Universal Adhesives:
The Next Evolution in Adhesive Dentistry?
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Abstract: Every so often a new material, technique, or technological breakthrough spurs a paradigm shift in the way dentistry is practiced. The development and evolution of reliable enamel and dentin bonding agents is one such example. Indeed, the so-called “cosmetic revolution” in dentistry blossomed in large part due to dramatic advances in adhesive technology. It is the ability to bond various materials in a reasonably predictable fashion to both enamel and dentin substrates that enables dentists to routinely place porcelain veneers, direct and indirect composites, and a plethora of other restorative and esthetic materials. In fact, the longevity and predictability of many (if not most) current restorative procedures is wholly predicated on the dentist’s ability to bond various materials to tooth tissues. Adhesive systems have progressed from the largely ineffective systems of the 1970s and early 1980s to the relatively successful total- and self-etching systems of today. The latest players in the adhesive marketplace are the so-called “universal adhesives.” In theory, these systems have the potential to significantly simplify and expedite adhesive protocols and may indeed represent the next evolution in adhesive dentistry. But what defines a universal system, and are all these new systems truly “universal” and everything they are claimed to be? This article will examine the origin, chemistry, strengths, weaknesses, and clinical relevance of this new genre of dental adhesives.

The development of practical adhesive dentistry can be traced to Dr. Michael Buonocore who, in 1955, discovered he could increase the retention of acrylic-based restoratives by first treating the enamel with phosphoric acid. Subsequent research by Buonocore, Gwinnett, and Matsui elucidated the mechanism of adhesion as micromechanical attachment via resin infiltration and tag formation within the acid-demineralized enamel. While long-term bonding to phosphoric-acid-etched enamel surfaces has proven to be highly reliable and predictable, long-term bonding to dentin has been considerably more problematic. This is largely due to morphologic, histologic, and compositional differences between the two substrates. For one thing, dentin is a vital, dynamic, and highly variable substrate. Superficial, middle, and deep dentin can vary significantly in structural, physical, and chemical composition. Enamel, conversely, is quite consistent throughout and is also considerably more mineralized than dentin. The inorganic content of mature enamel is approximately 96% hydroxyapatite by weight; the remainder consists of water and organic material. Dentin, on the other hand, is approximately 70% hydroxyapatite by weight, 18% organic material (predominantly type I collagen), and 12% water. These percentages are not consistent and can vary significantly depending on several factors, including dentin depth, age of the teeth, and history of tooth trauma and/or pathology. This, coupled with the relatively high water content of dentin, presents a significant challenge for consistent and reliable long-term adhesion.

The few available adhesive systems of the 1970s and early 1980s were relatively hydrophobic in nature and unable to adequately penetrate the dentin smear layer, thus limiting their direct interaction with the tooth tissues. The smear layer is the residue that is left on the surface of the dentin after rotary instrumentation with diamond or carbide burs (Figure 1). It is a thin amorphous layer largely composed...
of degraded collagen, bacteria, and various inorganic dentin and enamel debris. Early adhesive systems were extremely limited and generally ineffective, in part because they bonded directly to the smear layer and were, thus, limited by the smear layer’s low intrinsic cohesive strength. Eventually, it was recognized that the smear layer needed to be removed and/or modified and bypassed in some fashion so that adhesive primers and resins could interact directly with the dentin. In the case of total-etch adhesive systems, the smear layer is essentially dissolved with phosphoric acid (H₃PO₄) and subsequently washed away during the rinsing step (Figure 2 and Figure 3). With self-etching systems, various acidic primers are used to modify, disrupt, and/or solubilize the smear layer and, although the remnants are not washed away as with total-etch systems, still permit direct adhesive interaction with the dentin substrate.

The acids and/or acidic primers and conditioners used with either total- or self-etching bonding systems do not just remove and/or disrupt the smear layer but create a thin zone of demineralization, exposing collagen fibrils that are either subsequently (total-etch) or concurrently (self-etch) infiltrated with various functional and cross-linking primers and resins. One of the goals in developing a successful adhesive interface is the infiltration and penetration through this acid-demineralized zone with various primers and/or resins that can be subsequently polymerized by light and/or chemical curing mechanisms. It is this thin layer of resin-infiltrated dentin, first described in a classic 1982 paper by Nakabayashi and colleagues, that is called the hybrid layer (Figure 4). Although micromechanical resin infiltration and entanglement with the tooth tissues appears to be the primary attachment mechanism to both enamel and dentin, strong evidence suggests that certain monomers (such as 10-MDP) chemically interact, via ionic bonding, to calcium in hydroxyapatite as well. The hybrid layer and associated resin tags form a thin polymerized micromechanically, and in some cases chemically, attached resinous surface layer that acts as the foundation for subsequently placed chemically compatible restorative materials and resin-based cements.

While an in-depth discussion of the imperfect, and sometimes confusing, classification categories typically used to describe adhesive systems (etch-and-rinse, etch-and-no rinse, three-bottle, two-bottle, one-bottle, all-in-one, various generations, etc.) is beyond the scope of this article, it is reasonably safe to say that all adhesive systems, regardless of classification category, contain some type of acidic conditioner, dentin primer, and bonding resin. For example, the basic protocol when using a three-step total-etch system (4th generation) is the sequential placement of the three primary components (acidic conditioner, hydrophilic primer typically consisting of HEMA and adhesive functional monomer(s), and hydrophobic resin). In the case of 4th generation systems the components are packaged in separate containers and applied sequentially. Typically, phosphoric acid is first placed on enamel and dentin for a period of time, rinsed off with water, then a hydrophilic primer is placed and

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**TABLE 1**

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<th>Universal Adhesives and Their Basic Chemistries</th>
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<td><strong>ADHESIVE UNIVERSAL</strong> (IVOCLAR VIVADENT)</td>
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<th>Phosphate Ester Components</th>
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<th>Compatibility with self-cure mechanism of self- and dual-cure resin cements</th>
<th>No dual-cure activator is required with dual-cure resin cements as long as adhesive is light-cured first, according to manufacturer.</th>
<th>Dual-cure activator is not required when using self- or dual-cure resin cements because of higher pH compared to other universal adhesives.</th>
<th>Dual-cure activator is required; however, it is not required if Clearfil DC Core Plus or Panavia 5A resin cement is used.</th>
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<td>Two-component adhesive. Components are mixed in single-dose “blister” pack just prior to use. Once mixed, dual-cure activator is not required.</td>
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air-dried, followed by the placement of a separate, relatively hydrophobic bonding resin. The simplified two- and one-bottle systems still contain the aforementioned three primary components, but the components are consolidated and combined in various configurations depending on the system. In any case, as most dentists know, the trend in recent years has been toward not just the simplification of adhesive systems, but making them universal as well.

Table 1 lists eight products that are marketed as universal adhesives, along with their basic chemistries.

What is a “Universal” Adhesive?
There does not appear to be any “official” definition as to what qualifies as a universal adhesive. In any case, universal adhesives should not be confused with 7th generation self-etching single-bottle or “all-in-one” systems such as iBond (Heraeus Kulzer), Xeno IV (DENTSPLY Caulk), Clearfil™ S3 Bond (Kuraray), and OptiBond All-In-One (Kerr Corporation). For one thing, universal adhesives are said to have much broader applications than 7th generation systems. Universal adhesives have been described by some manufacturers and opinion leaders as: ideally a single-bottle, no-mix, adhesive system that can be used in total-etch, self-etch, or selective-etch mode depending on the specific clinical situation and personal preferences of the operator. Additionally, manufacturers typically state that universal adhesives can be used for the placement of both direct and indirect restorations and are compatible with self-cure, light-cure, and dual-cure resin-based cements. It is further stated that universal adhesives can be used not only to bond to dentin and enamel, but as adhesive primers on substrates such as zirconia, noble and non-precious metals, composites, and various silica-based ceramics. In principle, this would enable bonding to these surfaces without the need for dedicated and separately placed primers such as silane and various products marketed as metal and zirconia primers.

