COMPARING THE PATTERNS OF ENAMEL ETCHING BY TWO IRANIAN ACID-ETCHES AND ONE FOREIGN STANDARD ACID-ETCH IN VITRO

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Abstract. Introduction: One of the main steps in composite restorations is enamel etching to create a strong bond between composite resin and dental surfaces and prepare these surfaces. This increases the bonding surface and the grip of composite resins, and improves the margin bonding of restorations. Given the fact that today there are various acid-etches with different features in the dental market, the answer to this question that which one of the different types of acid-etches used provides better etching pattern and greater bond strength in development of composite resin adhesion to dental surface compared to others is uncertain, therefore, we decided to conducted a study with the aim of comparing the patterns of enamel etching by two Iranian acid-etches and one foreign standard acid-etch in Vitro. **Materials and Methods:** In this study, 20 extracted human maxillary premolars that were free of decays and fractures were selected, and after cleaning them with Pumice powder, they were stored in 0.2% Thymol solution at room temperature. Prior to acid-etching, the teeth were divided into two groups of 10. The labial surfaces of teeth were polished, and then, with a nail polish, the labial surface of each tooth was divided into two distinct mesial and distal parts. In one group, the mesial part of each tooth was etched by standard acid (Ultradent) and the distal part of each tooth was etched by an Iranian acid (Kimia), and in other group, it was etched by another Iranian acid (MorvaBon). Both groups were etched according to the manufacturer's instructions. After washing the acids for 20 seconds, and also drying them up for 20 seconds, the etching patterns developed in the SEM study center was investigated and compared. **Findings:** The etched enamel surfaces by two type of acids showed corrosion around the prisms and no coverage in the center of prisms (Type 2 pattern) in the middle 1/3 and incisal 1/3, however, the cervical 1/3 was developed in most samples of type 3 pattern. In some areas, the Iranian acid also did not create a pattern associated with the shape of the prisms. One of the morphological differences observed in regions etched with the foreign acid in comparison to the Iranian acid was the better order and arrangement of etching pattern induced by the foreign acid. Another difference was the formation of more sediments in the sections etched by the Iranian acid, which may reduce the resin grip to enamel, and the last difference was more corrosion of foreign acid measured by an image analysis machine.
Discussion and conclusion: In present SEM study, the corrosion from the Iranian acid was visually more than foreign acid, thus, it is necessary to study the chemical composition of Iranian acid and make necessary amendments. Also, studies on the bond strength of resin to enamel and the degree of microleakage in applications of Iranian acid seems necessary.
Keywords: Acid etch, Etching pattern, Enamel

Introduction

The beauty if one of the things that is very important for patients. Dentists, by utilizing the advances made in the field of tooth-colored restorations and using conservative techniques, in addition to dental restoration, also provide beauty (1). Despite the expansion of the use of resin composites and the development of adhesive systems, microleakage is still considered as one of the most important problems in these restorations. This problem occurs as a result of many factors such as the inability of available bonding materials, improper etching, poor physical properties of composites, or a set of these factors (2, 3). Using acid for bonding the composite resin to the tooth structure is one of the most sensitive steps in this procedure. The shape formed by the acid depends to a large extent on type of acid, which depending on the fixed time use of acid or the quality of the acid used, it can produce useful or inappropriate microscopic patterns (4.5). Preparation of these surfaces is necessary to create a strong bond between composite resin and dental surfaces (6). The acid-etching method by phosphoric acid, introduced by Bonocoure in 1955, is used to create an irregular surface on a beveled enamel. This increases the bonding surface and the grip of composite resins, and also improves the margin bonding of restorations (7). In this method, in fact, using mineral acids, a surface layer with a thickness of 0.5 to 5 microns is changed or completely dissolved in the scraped surface of tooth, and by creating an irregular surface, the contact and reaction between teeth and bonding materials can be achieved. Acid-etching changes the surface of the enamel to a very irregular and rough surface, increases its surface energy, and when a resin-based material with a liquid property is placed on the irregular etched surface, the resin penetrates into the surface and this penetration is intensified by the capillary process (10). Therefore, the formation of microscopic elongations of resin within the surface of the enamel is the basis to the process of resin bonding to enamel. In the normal method, 37% phosphoric acid in the form of a gel or solution is placed on the enamel surface for 30 seconds, and then the adhesives enter into the porosities formed by phosphoric acid, and ultimately, the micromechanical

