

Evaluation of Effect of Different Enamel Surface Preparation Methods on Shear Bond Strength and Enamel Surface Texture

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Abstract: In an attempt to minimize bond failures, orthodontists have sought out ways to ensure adequate bond strengths while still maintaining an efficient procedure with the least invasive method of bonding. Enamel loss with acid etching ranges from 3-10 μ . These limitations have led to the search for newer, enamel friendly technique for surface preparations. To evaluate & compare the effects of different concentrations of phosphoric acid, self-etch primer and air abrasion on the surface characteristics of enamel and shear bond strength .100 extracted premolar teeth were divided in to 5 groups (20 teeth in each group) and etched with different etchants (Group I -2% H_3PO_4 , Group II-15% H_3PO_4 , Group III -37% H_3PO_4 , Group IV- Sand blasting and Group V- Self etching Primer) .After etching tooth surface was studied under SEM. Then the brackets were bonded and shear bond strength was measured by universal testing machine. Group III (37% phosphoric acid) & Group V(Self-etching primer) showed less destruction to the enamel surface as compared to other groups during SEM study. Maximum SBS was found for group III (13.73 \pm 0.25 MPa) followed by Group II (11.74 \pm 0.27 MPa), Group V (10.29 \pm 0.24 MPa), GroupI (8.31 \pm 0.26 MPa) and Group IV (7.40 \pm 0.26 MPa). 37% phosphoric acid and Self- Etching primer were found more favorable etching agent. 37% phosphoric acid and 15% phosphoric acid can be used for etching the enamel surface to get maximum shear bond strength as compared to other surface treatment methods.

Keywords: Scanning Electron Microscopy, Shear Bond Strength, Acid etching.

INTRODUCTION

Direct bonding of orthodontic brackets using the acid etch technique has eventually become a common technique in the orthodontic field. The acid solution used, act by partially decalcifying the enamel, creating micro irregularities on the surface of the teeth. The minimum damage with the maximum clinically useful bond strength is the most optimal requirement. Shear bond strength of the bonded brackets depends not only on the material and quality of the bracket base and material and method of the bonding, but also on the enamel surface treatment method (Acid etching, Sand blasting or combination, use of self-etch primers). Reduction in etching time and acid concentration that produce optimal bond strength should be strived for. Currently, researches are ongoing to develop time-

conserving and tooth-friendly enamel conditioning system for bracket bonding.

Studies [1] had shown that the use of phosphoric acid can dissolve the enamel prisms to a depth of 5-25 μ m with a diameter of 5-6 μ m allowing for resin penetration with micromechanical bonding. In contemporary practice, 37% orthophosphoric acid gel or solution is applied to enamel for 30 seconds and the minerals present on the enamel surface get dissolved. These demineralized areas, which are present after water rinsing and air drying, do not have uniform depths, and fixed attachments are bonded to the enamel surface by bonding adhesive material to the gaps on the walls or in the center of the hexagonal enamel prisms. Advances in etching and bonding materials, which took place over time with new developments in technology

and clinician demands, have led to increased clinical efficiency. But the varied results, as well as the potential for undesirable excessive loss of enamel from etching techniques and a cumbersome multiple step application process, has motivated clinicians to pursue alternative materials and methods to prepare teeth for bonding. Studies have been carried out to look at shear bond strengths when using alternative materials and methods such as 10% maleic acid, sandblasting, and self-etch primers as means to prepare the enamel for resin bonding.

Under scanning electron microscope unetched tooth surfaces showed transversally oriented, superimposed prisms. These prisms had different orientation which are perpendicular to the surface and appear like small polygonal round holes (resembling honey comb appearance). Krisztina Mártha *et al.* [2] did a comparative SEM study and evaluated the effect of phosphoric acid etching application time on the enamel morphology. The results showed different micro morphological changes depending on the time duration of acid treatment. The etching patterns were divided into 3 types by Silverstone *et al.* [3].

Type 1- Preferential dissolution of the prism cores resulting in a honeycomb appearance

Type 2- Preferential dissolution of the prism peripheries giving a cobblestone appearance

Type 3- A mixture of Type I and Type II patterns.

Galil and Wright [4] included two more types of the etching patterns.

Type 4- Pitted enamel surfaces as well as structures which look like unfinished puzzles, maps or Networks.

Type 5 - Flat, smooth surfaces.