If this unofficial definition as to what constitutes a universal adhesive is accepted, it becomes apparent that a degree of ambiguity exists as to where certain products that are sometimes marketed as universal adhesives actually fit in. For example, OptiBond™ XTR (Kerr Corp.) is a two-bottle system that the manufacturer describes on its website as a “self-etch, light-cure, universal dental adhesive” that can be used for direct and indirect restorations, is compatible with light-, self-, and dual-cure resin cements, and bonds to metals, zirconia, porcelain, and composite. While this may all be true, as a two-bottle self-etching system with individual components that are applied separately and in a sequential fashion when bonding to tooth tissues, would OptiBond XTR be better described as a 6th generation self-etching system with expanded functions? Some products marketed as universal adhesives, such as Futurabond Universal Bond (VOCO), require the mixing of two separate components prior to use. This product comes in individual disposable blister packs that are easily activated just prior to use. There may be very sound reasons for keeping the chemistry of an adhesive...
It’s All About the Chemistry
In order to develop a truly universal adhesive, very specific and synergistic functional and cross-linking monomers that are multifunctional in nature are required. They must be capable of reacting with a number of different substrates, be able to copolymerize with chemically compatible resin-based restoratives and cements, and have some hydrophilic character in order to properly “wet” dentin that has a significant water content, yet at the same time be as hydrophobic as possible once polymerized to discourage hydrolysis and water sorption over time. Film thickness of the polymerized adhesive must also be thin enough to not interfere with the seating of indirect restorations. In addition, universal adhesives ideally should be acidic enough to be effective in a self-etching mode but not so acidic that they breakdown initiators needed for the polymerization of self-and dual-cure resin cements.15

Universal adhesives must also contain water, as it is required for dissociation of the acidic functional monomers, inherent in all these systems, that makes self-etching possible. One of many dilemmas faced by chemists developing universal adhesives is that while some water is needed, too much can degrade the chemistry of these systems, contribute to phase separation of monomers, decrease shelf-life, and be difficult to completely evaporate during the air-drying step.16,17 Residual water after air-drying could result in incomplete adhesive polymerization, increased hydrolysis after polymerization, and a generally compromised adhesive interface. Adding ethanol or acetone into universal adhesive formulations enhances resin wetting and infiltration of tooth tissues and also aids in water removal and evaporation during the air-drying step. There are many other important subtle factors and nuances that vary from manufacturer to manufacturer, such as pH, initiator and solvent chemistry, and specific monomer types and ratios, that also play a vital role in the viability of these systems and, in some situations, give certain products advantages over others. An examination of the chemistry of the systems listed in Table 1 shows some remarkable similarities, as well as some subtle and important differences.

The 10-MDP Adhesive Functional Monomer
All of the universal adhesives listed in Table 1 use phosphate esters (R-O-PO₂H₂) as their primary adhesive functional monomer. In fact, phosphate esters form the backbone of virtually all current materials marketed as universal adhesives. They are well documented for their ability to promote strong and stable bond strength to porcelain and zirconia, while Prime&Bond Elect® (DENTSPLY Caulk) requires the addition of a separate activator for all self- and dual-cure resin cements. In addition, the manufacturers of some universal adhesives still recommend the use of separate and dedicated primers to optimize bond strength to substrates such as porcelain and zirconia.

Thus, it appears, at least in certain situations and with some products, that universal adhesives actually consist of two bottles, or require the use of an additional activator, or have chemistries that must be mixed prior to use, or bond most optimally to porcelain and zirconia with separately applied and dedicated primers, or are not compatible with a total-etch protocol. Suddenly, the definition of what qualifies as a universal adhesive becomes a bit muddled. Does this mean some products should not be classified as universal adhesives, or does the definition of exactly what a universal adhesive is need to be broadened? In any case, can universal adhesives, however they are defined, really do all of the things they are said to be able to do? Clearly, there must be significant practical and chemical challenges in developing such a versatile product, placing all the chemistry required into one or even two bottles, have it perform as claimed, and have it remain stable for a reasonable period of time. So, how is it done?

Fig 1. Typical “peanut butter spread on toast” appearance of the dentin smear layer. (SEM courtesy of Jenny Wang.) Fig 2. Dentin treated with 37% phosphoric acid for 15 seconds, rinsed, and blot dried. The smear layer has been removed and a thin zone of demineralization created. Collagen fibrils, which are inherent in dentin, are now exposed as they are no longer supported and surrounded by their inorganic hydroxyapatite scaffolding, which has essentially been “dissolved” away by the acid. It is important to penetrate this “collagen network” as completely as possible with subsequently placed primers and adhesives in order to achieve good bonding. (See Fig 3.) This SEM is basically the same as Fig 2 except the collagen seen in Fig 2 has been removed with collagenase enzymes exposing the dentin underneath that has not been, or has been minimally, demineralized by acidic pretreatment. It is this interface that is important to engage, via hybrid layer formation, to achieve good bonding and a well-sealed interface. (SEM courtesy of the late Dr. John Gwinnett.)
universal adhesive systems and enable them to do much of what they do. These monomers have many positive attributes, including the potential to bond chemically to metals, zirconia, and to tooth tissues through the formation of non-soluble Ca++ salts. In addition, their acidic nature (they are esters of phosphoric acid) gives them the potential to etch and demineralize tooth tissues, which makes them good candidates for use in adhesives that require self-, selective-, and total-etching options.

A very practical and proven phosphate ester, which also happens to be the one used in the formulations of many current universal adhesives, was actually synthesized more than 30 years ago. In the early 1980s chemists at Kuraray (Osaka, Japan) synthesized the adhesive functional monomer 10-MDP (methacryloyloxydecyl-dihydrogen-phosphate) (Figure 5). One of the first practical applications of this new monomer was its use in the company’s Panavia™ adhesive resin cement. Panavia proved to be especially adept at bonding to metals, and its success led to the use of the MDP monomer in virtually all subsequent Kuraray adhesives. The 10-MDP monomer has many positive attributes that make it practical for use in a universal adhesive. It is a versatile amphiphilic functional monomer with a hydrophobic methacrylate group on one end (capable of chemical bonding to methacrylate-based restoratives and cements) and a hydrophilic polar phosphate group on the other (capable of chemical bonding to tooth tissues, metals, and zirconia). This attribute alone makes it desirable for use in a “universal” adhesive. Moreover, the long carbon chain backbone of the monomer renders it fairly hydrophobic. In fact, with a partition coefficient of 4.1 (partition coefficient is essentially a measure of how hydrophilic or hydrophobic a chemical substance is) 10-MDP is the most hydrophobic of all the functional monomers typically used in dental adhesives. This may be important in terms of durability, as water sorption and hydrolytic breakdown of the adhesive interface over time has been implicated as one of the primary causes of bond failure. It makes sense that, once they are placed and polymerized, adhesive monomers that discourage water sorption are desirable. The hydrophobic nature of 10-MDP also makes it relatively stable in solution, which is important in terms of shelf-life. Additionally, 10-MDP is one of the few monomers used in adhesive dentistry that has been shown to actually bond chemically to the tooth tissues via ionic bonding to calcium found in hydroxyapatite (Ca$_{10}$[PO$_4$]$_6$[OH]$_2$). Stable MDP-calcium salts are formed during this reaction and deposited in self-assembled nano-layers of varying degrees and quality depending on the adhesive system. This type of molecular interaction and self-organization, coupled with the relatively hydrophobic nature of polymerized 10-MDP, helps explain why this monomer appears to be so effective in creating adhesive interfaces that are resistant to biodegradation (J. Oxman, chemist, 3M ESPE, personal communication).

When Kuraray’s patent on 10-MDP expired (around 2003) other manufacturers began to explore its potential. In October 2009, Bisco, Inc. introduced a dedicated zirconia primer, Z-Prime™, that utilized a combination of 10-MDP and biphenyl dimethacrylate (BPDM) in its formulation (C. Suh, Bisco, Inc., personal communication). The use of secondary ion mass spectrometry (SIMS) at Northwestern University showed evidence of actual chemical bonding between the 10-MDP in Z-Prime and zirconia and the product received very favorable reviews for use as a dedicated zirconia primer (Figure 6 through Figure 11). Two years after the introduction of Z-Prime, 3M ESPE introduced the first “universal” adhesive (Scotchbond Universal), which also utilized 10-MDP in its formulation (J. Fundingsland, 3M ESPE, personal communication). This product was followed shortly thereafter by Bisco’s...
All-Bond Universal®, also featuring the 10-MDP monomer. In fact, six of the eight universal adhesives listed in Table 1 use 10-MDP in their formulations (including one that uses a modified 10-MDP [information provided by G. Connell, Director of Clinical Education & North American Training, VOCO Canada]). In addition to 10-MDP, other phosphate esters such as PENTA-P (dipentaerythritol penta acrylate monophosphate) and GPDM (glycerophosphate dimethacrylate) also appear to be viable alternatives. In any case, it is apparent that the use of phosphate esters is an important part of the universal adhesive story, but it’s only part of the story.

The Delicate Balance Between Hydrophilic, Hydrophobic, And Adhesive Functional Monomers

A great paradox of adhesive dentistry is that the chemistry that helps make adhesive systems so effective initially can also contribute to their eventual breakdown. One reason for the success of current adhesive systems is the use of hydrophilic monomers that are able to interact with and “wet” tooth tissues that are, to some degree, inherently moist. The problem is that the same hydrophilic groups that initially facilitate primer/resin interaction with the tooth tissues can become a liability in the long term by encouraging water sorption and hydrolysis of the adhesive interface. Indeed, one of the major problems with 7th generation single-bottle self-etching systems is that the inherently hydrophilic nature of the polymerized adhesive, coupled with residual water that may remain, has been shown to act as a semi-permeable membrane permitting water diffusion that, over time, could lead to hydrolysis and breakdown of the adhesive interface. One could argue that an “ideal” dentin bonding agent would be one that is hydrophilic when first placed (to facilitate interaction with the tooth tissues) but then becomes hydrophobic once polymerized (to discourage water sorption). The next best thing would be gradation from hydrophilic to hydrophobic as one moves from the tooth surface to the restorative interface. Indeed, this is the strategy employed by the original three-step total-etch (4th generation) and two-step self-etch (6th generation) adhesive systems—that is, the initial placement of a hydrophilic primer, which is then overlaid by a relatively hydrophobic bonding resin.