grip is developed (10). Three types of etching patterns are described after the exposure of enamel prisms to acid-etching solutions: in the type I pattern, the center of the enamel prisms is removed and their surroundings remains intact. In type II pattern, prism surroundings are removed, and the center of the prisms remains relatively intact. Type III pattern is an accidental etching pattern that may be seen in some areas of the type I and II patterns together, and in some areas, a pattern can be created without any association with the shape of the prisms (9). On the other hand, factors such as the type of acid-etching solution, concentration of solution, techniques used, and placing time of acid are effective on the effects of acid-etching on the enamel (11). Application of acid-etching and bonding is one of the most important factors in increasing the bond strength and ultimately, the success of composite resin restorations. By performing the acid-etching process, the hydroxyapatite crystals are dissolved in the surface and the fluid components penetrate into the newly created surface irregularities. The abrasion process also results in massive surface roughnesses, and creates a smear layer from reshaped collagen and hydroxyapatite crystals that has a thickness of about 1-3 microns and should be adhesive to the substrate. Therefore, it is necessary that either this layer is removed or the adhesive material penetrates into it. At the same time, etch by using acid or conditioner removes this layer and creates a surface without smear layer and with negative grip points to provide an opportunity for a micro-mechanical grip. When a resin-based substance with fluidity is placed on the irregular acid-etched surfaces, the resin penetrates into the surface and at the same time, this penetration is exacerbated by the capillary process. Then, the monomers in this material are polymerized and the material is locked in the enamel surfaces. As a result, the use of the preparation method through the use of phosphoric acid with adhesive resin is effective on the bonding to enamel (10, 12, 13). A study by Ibrahim et al. (2010) was conducted to investigate the effect of preparation of enamel through the use of phosphoric acid and EDTA on the bond strength of mild, intermediate and strong self-etch adhesive systems. The results of this study showed that preparation with phosphoric acid causes more deepening of the etching pattern in strong and medium adhesive systems similarly, however, in the self-etch adhesive system, this process changed the irregular etching patterns to regular etching patterns. SEM observations from the resin/enamel region also showed that more deepening of etching patterns was consistent with the length of resin tags. Also, preparation through EDTA showed limited effects on the morphological properties of samples. Based on this, the use of the preparation method through the application of phosphoric acid with a mild adhesive system has been effective on the bonding to enamel (13). Given the fact that today there are various acid-etches with different features in the dental market, the answer to this question that which one of the different types of acid etches used provides better etching pattern and greater bond strength in development of composite resin adhesion to dental surface compared to others is uncertain, therefore, we decided to conducted a study with the aim of comparing the patterns of enamel etching by two Iranian acid-etches and one foreign standard acid-etch in Vitro.

Materials and Methods

In this experimental and laboratory study, 20 extracted human maxillary premolars with no decay, fracture-crack, erosion and hypoplastic defects were selected and enrolled in the study. Teeth were stored in 0.2% thymol solution. The teeth were removed from the thymol solution before the beginning of the study, and after being thoroughly cleaned by a scalpel, pumice solution and brush, they were kept in the physiological serum at the room temperature. At the beginning of the procedure, after removing the teeth from the physiological serum, the labial surface of enamel of each premolar was one-way fresh by a medium diamond burs, then each sample was individually put under abrasion by silicon carbide (SiC) paper discs and then micronized silicon carbide powders to achieve a smooth surface for the SEM study. In the next step, the crown region of each tooth towards the root was completely covered by the nail polish, and both mesial and distal regions of labial surface of each tooth were also separated and identified. Then, the teeth were divided into 2 groups of 10. In the first group, the foreign acid (Ultradent) was placed at the mesial part and the Iranian acid (Kimia) at the distal part of each tooth simultaneously according to the manufacturer's instructions, and then, acids were washed simultaneously for 20 seconds and also dried for 20 seconds. In the second group, steps were done similar to these steps only with the difference in the Iranian acid of MorvaBon at the distal, and then acids were washed for 20 seconds and also dried for 20 seconds. The etched teeth were placed in a sealed box and send to the SEM study center. In the SEM study center, the samples were placed in a silica gel desiccator for dehydration. Then, they were coated with gold by Sputter Coater machine for 60 seconds and a current of 18 mA, and they were prepared for the evaluation by SEM device (Cambridge Stereoscan 360 SEM, with a maximum magnification of 3000000) under a voltage of 10 KV and using secondary electron detectors (for morphological evaluation of surface and microstructure) and

backscattere electron (for separation of phases and based on the contrast difference of atomic number). The SEM study was conducted to examine the patterns created by the domestic acid and to compare it with internationally standard accepted and approved patterns. The method and tools for collecting data and the method for determining its validity and reliability were done through SEM images, and finally, for the statistical analyzes of the study, giving the qualitative nature of studying the teeth, similar studies in this field were used to determine the number of study samples, and examination of images and determination of etching patters were carried out by a restorative specialist.

