin, in comparison with adhesives that do not contain MDP.

Presence of Fluoride and Calcium Ions

In studies included in the review following combinations of two types of ions were considered: adhesive systems with the presence of fluoride [10,14,15,21,23], preconditioning procedure with fluoride [17], adhesive systems with the presence of calcium [21] and adhesive systems with the presence both calcium and fluoride [21]. The presence of fluorine ions provided a butt-joint at the ABRZ/HL boundary, without erosion and defects; the presence of both calcium and fluorine ions also formed a compound without erosion; however, the presence of calcium ions alone in the adhesive led to the formation of funnel-shaped erosion. Adhesives with the presence of fluorine ions created a thicker ABRZ, which was 1.0 μm , while calcium ions did not affect the thickness of the layer in any way.

Discussion

Currently, there is a wide selection of adhesive systems on the market, and despite the existing “gold standard”, there are systems that have certain advantages.

Self-etching adhesive systems, thanks to their etching component, provide a “soft” gradual demineralization of the underlying layer of hard tooth tissues, creating a certain matrix. At the same time, hydrophobic adhesive monomers are infiltrated into this matrix in parallel, which interact with hydroxyapatite crystals, thereby creating a stronger zone of dental tissue. This zone was called ABRZ. Compared to HL, the remaining apatite crystals in ABRZ were relatively denser [14].

The formation of this zone on the dentin is observed only when using self-etching adhesive systems, according to the mechanism described above, and does not apply to etch and rinse adhesive systems [14]. Moreover, it is interesting to note that the degradation of adhesion between these systems also differs [14]. There are two phases of dentin bonding degradation: hydrolytic degradation of the collagen matrix and hydrolytic degradation of the bonding resin within the HL [25,26]. For a etch and rinse system, hydrolysis of unprotected collagen networks is typical, because of incomplete penetration of the resin to the entire etching depth; however, for SEA, this type of degradation is minimal [27]. Hydrolysis of polymer components occurs from the HLs of both etch and rinse and self-etching adhesives since water can remain and penetrate the interface as a result of diffusion from wet dentin during adhesive polymerization [28].

The formation of the enamel and dentin ABRZ has certain differences that are associated with the different structures of these tissues. Enamel has a significantly higher concentration of the mineral component and differs from dentin in the absence of a collagen network [16]. In this regard, demineralization occurs to a lower depth, which causes a smaller thickness of the enamel ABRZ. In studies [16,22], it was noted that preliminary etching of enamel with orthophosphoric acid increases the thickness of the ABRZ. In contrast with dentin, where the source of functional monomers is a hybrid layer, in enamel, these molecules are directly penetrated from the adhesive layer into the etched matrix. Due to the high percentage of the mineral component, soft etching of SAE demineralizes the enamel to a lower depth in comparison with etch and rinse with orthophosphoric acid; therefore, when using the latter, the ABRZ increases.

Comparing two SEA systems, both one-step and two-step, it can be concluded that the latter ensures the creation of a more reliable ABRZ dentin since no undercuts in the form of funnel-shaped erosion are formed at the HL/ABRZ interface [13-15,18]. However, this defect depends not only on the number of steps but also on the components of adhesive systems. Thus, the content of MDP and fluoride provided a connection at the HL/ABRZ boundary by the type of joint; sometimes, a slope was formed, i.e., an increase in the thickness of the ABRZ [10,13,15,16,23]. Regarding enamel, the results largely depended on preliminary etching with

orthophosphoric acid, which provided a butt joint [8,19]. On the contrary, the etching of dentin excluded the formation of ABRZ [4,15,18].

SEA containing the MDP monomer has shown good results with respect to the formation of ABRZ. MDP has two functional groups; one of them is methacrylate, and the other is phosphate. The phosphate groups interact with the calcium ions of hydroxyapatite crystals and form an insoluble, strong salt [1]. When MDP was included in the adhesive system, ABRO was clearly identified not only on the dentine but also on the enamel. Thus, it can be noted that functional monomers in adhesive systems strongly influence the morphology of the hard tooth tissues ABRZ [16].