Chemists developing single-bottle universal adhesives had to formulate an optimized blend of chemically compatible hydrophobic, adhesive functional, and hydrophilic monomers that would work in a synergistic fashion and when polymerized form a durable, and hopefully hydrophilic, bonded interface. In this regard, the different monomers employed in universal adhesives each have their own specific functions. Generally, the hydrophilic ends of monomers interact with the tooth tissues, while the hydrophobic ends interact with methacrylate-based restorative materials or cross-link with other functional and structural monomers. The terminal ends of some adhesive functional monomers are hydrophilic initially (such as the ionic phosphate group in 10-MDP) but become more hydrophobic once they chemically react with tooth tissues and are polymerized.

There are significant challenges in striking just the right balance between hydrophilic and hydrophobic character when developing a universal adhesive, as the monomers need to initially be hydrophilic enough to wet, infiltrate, and interact with the dentin substrate, but once they are polymerized, not so hydrophilic that they encourage water sorption that could lead to hydrolysis and breakdown of the adhesive interface over time. Manufacturers of universal adhesives address these issues by blending some well known and widely used monomers such as bis-GMA (hydrophobic) and hydroxyethyl methacrylate (HEMA) (hydrophilic) along with proprietary and various adhesive functional monomers that vary from manufacturer to manufacturer. One of the goals after placing, drying, and curing a universal adhesive should be the creation of a highly cross-linked hydrophobic polymer matrix that is well bonded to the tooth tissues on one end and to restorative materials, such as composites, on the other. Some manufacturers may have advantages over others in this regard.

Almost all adhesive systems, including universal systems, utilize HEMA in their formulations (Figure 12). HEMA is a versatile low molecular weight hydrophilic monomer that is particularly adept at infiltrating and “wetting” dentin substrates. It is extremely soluble in water, ethanol, and acetone, and thus easy to incorporate into adhesive formulations. The hydrophilicity of HEMA makes it an excellent adhesion-promoting monomer that has been shown to

Fig 6. Two zirconia winged resin bonded bridges replacing Nos. 7 and 10. The wings and frame are 100% zirconia. Porcelain has been pressed over the frame, creating the veneers Nos. 7 and 10. Fig 7. After the bridges were tried in and the intaglio surface of the zirconia wings were sandblasted with 50-µm aluminum oxide at 30 PSI to 40 PSI of air pressure, a 10-MDP dedicated primer was placed and warm-air-dried for 30 seconds.
improve immediate bond strengths of adhesive systems by enhancing monomer diffusion into dentin and facilitating the formation of the “hybrid” layer.\textsuperscript{30,32} HEMA is frequently added to adhesives, not only to ensure good wetting, but also because of its solvent-like nature. This improves stability and helps keep hydrophobic and hydrophilic monomers in solution by minimizing phase separation in the presence of water (HEMA-free adhesives can have issues with phase separation).\textsuperscript{30,33,34}

While HEMA has many positive attributes there is also a downside. HEMA in both the uncured and polymerized state readily absorbs water. Once polymerized it can swell, discolor, and contribute to hydrolysis of the adhesive interface.\textsuperscript{29,32,35} High amounts of HEMA can also decrease mechanical properties of the resulting polymer. Uncured HEMA also has the potential to lower the vapor pressure of water and may make it more difficult to evaporate during the air-drying step.\textsuperscript{36} While the concentration of HEMA used in universal adhesives varies from manufacturer to manufacturer, the goal should be to optimize the HEMA concentration (use least amount possible) to take advantage of the benefits of this monomer while concurrently minimizing its undesirable properties.

The pH Puzzle

The pH of current universal adhesives varies from about 2.2 to 3.2 depending on the product. Universal adhesives are generally considered to have “mild” (pH > 2) or “extra-mild” (pH > 2.5) etching capabilities.\textsuperscript{37,38} Adhesives in this pH range can be very effective in terms of bonding to dentin. The concern is that they may not be as effective when it comes to bonding to enamel (especially to uncut enamel).\textsuperscript{39-41} As an example, a popular two-bottle 6th generation self-etching system (Clearfil™ SE Bond, Kuraray) with a pH in the range of current universal adhesives and which also contains the 10-MDP monomer has been shown to bond more predictably to enamel in some clinical studies when the enamel is first etched with phosphoric acid.\textsuperscript{42-44} In fact, a common clinical technique when using this product is to first etch the enamel with traditional phosphoric acid gel (selective-etch technique). While there are in-vitro studies that do demonstrate acceptable bond strength values to enamel using the self-etching mode of some universal adhesives,\textsuperscript{45,46} caution is urged as there is equivocation in the literature\textsuperscript{47-50} and some systems may perform significantly better (or worse) than others when it comes to enamel bonding.\textsuperscript{51} The author’s personal preference, and recommendation, is to use universal adhesives only in the selective- or total-etching modes when enamel is present to ensure predictable long-term bonding to this substrate. In the case of bonding in full-coverage restorations, where there is little or no enamel remaining, then universal adhesives used in the self-etching mode is a viable, and perhaps even preferred, option.

There is also a direct correlation between pH and the compatibility of universal adhesives with self- and dual-cure resin cements and composites. As a generalization, the more acidic the adhesive the less compatible it is with the self-cure mode of dual-cure resin-based materials.\textsuperscript{52,53} This is primarily due to acid deactivation of the aromatic tertiary amines that play a crucial role in chemical curing mechanisms of most of these materials.\textsuperscript{29} To overcome this issue, several universal adhesives, when used in conjunction with self- and dual-cure resin cements, require the addition of a separate “activator” (typically arylsulfinate salts) either all of the time (Prime&Bond Elect) or unless specific dedicated amine-free resin cements are used (Scotchbond Universal, Clearfil Universal). At least one universal adhesive (All-Bond Universal) is compatible with most common self- and dual-cure resin cements without the use of a separately applied activator. This is because this adhesive is less acidic, with a pH of 3.2, than other universal adhesives that have pHs ranging between 2.0 and 3.0. This is enough of a difference to allow the reactions necessary for chemical curing of self- and dual-cure resin cements.
Will Universal Adhesives Replace Dedicated Primers?

The manufacturers of most universal adhesives state they can be used not only for bonding to dentin and enamel, but as adhesive primers on substrates such as zirconia, noble and non-precious metals, composites, and silica-based ceramics. The question isn’t whether universal adhesives are capable of bonding to these substrates (they are), but are they as effective, both initially and, more importantly, over time, as separately placed dedicated primers? There is some controversy surrounding this question. Opinions differ from manufacturer to manufacturer, and, in the author’s view, additional objective independent research is needed before making definitive recommendations.

In terms of bonding to zirconia, there are studies that demonstrate some universal adhesives are very effective as zirconia primers. However, at least two studies also found that the bond strength values of some universal adhesives to zirconia decreased significantly after thermocycling or 6 months of water storage. There are also respected chemists and researchers who feel that the most durable bond to zirconia is attained when separate and dedicated primers are employed. One such primer (Z-Prime), consisting of a phosphate ester (10-MDP) and carboxylic acid monomer (BPDM), has proven to be especially effective in this regard and does not require light-curing, which alleviates concerns some may have regarding film thickness (universal adhesives used as zirconia primers should be air-thinned and light-cured). Some self-etching 10-MDP-containing resin cements, such as Panavia SA Cement (Kuraray), have also shown promise bonding to both tooth tissues and substrates such as zirconia without the use of separately placed adhesives or primers. It is possible that products of this nature would perform even better if the zirconia surface were first treated with a universal adhesive or dedicated zirconia primer (the author is unaware of any research that has specifically examined this).

Another issue is how the dentist should treat the zirconia surface prior to placing a universal adhesive or dedicated zirconia primer. It is the author’s strong opinion that the zirconia surface should be sandblasted prior to utilizing any adhesive, primer, or resin-based cement. There is significant support in the literature for this recommendation. While there is some concern that sandblasting has the potential to induce surface and subsurface cracks and/or defects that could reduce physical properties, the author is unaware of any studies or anecdotal evidence that demonstrates this to be a clinical problem. Sandblasting zirconia is useful in terms of cleaning the target surface of impurities, increasing surface roughness, raising surface energy, improving the bond to primers and adhesives, and generally optimizing the surface prior to bonding or cementation.