Findings

Based on similar studies, images were taken from the sample, which was etched for 30 seconds by the foreign acid (Ultradent) at the mesial region and by the Iranian acid (Kimia) at the distal region, by the SEM device at magnification of 700 and 1000 from the regions where more pronounce etching patterns were developed. The result was that the Ultradent acid, both at a magnification of 700 and at 1000 magnifications, had created a typical type II pattern, meaning that the prisms had been corroded from their peripheral parts, and their central regions were almost healthy and intact. However, in the case of Kimia acid, the appearance created was very irregular and no specified standard pattern was developed, and in the electron microscopic images, most of the lines were caused by the freshening of the enamel by diamond discs, not from the corrosion of the prisms. Therefore, it was decided that in the study of the main samples, before the SEM study and after the fresh-up of the enamel, the teeth should be polished to achieve a roughly uniform surface for the SEM study, and we would be able to study the etching patterns at higher magnifications.

In the study of first group (10 teeth with Iranian acid-etching of Kimia and standard acid-etching of Ultradent), these samples were first examined at low magnification and then we increased the magnification to obtain a clear and distinct pattern from enamel etching. Then, the area was examined at several other magnifications, and the photos were taken from them. The magnifications used for such studies according to the references are 1500, 2000, 3000 and 5000. In this study, photos were preferably taken at magnifications of 900 and 2000, and sometimes 3000.

In the first group, the results showed that the first thing seen at a magnification of 900 is a specific order that was in patterns developed by the foreign acid of Ultradent, however, this order and arrangement was far less regular in etching patterns induced by the Iranian acid of Kimia and the placement of etched prisms seemed scattered and irregular. The etching patterns developed by both types of acid was type II pattern, meaning that peripheral parts of prisms were removed and their central parts were relatively untouched. At magnifications of 2000 and 3000, the differences evident in the created surfaces were deposits, which were visible in the distal region, the site of Iranian acid etching, which apparently are the same products resulted from acid corrosion, and seen in the form of glass-like surface around the etched prisms, in the dark areas of the photos. However, in the mesial part, the site of foreign acid-etching, these deposits were much less or even nonexistent. Another difference was seen in the corrosion from the Iranian acid compared to that of the foreign acid. Since the darks spots, indication of corrosion and penetration of acid, in the images of the Iranian acid, in addition to peripheral section of prisms, were also observed in many cases in the form of black spots in the center of prisms. The corrosion from the Iranian acid seemed deeper and more severe in the prism surroundings. Because these areas created more dark areas around the prisms, and therefore, the prisms seemed to be longer. However, it can be said that in most regions, especially incisal 1/3 and middle 1/3, the etching patterns caused by both acids were of type II patterns, but in areas close to 1/3 cervical, no specific pattern was observed and only corrosion products were observed. In the examination of second group (10 teeth with Iranian acid-etch of MorvaBon and standard acid-etch of Ultradent), similar to the previous group, these 10 samples were examined at magnifications of 900, 2000 and 3000. At a magnification of 900, an overall image from the pattern developed by both types of acid was obtained, and then, more details were revealed at magnifications of 2000 and 3000. In the studies conducted, it was found that in this group, as in the first group, the dominant pattern developed was type II pattern, and periphery of prisms was corroded and the central parts were relatively intact. Although, in these samples, the pattern developed by the foreign acid had also more order and showed a unified form of etching of enamel prisms, but it can be said that, as in the first group, there was not a clear difference in the arrangement of prisms between the patterns developed by the foreign acid and the domestic acid. That is, the Iranian acid of MoraBon had created a more regular pattern than the Iranian acid of Kimia, which in some samples, this regularity was similar to that of the foreign acid. There was more sediments and products from corrosion in the images of the Iranian acid compared to the images of the foreign acid. Also, the peripheral regions of the prisms

showed a deeper corrosion in the parts related to the Iranian acid-etching.