Adhesive systems, which included fluoride, created a slope-like increase in the thickness of the ABRZ from the border of the sample to the bottom of the outer lesion, which was confirmed by a few studies [7,29-31]. On the contrary, systems without fluorine did not lead to such a phenomenon [16].

The main limitation of this review is that the same group has done all of the work on this topic, so there is no real independent confirmation or otherwise of the formation of the ABRZ and the differences noted with Etch and Rinse systems. In addition, an overall limitation is that a number of the conclusions about restoration longevity, recurrent caries resistance, etc., have not been fully tested in a laboratory setting or clinically. Much of what has been described is a supposition with some evidence for the longevity of some restorations in clinical trials, but the actual effect of the ABRZ cannot be easily tested clinically.

Conclusion

Self-etching adhesive systems ensure the creation of an ABRZ resistant to acid-base tests. This phenomenon can provide the resistance of tooth tissues to demineralization and therefore increase their resistance to caries.

Authors' Contributions

ZK		https://orcid.org/0000-0002-7257-5503	Investigation, Funding Acquisition.
ES		https://orcid.org/0000-0001-9937-5969	Conceptualization, Software, Formal Analysis, Investigation, Data Curation.
AK		https://orcid.org/0000-0001-6359-4561	Methodology, Formal Analysis, Resources, Visualization.
DT		https://orcid.org/0000-0002-6285-3370	Software, Writing - Original Draft, Supervision.
YB		https://orcid.org/0000-0002-0179-4717	Methodology, Investigation, Resources, Data Curation.
SA		https://orcid.org/0000-0002-4471-2128	Data Curation, Writing - Review and Editing.
OM		https://orcid.org/0000-0002-9878-7045	Formal Analysis, Project Administration.

All authors declare that they contributed to critical review of intellectual content and approval of the final version to be published.

Financial Support

None.

Conflict of Interest

The authors declare no conflicts of interest.

Data Availability

The data used to support the findings of this study can be made available upon request to the corresponding author.

References

- [1] Nikaïdo T, Takagaki T, Sato T, Burrow MF, Tagami J. The concept of super enamel formation - relationship between chemical interaction and enamel acid-base resistant zone at the self-etch adhesive/enamel interface. *Dent Mater J* 2020; 39(4):534-8 <https://doi.org/10.4012/dmj.2020-165>