The author recommends using a sandblaster (eg, Microetcher™ II, Danville Materials) with 30-µm to 50-µm aluminum oxide (Al₂O₃) at 30 PSI to 40 PSI of air pressure (2.0 to 2.8 bar). The intaglio surface of the restoration is sandblasted after it has been tried in and just prior to dedicated primer or universal adhesive application. If the dentist does not have a sandblaster then the author recommends the dentist have the laboratory sandblast the restoration and then use a product such as Ivoclean (Ivoclar Vivadent), which is a solution of sodium hydroxide, polyethylene glycol, water, and zirconia oxide, after the restoration has been tried in and prior to primer application. While the use of phosphoric acid (H₃PO₄) can be an effective cleaning agent for saliva-contaminated silica-based ceramics (such as lithium disilicate) it is contraindicated for cleaning zirconia surfaces. This is because phosphate ions from the phosphoric acid remain bound to the zirconia surface (even after rinsing) and compete with phosphate...
ions from zirconia primers for reaction sites on the surface (zirconia has a strong affinity for phosphate ions). Likewise, the phosphate ions in saliva can tie-up reactive sites on the zirconia surface. Studies show that the best way to treat saliva-contaminated sandblasted zirconia surfaces is by re-sandblasting or using a strongly alkaline cleaning solution such as Ivoclean.71–73

Some universal adhesives also claim they can be used in lieu of dedicated silane coupling agents when bonding to silica-based ceramics (feldspathic, lithium disilicate, etc.). Historically, the use of hydrofluoric acid (HF) followed by the application of a dedicated silane coupling agent has been the treatment of choice in this regard, and the author has previously discussed this topic in great detail.74 Silanes are a class of organic molecules that contain one or more silicon atoms. Dozens of different silane compounds exist and are used extensively in industry and manufacturing. The silane typically used in dentistry, for both intraoral repair and the priming of silica-based ceramic restorations prior to placement, is 3-methacryloxypropyltrimethoxysilane (Figure 13 through Figure 16). The manufacturers of some universal adhesives have incorporated silane directly into their adhesive formulations, as noted in Table 1, with the idea that the universal adhesive can now be used instead of a separately applied dedicated silane solution. This makes sense as long as all the other chemistry found in universal adhesives does not interfere with silane stability and performance. The manufacturers of other universal adhesives have chosen not to add silane into their formulations because they question the stability of silane in the acidic environment of a universal adhesive and/or believe the chemical interaction of silane with silica-based ceramics is significantly inhibited when combined with all the other monomers found in universal adhesives. Indeed, contact angle studies have found that the incorporation of bis-GMA resin or MDP (both of which are used in universal adhesives) into silane solutions substantially reduced the priming efficiency and chemical interaction of the silane with silica-based lithium disilicate compared to pure silane controls.75,76 Once again, in the author’s opinion, the efficacy and wisdom of incorporating silane into universal adhesives is an area that requires further study and clarification.

When bonding to silicate-based ceramics, the author’s personal preference, at least at this time, is to etch the porcelain with HF followed by the use of a dedicated pre-hydrolyzed silane primer (eg, RelyX™ Ceramic Primer, 3M ESPE; Porcelain Primer, Bisco, Inc.; Ultradent® Silane, Ultradent Products, Inc.) that is free of any added monomers or resins (pure silane).

Fig 15. The hydroxyl groups on the activated silane molecule are now capable of reacting directly with corresponding hydroxyl groups present on the surface of silica-based ceramics. The opposing hydroxyl groups first interact with one another via hydrogen bonding. As water is lost, a condensation polymerization reaction occurs and covalent bonds are formed. Warm, dry air encourages this reaction. Fig 16. In this illustration, the individual silane molecules have covalently bonded not just to the porcelain surface, but to adjacent silane molecules, essentially forming a polymer “network” on the surface. The methacrylate group on the other end of the silane molecule can now react—via free radical addition polymerization—with methacrylate groups in subsequently placed adhesives and methacrylate-based resin cements.
Is It Time to Switch to a Universal Adhesive?
In August 2014, at a “key opinion leader” meeting comprised of 32 dentists, academicians, researchers, and chemists, the author conducted a written survey that included several questions, some of which, along with responses and attendee comments, are listed below:

- **Question:** “Do you, or have you, used any of the new universal adhesive systems?” Of the 32 responses, 30 answered “yes.”
- **Question:** “At this time what is your ‘go-to’ adhesive, ie, the one you use, or would recommend, most often?” There were 19 responses answering universal adhesives (two such products in particular were mentioned). Nine respondents answered either 4th or 5th generation total-etch systems, and four answered 6th generation self-etching systems.
- **Question:** “Even though universal adhesives are supposed to bond to a variety of substrates, do you still feel separate dedicated metal, zirconia, and porcelain primers should be used? If yes, why do you feel that way?”

Most interestingly, 29 of the 32 respondents to the last question said they would still use a dedicated primer. Comments included: not trusting that universal adhesives would work as well as a separate primer; stability being an issue; Z-Prime Plus giving the best bond strength to zirconia; longer-range research data existed for dedicated primers; more steps still proves to be more durable, reliable, and longer lasting. Another concern was “never felt comfortable with silane being contaminated.” It should be pointed out that comments such as these are sometimes visceral in nature and not necessarily based on science. In any case, it is clear that there are some concerns about the ability of universal adhesives to perform as predictably as dedicated primers (especially when stressed by aging or thermocycling), and more research is needed that directly compares the two.

So, is it time to make the switch to a universal adhesive? Certainly, the versatility, simplicity, potential to reduce product inventory, chemistry, and relatively hydrophobic nature of polymerized universal adhesives do make them an attractive option. The author has been using a 10-MDP-containing universal adhesive system, most often in total- or selective-etching mode, almost exclusively now for just over 2 years with excellent clinical success. There are certain points to keep in mind that will optimize the performance of universal adhesive systems (as well as all adhesive systems):

1. **Make sure to evaporate the solvents.** All adhesive systems employ acetone, ethanol, water, or a combination of these as solvents for their particular monomers. It is extremely important to evaporate these solvents as completely as possible by air-drying for an adequate period of time prior to polymerization. Inadequate solvent evaporation has been associated with incomplete resin polymerization, nanoleakage, and decreased bond strength. Increasing the manufacturer’s recommended air-drying times and/or the use of warm air dryers may be prudent in this regard.

2. **Even good chemistry will not overcome poor clinical technique.** For example, all universal adhesives recommend “rubbing” or “scrubbing” the tooth preparation with adhesive for at least 20 seconds, drying, and then light-curing for an adequate period of time with a quality bonding light. Dentists should critically examine their own technique to make sure these guidelines are being followed.

3. **Check the expiration date.** All bonding agents utilize chemistries that can deteriorate significantly over time. This is especially true when they are subjected to high temperatures. Refrigeration may be useful in this regard, but adhesives should be removed and allowed to warm up to room temperature at least 30 minutes prior to use.

4. **Be sure to read the directions.** All adhesive systems tend to have their own specific placement and handling idiosyncrasies that must be followed precisely for optimal results. What works well for one system may not be applicable for another.

**Conclusion**
Proper management of the adhesive interface is crucial for the predictable placement of many current dental restorations. This requires an understanding of the materials being utilized, the substrate being bonded to, and a correct and precise clinical protocol. It is incumbent on every dentist to learn about the specific adhesive system being used, its idiosyncrasies, strengths, and weaknesses, and how to maximize its performance. While clinical trials and clinical experience remains the ultimate test for all dental materials, universal adhesives represent an exciting and promising new class of dental adhesives that the author suspects will soon dominate the adhesive marketplace.

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**DISCLOSURE**

The author has no affiliation with any of the companies mentioned in this article.

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New Material Options, New Cement Selections
Labs share valuable insight and perspective when it comes to adhesion
Gary Alex, DMD, AAACD | Larry Borman

Due to the upsurge of new and innovative indirect ceramic, zirconia, and composite-based materials, it is understandable that dentists and laboratory technicians are sometimes confused regarding optimal placement protocols. Questions often arise about whether the dentist’s current cementation products and protocols are acceptable and compatible with these new materials, or whether an entirely new cementation system and protocol are required.

Understandably, most dentists prefer sticking with materials they already know, have, and trust. No dentist wants to have to purchase another cementation kit just to place a new prosthetic or restorative material they may use only occasionally or even abandon entirely. It is helpful to briefly examine the types of cements and resin adhesives currently in use and determine their applicability when using these newer materials.

Exploring the Options
Cementation options for indirect restorations usually entail the use of one of three distinct classes of materials: resin-modified glass ionomers (RMGI) (eg, RelyX™ Luting Plus Automix Resin Modified Glass Ionomer Cement, 3M ESPE, www.3mespe.com; FujiCEM™ 2, GC America, www.gcamerica.com); self-etching/priming dual-cure resin cements that are used without a separate dentin bonding agent (DBA) (eg, RelyX™ Unicem Self-Adhesive Universal Resin Cement, 3M ESPE; Maxcem, Kerr Corporation, www.kerrdental.com; G-CEM™, GC America); and dual-cure resin cements that are used with a separate DBA (eg, RelyX™ Ultimate Adhesive Resin Cement, 3M ESPE; DUO-LINK™, BISCO, Inc., www.bisco.com; Multilink®, Ivoclar Vivadent, www.ivoclarvivident.com).

It can be argued that the two classes of resin cements have a distinct advantage over RMGI cements when it comes to bonding restorations on or in minimally retentive preparations, because resin cements have the potential to bond more durably to both the tooth structures and the restorative material. They are also generally the best choice when placing porcelain restorations that can be etched with hydrofluoric acid and silane treated, as resin cements bond extremely well to etched porcelain and optimize overall assembly strength.

The resin cements are also often the best choice for restorations placed in the esthetic zone, because RMGI cements tend to be opaque and whitish in color and can “show through” many restorations, creating unacceptable esthetics.