Discussion and conclusion

The use of acid for bonding of resin composite to the tooth structure is one of the most sensitive steps in this procedure. The form developed from the acid depends largely on the type of acid, which when used in a fixed time or the quality of acid used can develop useful or useless microscopic patterns (4-5). Preparation of these surfaces is necessary to create a strong bond between composite resin and dental surfaces (6). The method of acid-etching with phosphoric acid, introduced by Bonocoure in 1955, is used to create an irregular surface on the beveled enamel. This increases the bonding surface and the grip of composite resins and also improves the margin bonding of the restorations (7). In this method, in fact, using mineral acids, a surface layer with a thickness of 0.5 to 5 microns is changed on the scraped dental surface or is completely dissolved, and by creating an irregular surface, the contact and reaction between dental tissue and bonding materials are possible (8, 9). In the present study, 20 healthy maxillary premolars, after getting cleaned and polishing of the labial surface, were prepared for the study. The etched teeth were examined in the SEM study center and the results showed that: **1-** the dominant pattern created by both types of acid was type II pattern, that is, the peripheral parts of the prisms were removed, but the central parts seemed almost intact. A significant issue was that there was a specific order in the patterns created by the foreign acid of Ultradent, however, in the etching pattern from the Iranian acids, this order was far less and the orientation of etched prisms was scattered and irregular. **2-** Another difference was in the formation of sediments that were very low or not existence in the etched parts by the foreign acid, however, in the etched parts by the Iranian acids, the products from acid corrosion were clearly seen in most of these areas. The only difference that the second group had in comparison with the first group was that the etching patterns induced by the Iranian acid, which was the MorvaBon acid, had more order, and in some samples, this order was equal to the order of etching patterns induced the foreign acid. There is still debate as why acid etches produce different surface patterns. The most commonly accepted theory is that the etching pattern is dependent on the direction crystals. Studies done with an electron microscope in relation to dissolution of crystals show that they are much easier to dissolve in the end regions than other regions. Therefore, the crystals that are perpendicular to the enamel surface are more suitable for this operation. The type I etching pattern is easily explained by the fact that the crystals of the rods reach the surface of the enamel with different angles than the crystals of the regions around the rod. Similar areas on the contralateral teeth have a roughly identical etching pattern, although regional differences may also have a role in this regard. There is, of course, evidence that the etching pattern also results from the differences in nature of etching agents (5). The study by Kadaka and Koruva (1991) shows that all etching pattern at the adjacent areas are also visible in one tooth (14). In the present SEM study, the predominant pattern in all samples in the 1/3 middle and 1/3 incisal regions was type II etching, but in 1/3 cervical, the type III pattern was observed. Gwinnett (1971) also showed that type III pattern is usually seen in the cervical areas of the teeth. Because in the cervical regions, prisms do not reach the surface of the enamel (15). The reason for the development of type II pattern in this study is probably the perpendicularity of the crystals in the regions between rods on the enamel surface of maxillary premolars, which makes it easier to dissolve these regions than the other parts, and therefore exhibites the type II pattern, meaning the removal of peripheral parts of enamel prisms and no coverage of rod core. In the study of Guba and Cochran, it was shown that liquid and dilute gel produced a more uniform etching pattern than gels, and the diluted gel had the most distinct etching pattern among three types of conditioner (solution, diluted gel, thick gel) (16). In this study, considering that all three Iranian and foreign acids had a concentration of 37% and the study conditions were the same for each sample in both the mesial and distal parts, the regularities of the patterns induced by the foreign acid in both groups are due to the type of the foreign acid, since it is a diluted gel. Another difference is the formation of deposits. Chow and Brown's hypothesis states that the most effective concentration of phosphoric acid should be above 30%. They reported that for phosphoric acid concentrations below 30%, a calcium salt, causes the insoluble precipitation on the enamel, and prevents the access to enlarged small porosities, and thus reduces bond strength (7). In the present study, although the concentration of acid was higher than 30%, that is 37%, but insoluble depositions on the enamel surface were observed in the SEM study. These sediments were found in large numbers in etched sections by the Iranian acid and were found very low in the etched sections by the foreign acid, and in some cases, they did not exist at all. Since these sediments have not been removed at the usual wash time of 20 seconds, they can be considered as dicalcium phosphate dihydrate, because this product is less soluble in clinical condition and, as investigated in previous studies, these products

produced from the corrosion by acid can block the entry of micro-porosities developed by the acid, and prevent the penetration of the resin into the micro-porosities (17, 18). Therefore, the rinsing of Iranian acid from the surface of the enamel should be stronger in comparison with the foreign acid. The other reason for the formation of these deposits may be due to the presence of a thickener used in the chemical structure of Iranian acid, as well as the presence of impurities. In some studies, it has been reported that if phosphoric acid is used with the action of rubbing, the etching pattern would not be clear in the SEM study (15). In a SEM study, it was found that if the mechanical stimulation of etching agent is done during the etch time, the decalcification of the enamel surface increases (6). However, some believe that this does not have much effect on the bonding of enamel to the composite. In the present SEM study, the corrosion of Iranian acid appeared to be visually more than the foreign acid, since more corrosion and dark spots were observed in the surroundings and center of the enamel prisms. Finally, according to the results obtained, it can be said that Iranian acid, due to having mentioned problems, may not be as effective as standard foreign acids, and it is necessary that more laboratory studies are conducted on Iranian acid composition and structure, and the level of loss of the surface as well as the bond strength of composite resin to the surface of teeth etched by the Iranian acid should be compared with the foreign acid, and if there are differences in these areas, modifications should be done on the Iranian acid, so the efficiency and life expectancy of composite restorations done by using common Iranian acid-etchings in the dental market reach a more favorable level.

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