- [2] Bista B, Nakashima S, Nikaido T, Sadr A, Takagaki T, Romero MJ, et al. Adsorption behavior of methacryloyloxydecyl dihydrogen phosphate on an apatite surface at neutral pH. *Eur J Oral Sci* 2016; 124(2):195-203. <https://doi.org/10.1111/eos.12254>
- [3] Fujita Nakajima K, Nikaido T, Francis Burrow MF, Iwasaki T, Tanimoto Y, Hirayama S, et al. Effect of the demineralisation efficacy of MDP utilized on the bonding performance of MDPbased all-in-one adhesives. *J Dent* 2018; 77:59-65. <https://doi.org/10.1016/j.jdent.2018.07.009>
- [4] Sato T, Takagaki T, Baba Y, Vicheva M, Matsui N, Hiraishi N, et al. Effects of different tooth conditioners on the bonding of universal self-etching adhesive to dentin. *J Adhes Dent* 2019; 21(1):77-85. <https://doi.org/10.3290/j.jad.a41917>
- [5] Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, et al. Comparative study on adhesive performance of functional monomers. *J Dent Res* 2004; 83(6):454-8. <https://doi.org/10.1177/154405910408300604>
- [6] Van Meerbeek B, Yoshihara K, Van Landuyt K, Yoshida Y, Peumans M. From Buonocore's pioneering acid-etch technique to self-adhering restoratives. A status perspective of rapidly advancing dental adhesive technology. *J Adhes Dent* 2020; 22(1):7-34. <https://doi.org/10.3290/j.jad.a43994>
- [7] Waidyasekera K, Nikaido T, Weerasinghe DS, Ichinose S, Tagami J. Reinforcement of dentin in self-etch adhesive technology: a new concept. *J Dent* 2009; 37(8):604-9. <https://doi.org/10.1016/j.jdent.2009.03.021>
- [8] Li N, Nikaido T, Alireza S, Takagaki T, Chen JH, Tagami J. Phosphoric acid-etching promotes bond strength and formation of acid-base resistant zone on enamel. *Oper Dent* 2013; 38(1):82-90. <https://doi.org/10.2341/11-422-L>
- [9] Ichikawa C, Nikaido T, Inoue G, Sadr A, Tagami J. Ultramorphologies of the dentin acid-base resistant zone of two step self-etching systems after long-term storage in water. *J Adhes Dent* 2012; 14(3):207-13. <https://doi.org/10.3290/j.jad.a22710>
- [10] Kirihara M, Inoue G, Nikaido T, Ikeda M, Sadr A, Tagami J. Effect of fluoride concentration in adhesives on morphology of acid-base resistant zones. *Dent Mater J* 2013; 32(4):578-84. <https://doi.org/10.4012/dmj.2013-041>
- [11] Nikaido T, Weerasinghe DD, Waidyasekera K, Inoue G, Foxton RM, Tagami J. Assessment of the nanostructure of acid-base resistant zone by the application of all-in-one adhesive systems: Super dentin formation. *Biomed Mater Eng* 2009; 19(2-3):163-71. <https://doi.org/10.3233/BME-2009-0576>
- [12] Takagaki T, Nikaido T, Tsuchiya S, Ikeda M, Foxton RM, Tagami J. Effect of hybridization on bond strength and adhesive interface after acid-base challenge using 4-META/MMA-TBB resin. *Dent Mater J* 2009; 28(2):185-93. <https://doi.org/10.4012/dmj.28.185>
- [13] Aung SSMP, Takagaki T, Ko AK, Halabi S, Sato T, Ikeda M, et al. Adhesion durability of dual-cure resin cements and acid-base resistant zone formation on human dentin. *Dent Mater* 2019; 35(7):945-52. <https://doi.org/10.1016/j.dental.2019.02.020>
- [14] Nikaido T, Nurrohman H, Takagaki T, Sadr A, Ichinose S, Tagami J. Nanoleakage in hybrid layer and acid-base resistant zone at the adhesive/dentin interface. *Microsc Microanal* 2015; 21(5):1271-7. <https://doi.org/10.1017/S1431927615015068>
- [15] Guan R, Takagaki T, Matsui N, Sato T, Burrow MF, Palamara J, et al. Dentin bonding performance using Weibull statistics and evaluation of acid-base resistant zone formation of recently introduced adhesives. *Dent Mater J* 2016; 35(4):684-93. <https://doi.org/10.4012/dmj.2016-059>
- [16] Nikaido T, Ichikawa C, Li N, Takagaki T, Sadr A, Yoshida Y, et al. Effect of functional monomers in all-in-one adhesive systems on formation of enamel/dentin acid-base resistant zone. *Dent Mater J* 2011; 30(5):576-82. <https://doi.org/10.4012/dmj.2010-214>
- [17] Ko AK, Matsui N, Nakamoto A, Ikeda M, Nikaido T, Burrow MF, et al. Effect of silver diammine fluoride application on dentin bonding performance. *Dent Mater J* 2020; 39(3):407-14. <https://doi.org/10.4012/dmj.2019-057>
- [18] Nurrohman H, Nikaido T, Takagaki T, Sadr A, Ichinose S, Tagami J. Apatite crystal protection against acid-attack beneath resin-dentin interface with four adhesives: TEM and crystallography evidence. *Dent Mater* 2012; 28(7):e89-98. <https://doi.org/10.1016/j.dental.2012.04.025>
- [19] Sato T, Takagaki T, Ikeda M, Nikaido T, Burrow MF, Tagami J. Effects of selective phosphoric acid etching on enamel using "no-wait" self-etching adhesives. *J Adhes Dent* 2018; 20(5):407-15. <https://doi.org/10.3290/j.jad.a4135>
- [20] Vicheva M, Sato T, Takagaki T, Baba Y, Ikeda M, Burrow MF, et al. Effect of repair systems on dentin bonding performance. *Dent Mater J* 2021; 40(4):903-10. <https://doi.org/10.4012/dmj.2020-277>
- [21] Ochiai Y, Inoue G, Nikaido T, Ikeda M, Tagami J. Evaluation of experimental calcium-containing primer in adhesive system on micro-tensile bond strength and acid resistance. *Dent Mater J* 2019; 38(4):565-72. <https://doi.org/10.4012/dmj.2018-266>
- [22] Matsui N, Takagaki T, Sadr A, Ikeda M, Ichinose S, Nikaido T, et al. The role of MDP in a bonding resin of a two-step self-etching adhesive system. *Dent Mater J* 2015; 34(2):227-33. <https://doi.org/10.4012/dmj.2014-205>
- [23] Kakiuchi Y, Takagaki T, Ikeda M, Sato T, Matsui N, Nikaido T, et al. Evaluation of MDP and NaF in two-step self-etch adhesives on enamel microshear bond strength and morphology of the adhesive-enamel interface. *J Adhes Dent* 2018; 20(6):527-34. <https://doi.org/10.3290/j.jad.a41632>