Although self-etching/priming resin cements have the obvious advantage of not

### What to Look for in a Dual-Cure Resin Cement

- Low solubility
- Low film thickness
  - (the current ISO standard for combined film thickness of the adhesive/cement is ≤25 μm; however, a film thickness ≤20 μm is much more ideal)
- High compressive strength
- High fracture toughness
- High flexural strength
- Easy delivery and self-mixing capability
- Easy cleanup
- No postoperative sensitivity
- Predictable self-cure mechanism
  - (When light can’t reach an area of the restoration, it is still very important for the cement to cure predictably. Some “dual cure” materials actually do not self-cure nearly as well as they light cure. Ideally, a resin-based cement will self-cure as well as it light cures)
require a separate DBA, the highest bond strengths are generally achieved when a dual-cure resin cement is used in conjunction with a separate DBA. In fact, some studies have showed that even the self-etching/priming “Cem” type cements perform better (in terms of bond strength) when a separate cure resin cement is used in conjunction with a dual-cure cement for which it is specifically designed. Guidelines for choosing a dual-cure resin cement are found in Table 1.

This integrated system of cement, adhesive, and restorative material serves as a strong example of the benefits that can be achieved with new material options. To ensure maximum efficiency and optimal results, even experienced dentists can gain from communicating closely with their lab when a new restorative is introduced.

Cementation may not be one of the most frequent topics of conversation between the dentist and the lab, but that doesn’t mean that labs don’t have good advice to offer dentists when it comes to this important step. A lab expert’s advice on general topics, including reliable manufacturers and combinations and specific product recommendations, can help dentists shave time off the cementation procedure and achieve optimal results. Open lines of communication can help both parties keep each other apprised of their own experiences with new cements and materials, as well as news from trade shows and continuing education opportunities.

Guidance for New Restorative Materials
With the introduction of any new restorative material, dentists and labs should carefully consider placement recommendations of the manufacturer. For example, one recently introduced product—3M ™ ESPE ™ Lava ™ Ultimate Restorative—is a resin nanoceramic, representing a new class of materials. As such, dentists may be unsure about the optimal placement protocol. Labs will often advise dentists to stay within the manufacturer’s product line when placing indirect restorations, and this logic is applicable when using this material. Lava Ultimate restorations can be placed using Scotchbond™ Universal Adhesive (3M ESPE) along with RelyX Ultimate cement (a dual-cure resin cement). These three products are designed to work together. The resin cement has an integrated dark-cure activator that works in conjunction with the adhesive to ensure complete curing even in the absence of light. The cement was developed to work especially well with glass ceramics, but it is also indicated for composite and metal materials.

One additional feature of this cement that requires a separate DBA, the highest bond strengths are generally achieved when a dual-cure resin cement is used in conjunction with a separate DBA. In fact, some studies have showed that even the self-etching/priming “Cem” type cements perform better (in terms of bond strength) when a separate cure resin cement is used in conjunction with a dual-cure cement for which it is specifically designed. Guidelines for choosing a dual-cure resin cement are found in Table 1.

Even experienced dentists can gain from communicating closely with their lab.”

Reliability and Simplification
Both dentists and labs can appreciate the importance of simplified and time-saving systems, and both partners obviously have a strong interest in providing patients with restorations that will look natural and last for the long term. New material options can throw many established procedures into question, but by recommending and choosing cements from proven and reliable manufacturers, labs and dentists can take much of the guesswork out of the process.

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Preparing Porcelain Surfaces for Optimal Bonding

Gary Alex, DMD*

Abstract: When placing an indirect restoration such as a porcelain veneer, inlay, or onlay, the intaglio surface is typically treated in some fashion to ensure optimal bonding to the resin-based adhesives and luting cements required to affix it to the tooth tissues. A variety of surface preparation techniques have been advocated, including the use of acids, particle abrasion, various adhesives, and chemical couplers such as silane. There is often equivocation in the literature regarding the effectiveness of these materials and surface preparation techniques. This article will attempt to make some sense of the various methodologies currently advocated for the treatment of porcelain surfaces before placement.

Optimal surface preparation techniques for chemical and/or mechanical bonding to porcelain substrates are crucial to ensure clinical success when placing indirect porcelain restorations and, when required, repairing them intraorally. Clinicians are often confused regarding the most effective way to treat the intaglio surfaces of indirect porcelain restorations before placement with various adhesives and luting resins. They are often equally perplexed about the “ideal” surface treatment for the intraoral repair of preexisting porcelain restorations. This is not surprising, as there appears to be no clear consensus in the dental literature, among “opinion leaders,” or from dental manufacturers on exactly what the optimal surface treatment should, in fact, be. Dental laboratory technicians also appear to lack standardized protocols on how they should treat the surface of finished porcelain. For example, in 2007, the author spoke with the owners and/or ceramists from 11 different dental laboratories regarding their specific surface treatment protocol before sending out finished feldspathic porcelain veneers. All treated the inner surface of the veneers with hydrofluoric acid (HF). Only two knew the actual concentration of the HF they were using. Application times varied from as short as 30 seconds to as long as 10 minutes. Several laboratory technicians stated they simply etched the veneers until the surface looked “frosty.” Some laboratories sandblasted the veneers before HF treatment and some did not. After etching, some laboratories placed the veneers in an ultrasonic cleaner, some “steam” cleaned the veneers, some placed the veneers back in the oven at a low temperature to “burn off” any white “residue” left over from etching, and others merely rinsed them with tap water. Three laboratories stated they “sometimes” used a basic solution to “neutralize the acid” after HF treatment, although they were not sure if this was a necessary step. This author also questioned approximately 24 “opinion leaders” at a recent symposium regarding their recommendations for the intraoral repair of both composite and porcelain restorations. While there were many similarities, there were also significant differences, and no treatment protocols were exactly the same.

Learning Objectives:
After reading this article, the reader should be able to:
- discuss how hydrofluoric acid etching affects the bonding potential of porcelain materials.
- describe how silane reacts with porcelain and creates a bond.
- explain the approach required to bond high-content alumina and zirconia core-based ceramics.
- compare the general treatment protocols for porcelain veneers that have been etched and have not been etched in the dental laboratory.

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The lack of clear and consistent guidelines regarding the treatment of composite and porcelain surfaces raises several significant questions. Is there, in fact, an optimal surface treatment protocol for porcelain restorations before placement and for intraoral repair? Is it possible that several viable surface treatment options exist? Is it even practical to recommend a specific “universal” treatment protocol because of material differences (eg, not all porcelains are the same)?

This article will attempt to address these questions, provide some general guidelines, and no doubt raise additional questions regarding the management of porcelain surfaces. A good place to start is by examining what we “think” we know.

**BONDING TO PORCELAIN—USE OF HF**

HF is an inorganic acid capable of etching glass surfaces. It has been used for hundreds of years to create decorative designs and patterns on glass and various ceramic materials. It is also used as a precursor in the manufacturing of numerous pharmaceuticals, various polymers (eg, Teflon), and many other synthetic materials that contain fluorine.

Contrary to what most dentists believe, because of its low tendency to dissociate into H+ and F- ions, HF is considered to be a relatively weak acid from a chemical standpoint. This doesn’t mean HF is not dangerous. Quite the contrary; due to its low dissociation potential, HF has the ability to readily penetrate skin tissues (often without causing an external burn), where it can cause extensive internal tissue damage, as well as alter blood calcium levels (due to the formation of CaF2), which can lead to dangerous heart arrhythmias. There have been industrial accidents where death has occurred from accidental skin exposure to HF.

The duration of exposure, the concentration of HF, and the total surface area affected will obviously have an effect on clinical outcomes relating to accidental dermal exposure.

In typical dental applications (eg, the etching of porcelain veneers and the intraoral repair of fractured porcelain restorations), concentrations of 4% to 10% HF are typically used. In this author’s opinion, HF in this concentration range can be used safely for dental procedures, including intraoral repair, provided caution and common sense are employed. Whenever HF acid is used for intraoral repair, the use of appropriate barrier techniques, viscous HF gel formulations (ie, that stay where you put them), and continual visual observation during the application period are mandatory. For those dentists not comfortable with the extraoral use of HF, acidulated phosphate fluoride gel (APF) has been shown to have the ability to etch porcelain, although the application time must be considerably longer than for HF etching. One recent study showed that a 7- to 10-minute application of 1.23% APF gel on a leucite-containing porcelain (IPS Empress®, Ivoclar Vivadent, Inc,Amherst, NY) produced a shear bond strength to composite (Filtek™ Z250 Universal Restorative, 3M ESPE, St. Paul, MN) similar to a 4-minute etch with 9.6% HF. It should be pointed out that this same study showed that etching with 9.6% HF for 4 minutes resulted in a very definitive microscopic etching pattern, while etching for 7 to 10 minutes with 1.23% APF resulted in a pitted, but much smoother-appearing surface. This difference in surface morphology could be significant over the long term, and it would have been interesting to see what effect thermocycling of the samples, which was not done, would have had on bond strength. Also, it is unclear why the investigators used 9.6% HF for 4 minutes when the manufacturer of the porcelain tested recommends that 5% HF acid be used for only 60 seconds. It is possible that the bond strength to the HF-treated porcelain samples would have been even higher had the manufacturer’s recommended protocol been followed. Other studies have also shown that etching with APF, even with prolonged application times, results in very shallow etching patterns when compared with HF etching for much shorter time periods.

Silica coating, followed by silane application, has also been explored as an alternative to HF etching of feldspathic-based porcelains. The authors of one study found that both the high and low leucite-containing feldspathic porcelains they tested had significantly higher tensile bond strength to composite when samples were first etched with HF followed by silane treatment, vs just silica coating followed by the application of silane. Still another study looking at alternatives to HF etching found that simply roughening porcelain surfaces, followed by treatment with 35% phosphoric acid gel and silane priming, was as effective as HF treated samples to bonded composite. Once again, thermocycling of the samples, which was not done, might have had a significant effect on the results. Other products also exist and are marketed as alternatives to HF etching of porcelain.

In this author’s opinion, while other alternatives do exist, they are less predictable, especially over the long term, when compared with HF-etched porcelain. Numerous in vitro studies clearly demonstrate that etching feldspathic-based porcelain with HF has the potential to significantly increase its bond strength to composite resin. Dental porcelain...
generally consists of an amorphous glassy phase or matrix and a crystalline phase. Treating porcelain with HF tends to selectively dissolve the glassy matrix, resulting in a microscopically porous, high-energy, and microretentive surface\(^\text{12,17,18}\) (Figure 1). In principle, this is similar to what happens to enamel surfaces after etching with phosphoric acid.

Several important questions arise when using HF to etch porcelain. Is there an optimal etching time and concentration of HF that should be used? Can porcelain be over-etched? What is the white "residue" often seen on the surface of the porcelain after etching with HF, and how should it be handled? The first question is not easily answered. This is because the chemical and physical makeup of dental porcelain is variable, depending on the type of ceramic being used. Years ago, Calamia recommended adjusting the etching times and concentration of HF depending on the specific porcelain being treated.\(^\text{19}\) While all feldspathic porcelains contain silica dioxide (SiO\(_2\)), aluminum oxide (Al\(_2\)O\(_3\)), and potassium oxide (K\(_2\)O), the relative amount of each can vary considerably depending on the specific porcelain used. Alumina content, crystalline microstructure, size, and concentration can all influence HF etching times and concentrations.\(^\text{11,20,21}\) As an example, leucite crystals, which affect optical, thermal, and physical properties, are incorporated into the matrix of many dental porcelains.\(^\text{22,23}\) A study by Barghi showed a 50% leucite content porcelain (Fortress\(^\text{TM}\), Chameleon Dental Products, Inc, Kansas City, KS) required a longer etching time (ie, 150 to 180 seconds) with 10% HF when compared with a 27% leucite content ceramic (Ceramco\(^\text{®}\) II, DENTSPLY Ceramco, Burlington, NJ) that only required a 60-second etch with 10% HF.\(^\text{24}\) Some manufacturers recommend very specific HF concentrations and etching times for their ceramics. The manufacturer of one popular pressable ceramic (IPS Empress), which has a reported leucite concentration of 35% to 40%, recommends etching for 60 seconds with 5% HF.\(^\text{25,26}\) Longer etching times or higher concentrations of HF with this particular ceramic may actually result in "overetching" and weakening of the porcelain substrate by excessively degrading the surface.\(^\text{26}\) At least one study supports this contention and found that extending the etching time of this particular ceramic’s ingots from 1 to 2 minutes with 5% HF significantly decreased the biaxial flexure strength of the ceramic.\(^\text{27}\) The manufacturer of another popular pressable leucite reinforced ceramic (Authentic\(^\text{®}\), Jensen Industries, Inc, North Haven, CT) recommends "lightly sandblasting the internal surface with 50 µm Al\(_2\)O\(_3\) at a maximum of 20 PSI and then placing 5% to 12% HF for 90 seconds to 5 minutes."\(^\text{28}\) One logical interpretation of this is that dentists could use 12% HF for 90 seconds or 5% HF for 5 minutes. So, one manufacturer (Ivoclar Vivadent, Inc) recommends etching with 5% HF for 1 minute when treating the inner surface of its leucite containing pressable ceramic, and another manufacturer (Jensen Industries, Inc) appears to recommend etching with 5% HF for 5 minutes when treating its leucite containing pressable ceramic. Why there is such a difference in recommended 5% HF application times between these two ceramics is difficult to understand. It is certainly possible that a difference in leucite concentration, size/orientation of crystals, or microstructure might affect etching times. According to one source,\(^\text{29}\) the leucite concentration for the aforementioned popular pressable ceramic (Authentic) is approximately 8%, depending on the shade. This author was unable to corroborate this information despite an extensive Internet search and several phone and e-mail contacts with representatives of the manufacturer. At least one company representative suggested etching the ceramic with 7% HF for 1 to 2 minutes, although it was unclear on what this recommendation was based.\(^\text{30}\)

Two lithium disilicate-based ceramics (IPS e.max\(^\text{®}\) CAD, IPS e.max\(^\text{®}\) Press, Ivoclar Vivadent, Inc) replaced the manufacturer’s now defunct product (IPS Empress\(^\text{®}\) II).\(^\text{26}\)
manufacturer of these products recommends a very specific etching time of only 20 seconds with 5% HF;25,26 and studies exist supporting this protocol.17,31 Powder/liquid or “stackable” porcelains (Ceramco® 3 and Duceram® Plus, DENTSPLY Ceramco; Omega® 900 and VMK-68, Vident, Brea, CA; EX-3, Noritake Dental Supply Co, Ltd, Aichi, Japan; IPS d.SIGN®, Ivoclar Vivadent, Inc; Creation®, Jensen Industries, Inc) are used extensively in the fabrication of ceramic restorations. Some opinion leaders/studies recommend 90 seconds of etching with 10% HF for stackable feldspathic porcelains,32 while others recommend a 120- to 150-second etch with 9.5% HF.33 The manufacturers of two popular 9.5% HF etching gels (HF etching gel, Bisco, Inc, Schaumburg, IL; HF etching gel, Ultradent Products, Inc, South Jordan, UT) recommend etching times of 90 seconds (ie, the former manufacturer) and 60 seconds (ie, the latter manufacturer).34

In an unpublished study, Byoung Suh tested 10 different feldspathic porcelains (Synspar® and OPC®, Pentron Clinical Technologies, LLC, Wallingford, CT; IPS d.SIGN; CZR Press, Noritake Dental Supply Co, Ltd; Vintage™, Shofu Dental Corp, San Marcos, CA; Vita®, Vident; Initial, GC America, Inc, Alsip, IL; Ceramco® II and Duracem®, DENTSPLY Ceramco; and Creation, Jensen Industries, Inc) by HF etching (ie, 9.5% and 4%), followed by contact angle analysis and microscopic evaluation. He first particle-abraded the samples (ie, sandblasting) and found they all showed an acceptable microscopic etching pattern after a 4- to 5-minute etch with 4% HF. These same porcelains showed a similar etching pattern when 9.5% HF was used for 90 seconds.35 The authors of another study found statistically higher composite shear bond strength to several stackable porcelain disks when they were etched with 10% HF for 2 minutes as opposed to a longer etch of 4 minutes at the same HF concentration. They attributed the lower bond strength with the longer etching time of 4 minutes to “overetching” and actual weakening of the porcelain surface.36 The manufacturer of a popular laboratory ceramic etching gel sells a 12% to 13% HF etching gel for stackable porcelains (Etch-It Kit, American Dental Supply, Inc, Allentown, PA) and recommends what, to this author, appears to be a rather long etching time of 10 to 15 minutes. According to the manufacturer, this application time was based on observation of the contact angle of water droplets placed on ceramic surfaces and the microscopic appearance of the porcelain surface after etching for different time periods (no shear or tensile bond testing was performed).37 Interestingly, this same manufacturer sells another product marketed for pressed ceramics (Etch-It Kit for pressable porcelain, American Dental Supply, Inc) that uses 5% HF with a recommended etching time of only 30 to 60 seconds.

Most dentists and ceramists have noticed that a white “residue” sometimes remains on the surface of HF-etched porcelain restorations. At times this residue can be quite extensive and cover virtually the entire etched porcelain surface. The extent of this precipitate is no doubt related to the type of porcelain, concentration of HF, and the time of application. One study, through energy dispersive spectrometer analysis, determined this residue to consist of the reaction products of porcelain and HF.38 Basic chemistry has shown that when an acid and base react, various salts are produced as byproducts of the reaction. In the case of HF etching, the porcelain acts as a base, and when it reacts with HF, various insoluble metallic salts are formed.31 In this author’s opinion, the white residue consists not merely of “porcelain salts,” but also of numerous microscopic crystalline fragments exposed, possibly weakened by HF exposure, and displaced after the glassy matrix supporting them has been dissolved by HF and the porcelain surface washed and dried. This water insoluble “crystalline residue/salt mix” rather tenaciously

Figure 2 In this example, the intaglio surface of a porcelain veneer was purposely overetched by the author with 9.5% HF placed for 15 minutes. Much of the clearly visible “white residue,” a combination of porcelain salts and porcelain debris, could not be removed even after an aggressive air/water spray for 2 minutes and wiping with cotton pellets soaked in acetone.
adheres to the porcelain surface and is often difficult to remove. While many believe this white “frosting” to be indicative of a “good etch” of the porcelain, this is probably an inaccurate assessment. In fact, in this author’s opinion, if the white precipitate is excessive, this may be indicative of overetched porcelain (Figure 2). Several authors have discussed the potential for overetching porcelain and how this could adversely affect porcelain physical properties and/or bond strength to composite. In any case, this surface residue is a potential contaminant, and it makes sense to remove it before proceeding to the bonding phase of treatment.