- [24] Nurrohman H, Nikaido T, Takagaki T, Sadr A, Waidyasekera K, Kitayama S, et al. Dentin bonding performance and ability of four MMA-based adhesive resins to prevent demineralization along the hybrid layer. *J Adhes Dent* 2012; 14(4):339-48. <https://doi.org/10.3290/j.jad.a22764>
- [25] Sano H, Yoshikawa T, Pereira PNR, Kanemura N, Morigami M, Tagami J, et al. Long-term durability of dentin bonds made with a self-etching primer, in vivo. *J Dent Res* 1999; 78(4):906-11. <https://doi.org/10.1177/00220345990780041101>
- [26] Hashimoto M, Ohno H, Sano H, Tay FR, Kaga M, Kudou Y, et al. Micromorphological changes in resin-dentin bonds after 1 year of water storage. *J Biomed Mater Res* 2002; 63(3):306-11. <https://doi.org/10.1002/jbm.10208>
- [27] Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, Van Landuyt KL. State of the art of self-etch adhesives. *Dent Mater* 2010; 27(1):17-28. <https://doi.org/10.1016/j.dental.2010.10.023>
- [28] Tay FR, Pashley DH, Yoshiyama M. Two modes of nanoleakage expression in single-step adhesives. *J Dent Res* 2002; 81(7):472-6. <https://doi.org/10.1016/j.dental.2010.10.023>
- [29] Shinohara MS, Yamauti M, Inoue G, Nikaido T, Tagami J, Giannini M, et al. Evaluation of antibacterial and fluoride-releasing adhesive system on dentin-microtensile bond strength and acid-base challenge. *Dent Mater J* 2006; 25(3):545-52. <https://doi.org/10.4012/dmj.25.545>
- [30] Iida Y, Nikaido T, Kitayama S, Takagaki T, Inoue G, Ikeda M, et al. Evaluation of dentin bonding performance and acid-base resistance of the interface of two-step self-etching adhesive systems. *Dent Mater J* 2009; 28(4):493-500. <https://doi.org/10.4012/dmj.28.493>
- [31] Tsujimoto M, Nikaido T, Inoue G, Sadr A, Tagami J. Ultrastructural observations of the acid-base resistant zone of all-in-one adhesives using three different acid-base challenges. *Dent Mater J* 2010; 29(6):655-60. <https://doi.org/10.4012/dmj.2010-004>