In this author’s experience, removal of this material with water alone is usually not effective, even after a vigorous air/water spray. Acetone or alcohol rubbing is only slightly more effective. This author has found that placing the etched restorations in an ethanol solution followed by ultrasonication for 5 minutes is usually, but not always, effective in removing any residue (Figure 3A through Figure 3C). Sometimes light brushing with a toothbrush and/or longer ultrasonication times are also required. Other authors have recommended a similar protocol using distilled water. This author prefers an alcohol solution because it evaporates more easily than water and is probably a better solvent when it comes to removing potential contaminates (eg, grease and oils from handling, surfactants from acid gels, saliva, try-in paste residue, etc). Other authors have recommended the use of steam cleaning to remove any residue/salts left after HF etching of porcelain.

In dentistry, the silane most often used in dentistry is the 3-methacryloyloxypropyltrimethoxysilane, which is a difunctional molecule. The left side of this molecule is nothing more than a methacrylate group capable of copolymerization with methacrylate-based adhesives and resins routinely used for dental procedures. The right side, after hydrolysis, has the potential to form chemical bonds to the porcelain surface.

The silane most often used in dentistry is the 3-methacryloyloxypropyltrimethoxysilane, which is a difunctional molecule. The left side of this molecule is nothing more than a methacrylate group capable of copolymerization with methacrylate-based adhesives and resins routinely used for dental procedures. The right side, after hydrolysis, has the potential to form chemical bonds to the porcelain surface.

In dentistry, silane is potentially useful as a chemical coupler linking organics (ie, resin-based materials) to inorganics (eg, porcelain, some oxidized metals, and glass fillers in resin-based composites). A number of theories—ranging from its ability to simply act as a good wetting agent to the actual formation of covalent chemical bonds at the involved
interfaces—exist regarding how silane actually functions.44-46 (This author highly recommends an excellent paper on silane chemistry and interactions written by Meyer Rosen in 1978 while working for Union Carbide.49) Infrared spectroscopy has shown that silane has the potential to react with hydroxyl (-OH) groups present on the surface of porcelain and metal substrates.46-48 To be able to function as a coupling agent and interact chemically with porcelain surfaces, silane must first be hydrolyzed.45 Acetic acid is commonly used to “activate” or hydrolyze silane by reacting with the three methoxy (-OCH$_3$) groups located at one end of the silane molecule (Figure 4). Ideally, all three of the terminal methoxy groups will convert to -OH groups, although only one or two may actually react, resulting in incomplete hydrolysis and a potentially less effective silane.46,47

Single-bottle silanes are prehydrolyzed by the manufacturer and, while variations in chemistry exist, typically consist of 1% to 5% silane in a water/ethanol solution with an acetic acid adjusted pH of 4 to 5.44,45 Once hydrolyzed, silane molecules have a tendency to react with one another, forming high-molecular-weight oligomers44,47 (ie, polysiloxanes) that can actually function as a lubricant49-51 and potentially decrease bond strength to porcelain. It is important to remember that one-bottle prehydrolyzed silane solutions have a limited shelf life, and this author recommends refrigerated storage and replacement after 1 year, as well as bringing refrigerated silane to room temperature before use. If the silane solution appears at all cloudy or “milky” in appearance, or any type of precipitate is noticed, it should be discarded.

Two-bottle silane systems typically consist of an unhydrolyzed silane/ethanol solution in one container and an acetic acid/water solution in the other.35 These are mixed together by the clinician to hydrolyze the silane before application. It is unclear to this author precisely how much time is required for an acceptable degree of hydrolysis to occur once the solutions of two-part silane systems are mixed. The authors of one paper state “0.5 to 2 hours should be sufficient,”45 while another author cites a study showing hydrolysis was “complete 10 minutes after mixing with 0.1% acetic acid.”44 The authors of another study determined the hydrolysis rate of silane by the use of Raman spectroscopy and determined that 30 to 50 minutes was required.52 Still another study found that the two-part system (Silanit, Ivoclar Vivadent, Inc) they tested did not have the same degree of hydrolysis 20 minutes after activation when compared with the two prehydrolyzed systems they tested.47 One reputable manufacturer and respected researcher stated to this author that, based on contact angle analysis and in-house testing, its two-part silane was effective immediately after mixing, but performed optimally if left alone on the porcelain surface for 5 minutes after it was applied.35 This is probably an indication of further hydrolysis of the silane and/or increased chemical interaction with the porcelain during the waiting period. Two-bottle systems should have a longer shelf life than premixed systems and might be a better choice for dentists whose use of silane is infrequent.

The important point to remember is that the goal of silane hydrolysis is to create terminal hydroxyl groups on each silane molecule. These hydroxyl groups are then capable of reacting directly with corresponding hydroxyl groups present on the surface of feldspathic porcelain (through oxidation of SiO$_2$). The opposing hydroxyl groups interact with one another (via hydrogen bonding) and then, through a condensation polymerization (ie, loss of water) reaction, covalent bonds are formed between the silane and porcelain. Not only does the silane bond directly to the porcelain in this fashion, but also the individual silane molecules bond to one another, forming a polymer “network” on the porcelain surface44 (Figure 5 and Figure 6). In theory, the ideal situation would be one in which a “monolayer” of silane molecules lines the surface of the porcelain.45 Clinically,
this is not possible, but it still makes sense to use the minimum amount of silane required, and this author recommends that no more than one to two coats be placed. Excessive application of silane could, in principle, create too thick a coating by consecutive silane layers bonding to each other, one on top of another, and creating an unnecessarily thick and intrinsically weak layer, which could be prone to cohesive failure. Clinically, the surface of the porcelain should NOT look shiny after silane application and drying. In this author's opinion, a shiny surface on the porcelain after silane application and drying could be an indication of excessive silane deposition and, if seen, the surface should be sandblasted under low pressure, reetched with HF, cleaned with ethanol in an ultrasonic, and the silane reapplied. A properly silane-treated porcelain veneer visually appears essentially the same as it did before placing the silane (ie, matte/dull finish). Numerous studies have shown that heat treatment of silane-treated porcelain has the potential to significantly improve bond strength to composite.41,51,53-56 This author recommends that warm-air drying be used for 60 seconds to dry the surface of the porcelain after silane application. This is a simple and clinically feasible chairside procedure that has been shown in some studies to significantly improve bond strength of silane-treated porcelain to composite.51,54,55 Complete removal of water is critical as it is the basis behind the condensation polymerization reactions that allow silane to actually bond to porcelain. Warm/dry air is simply very effective at removing alcohol and water from the surface, and by “heating up” the substrate, one can speculate that the reaction rate will be accelerated, molecular interactions become more frequent, and the formation of chemical bonds occurs in greater numbers. Once the inorganic end of the silane molecule is bonded to the porcelain, the methacrylate
group on the other end is free to bond (via free radical addition polymerization) with methacrylate groups in resin-based dental materials and adhesives (Figure 6). While there is equivocation in the literature, it appears that most studies find proper use of silane significantly improves the bond strength of resin-based composites to porcelain. 

**BONDING TO PORCELAIN: HIGH-CONTENT ALUMINA AND ZIRCONIA CORE-BASED CERAMICS**

High-content alumina and zirconia core-based ceramics (In-Ceram®, Vident; Procera® All-Ceram, Nobel Biocare USA, LLC, Yorba Linda, CA; LAVA™, 3M ESPE; WöI-Ceram®, WöI Dental-Technik GmbH, Ludwigsafen, Germany; Cercon®, DENTSPLY Ceramco; CEREC® InLab, Sirona Dental Systems USA, Charlotte, NC; Everest®, KaVo Dental Corp, Lake Zurich, IL) are highly resistant to chemical attack from HF and a different approach is required if the clinician elects to bond these restorations into place using resin-based adhesives and luting cements (as opposed to conventional cementation). One method that has been shown to be quite effective in increasing bond strength to these materials is the technique of silica coating followed by silane application. One simple and effective technique uses silica-coated 30-µm aluminum oxide particles (CoJet™ Sand, 3M ESPE) followed by the application of silane. According to the manufacturer, sandblasting with this material uses “impact energy to apply a silica coating to the target surface.” Whether this transfer of silica is caused by particles actually becoming embedded in the target surface, actual mechanical/chemical transference (ie, tribochemistry), or both, is unclear to this author. In this author’s opinion, it is unlikely that, in the case of high alumina or zirconia cores, there is actual imbedding of silica-coated particles because of the intrinsic hardness of the target material. It may be more feasible that the silica-coated particles actually “bounce off” these ceramics, but before doing so there is an actual transference of silica from the particles to the target substrate (ie, tribochemistry). In any case, this technique has proven to be effective (more so than conventional sandblasting) not just with high-strength alumina and zirconia-based ceramics, but also when bonding to composite and metal surfaces. One can speculate that by silica-coating metal or composite substrates, not only is the surface mechanically roughened, but the number of available hydroxyl groups for surface silane coupling is significantly increased. Silica coating is not effective, or required, with conventional feldspathic porcelains simply because significant amounts of SiO₂ and free hydroxyl groups are already present (much more so than metals and composites). If the dentist opts to conventionally cement zirconia or high alumina core-based crowns/bridges, silane is not required. However, this author still recommends briefly sandblasting the inner surface of the restoration under low pressure (ie, approximately 20 PSI), followed by placement with whatever conventional cement is chosen by the dentist. (This author prefers resin-modified glass ionomer cements [RelyX™ Luting Plus, 3M ESPE; FujiCEM, GC America, Inc]). Care should be taken when sandblasting these restorations, making sure to use low pressure for short time periods, as at least one study has found that sandblasting with 50-µm Al₂O₃ (at 40 PSI) had the potential to induce flaws in dense alumina and zirconia ceramic plates, resulting in decreased physical properties.

**UNDERSTANDING ALL OF THIS**

After a fairly extensive literature review, numerous discussions with manufacturers, laboratory technicians, and researchers, it is apparent to this author that no single specific HF concentration and application time exists that is optimal for etching all porcelains. As a consequence, it is not surprising that currently recommended HF concentrations and application times vary significantly. It also appears that there is potential to both underetch and overetch porcelain. As Calamia suggested years ago, HF concentrations and application times should, ideally, be adjusted depending on the specific nature of the porcelain being treated. In this regard, dentists are somewhat at the mercy of manufacturers, and it should be incumbent on them to provide accurate and scientifically supported recommendations on precisely how to optimally treat and place their specific product(s). It is then incumbent on dentists and ceramists to actually follow these protocols. As it is, even when specific protocols do exist, this author suspects they all too often are ignored, not understood, or dentists and ceramists are simply not aware of them. In regards to etching porcelain, one goal should be to determine the time frame required, at a very specific HF concentration that will adequately etch various porcelains without excessively degrading and/or weakening the substrate.
In any case, while recognizing that exceptions are likely to exist, it is probably safe to make some generalizations regarding porcelain surface treatment.

1. It appears that low-pressure sandblasting, followed by 60 to 120 seconds of etching with 9% to 10% HF, has scientific support and validity when treating conventional powder/liquid stackable porcelains. A variation that also appears effective is sandblasting followed by 4% to 5% HF applied for 4 to 5 minutes.

2. In the case of a popular pressable ceramic (IPS Empress), the recommended treatment protocol of 5% HF applied for 60 seconds has scientific support and should be followed. The manufacturer of this particular product is one of the few that this author found to have very specific recommendations, as far as HF concentrations and application times, for a number of its bondable ceramics. Specific manufacturer recommendations for many of the other pressable and/or high leucite containing ceramics are vague, do not exist, or could not be found and/or verified by this author. Some ceramic manufacturers shift responsibility by simply recommending following the directions of the manufacturer of whatever HF-etching gel or liquid is being used. As a result, specific treatment protocols appear to be largely speculative. Perhaps these are the restorations many ceramists stated (to this author) that they simply etch until the “surface looks frosty.” While this is certainly not very scientific, and likely not optimal, in the absence of specific guidelines, it may be the best ceramists and dentists have in some situations.

3. High-strength alumina and zirconia core-based crowns cannot be etched with HF. As a result, it is not possible to “bond” these restorations with conventional HF/silane treatment. Silica coating, followed by silane application, has been shown to be a viable alternative.

4. Proper use of hydrolyzed silane, in conjunction with warm-air drying, has scientific support and is advisable after HF etching of feldspathic porcelains or silica coating of high alumina, zirconia, metal, and composite surfaces.

**TREATMENT PROTOCOLS**

Two general treatment protocols successfully used by the author for surface treatment of stacked (powder/liquid) porcelain veneers before placement are presented below. These protocols may not be valid for all stacked porcelains and when available, manufacturer recommendations regarding etching times and HF concentrations should be followed. The first protocol assumes that the veneers are not etched at the dental laboratory. The advantage of this protocol is that the dentist has complete control over the surface preparation. In the second protocol, the veneers have been etched at the dental laboratory. This requires a certain degree of faith in the laboratory technician and assumes he or she has properly etched the porcelain. The author recognizes other protocols exist, are valid, and are clinically successful.

**Protocol 1—Veneers NOT Etched by the Dental Laboratory**

1. Try in case for fit and esthetics. Once the decision to place the veneers is made, proceed to Step 2.
2. Briefly sandblast veneers under low pressure (ie, 20 PSI) with 50-µm Al₂O₃ or 30-µm aluminum oxide particles.
3. Etch with 9% to 10% HF gel for 90 seconds, wash, and dry.
4. Ultrasonicate for 5 minutes in ethanol; dry well.
5. Apply one to two coats of prehydrolyzed silane and warm-air dry for 60 seconds.
6. Fill veneers with a light-cure only resin veneer cement (RelyX™ Veneer Cement, 3M ESPE; Choice™ 2, Bis- co, Inc) and bond in place. Veneer cements that have a dual-cure capacity contain tertiary aromatic amines that may discolor with time, potentially altering the shade of the veneers.⁷¹-⁷³

**Protocol 2—Veneers Etched by the Dental Laboratory**

1. After receiving the veneers from the dental laboratory, inspect the inner surface. Properly etched veneers should have a dull/matt finish and no shiny areas. If the veneers have shiny areas, briefly sandblast and reetch with 9% to 10% HF for 90 seconds. If the veneers appear well etched, proceed to Step 2.
2. Ultrasonicate for 5 minutes in ethanol; dry well. (Note: an alternative to using an ultrasonic is to clean the veneers with phosphoric acid, wash, and dry.) The basic idea is to ensure a clean, fresh, high-energy surface before
applying silane, as it may be possible the veneers were contaminated from normal handling and exposure at some point between the laboratory and the time of placement.

3. Before the veneers are tried in, apply one to two coats of prehydrolyzed silane and warm-air dry for 60 seconds.

4. Try in veneers to access fit and esthetics. (Note: this author rarely uses try-in pastes.) After approval, place the veneers back in the ultrasonic to remove any surface contaminates. Silane does not need to be reapplied.

5. The cleaned veneers should be dried thoroughly and brushed with a thin coat of HEMA-free bonding resin. (Note: See Step 6, Protocol 1.)

6. Perform the same as Step 7, Protocol 1.

An acceptable variation in this protocol is to try in the veneers before cleaning with phosphoric acid, ultrasonication, and application of silane.

CONCLUSION
Optimizing porcelain surfaces before treatment with various adhesives and luting resins requires an understanding of the involved substrates and materials, as well as a logical and systematic methodology in their manipulation. It is clear that while much is known, even more remains to be learned. It is hoped that this article has answered some questions, raised others, and provided greater insight into the nature of bonding to porcelain.

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1. Hydrofluoric acid (HF) is:
   a. a strong organic acid.
   b. a relatively weak organic acid.
   c. a strong inorganic acid.
   d. a relatively weak inorganic acid.

2. Because of its low dissociation potential, HF:
   a. has the ability to readily penetrate skin tissues.
   b. can cause extensive internal tissue damage.
   c. has caused death from accidental skin exposure.
   d. all of the above

3. Acidulated phosphate fluoride gel (APF) has been shown:
   a. to have the ability to etch porcelain.
   b. to require considerably shorter application times than HF etching.
   c. to result in a rougher-appearing surface than HF etching.
   d. all of the above

4. All feldspathic based porcelains contain:
   a. silica dioxide (SiO₂).
   b. aluminum oxide (Al₂O₃).
   c. potassium oxide (K₂O).
   d. all of the above

5. The manufacturer of the pressable ceramic IPS Empress recommends an etching protocol using:
   a. 5% HF for 60 seconds.
   b. 5% HF for 2 minutes.
   c. 9% HF for 60 seconds.
   d. 9% HF for 2 minutes.

6. Some opinion leaders/studies recommend an etching protocol for powder/liquid or “stackable” porcelains of:
   a. 4 minutes with 10% HF or 5 minutes with 9.5% HF.
   b. 2 minutes with 10% HF or 2.5 minutes with 9.5% HF.
   c. 90 seconds with 10% HF or 120 to 150 seconds with 9.5% HF.
   d. 60 seconds with 10% HF or 90 to 120 seconds with 9.5% HF.

7. To remove the white “residue” from an etched porcelain surface, the author has found what protocol usually, but not always, effective?
   a. rinsing the etched restoration with water followed by a vigorous air spray
   b. placing the etched restoration in an ethanol solution followed by ultrasonication for 5 minutes
   c. scrubbing the etched restoration with alcohol followed by air drying
   d. all of the above

8. Silanes are:
   a. a class of organic molecules that contain one or more silicon atoms.
   b. potentially useful as chemical couplers linking organics to inorganics.
   c. able to interact chemically with porcelain surfaces only if they are hydrolyzed before use.
   d. all of the above

9. The terminal hydroxyl groups on each silane molecule interact with the opposing hydroxyl groups on the surface of feldspathic porcelain through:
   a. hydrogen bonding.
   b. anionic addition polymerization.
   c. formation of ionic bonds.
   d. formation of aromatic bonds.

10. One method that has been shown to be quite effective in increasing bond strength to high-content alumina and zirconia core-based ceramics is:
    a. etching with 9% to 10% HF.
    b. silica coating followed by silane application.
    c. ultrasonic brushing.
    d. applying 1.23% APF.

Please see tester form on page xxx.

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