Perikymata had a Type I etching pattern and appeared to etch better than the imbrication lines, which exhibited a Type 4 pattern.

In the present study different concentrations of phosphoric acid, sand blasting and self-etching primer were used for enamel surface preparation to obtain best results in terms of shear bond strength. The etched enamel surfaces were analyzed microscopically by SEM to find out the difference in the surface texture of enamel treated by various surface treatment methods.

Aims and Objectives

- To evaluate the surface characteristics of enamel by scanning electron microscopy (SEM) and shear bond strength of brackets after preparing the enamel surface with different concentrations of phosphoric acid (2%, 15%, 37%), self-etch primer and air abrasion.
- To determine the most appropriate etchant for preparing the enamel surface for bonding.

MATERIALS AND METHODS

125 extracted premolars stored in normal saline [5] after cleaning were included in the study. Buccal surface enamel preparation of 25 teeth (divided in 5 groups) were done by different methods (Group I - 2% H₃PO₄, Group II- 15% H₃PO₄, Group III - 37% H₃PO₄, Group IV- Sand blasting and Group V- Self etching Primer) (Table 1) and microscopic Evaluation of the etched enamel surface was done with a scanning electron microscope at Birbal Sahni Institute of Palaeobotany, Lucknow (Fig 2A). The specimens were viewed in an ISI-100 B scanning electron microscope operated at 20 kV [6].

100 teeth were divided in 5 groups (20 teeth in each group) and their bases were formed by embedding the apical 1/3 of the root in chemically activated resin using the wax blocks as a mould. After cleaning, the buccal surface of teeth in group I, II and III were etched with 2%, 15% and 37% Phosphoric acid respectively for 15 seconds (Fig 1A). The teeth were then rinsed with water spray and dried with an oil free air source until the buccal surface of the etched teeth showed frosted appearance. In Group IV, buccal surface of teeth were air abraded using 50 microns particles size of aluminum oxide abrasive powder at 100 psi, pressure for 15 seconds from a distance of 3mm, which was followed by rinsing with air spray for 10 seconds to remove the excessive aluminum oxide particles (Fig 1B). To prevent unnecessary etching a 4×5 mm aperture was made on 0.040 inch thick thermoplastic retainer and the sandblaster was directed perpendicular (90°) to the enamel surface through this aperture. Group V samples were etched with Self Etch primer for 15 seconds and then air dried to stop the reaction (Fig 1C).

Then after MBT .022 brackets were bonded on all the teeth with Trans bond X using proper bonding technique (Fig 1D). The shear bond strength was measured using an Instron Universal Testing Machine LLOYD LR-100K (Fig 2A-B), Occlusogingival load was applied at a bracket base-resin interface with the cross head speed of 1mm/minute [7]. Until debonding occurred. A sharpened chisel type blade was used to apply the force (Fig 3A-B). The force required for debonding failure was recorded in Newton's and then converted into force per unit area (MPa) dividing the measured force values by the surface area of the bracket (9.93mm²).

RESULTS

Evaluation of etched enamel surface by Scanning Electron Microscopy (Fig 4)

Group I- At 2% phosphoric acid concentration, light prism structures become evident. Additionally, a prismatic surfaces of type 4 and 5 were also visible [3, 4].

Group II- Etching pattern range from very distinct prism delineation (mainly type 2) to aprismatic surfaces (type 4 and 5). The presence of Perikymata is even more evident. There were good etching effects on the crests and worse effects in the grooves.

Group III-Etched surface showed pits measuring approximately 7-8 μ which are semilunar in shape. The etching pattern show preferential dissolution of prism (rod) resulting in cobblestone appearance (type 2). There were also isolated areas without prism structure showing a roughening of the enamel surface (type 3) pattern [3, 4].

Group IV- Air abrasion showed an irregular undulating surface with pits measuring approximately 4-5 μ and the shape was either triangular or square in shape (comparing to type 4 and 5).The enamel removed

was more irregular or the surface had irregularities which were fairly uniform in size[3, 4].

Group V-Etching with self-etching primer revealed that the enamel surface does not show aprismatic structure of enamel rods but spurs (arrow heads) was observed.

Evaluation of Shear Bond Strength as related to different etchants

Table 2 showed the range, mean and median values of Shear Bond strength of each group treated by different enamel surface treatment methods .The following order of shear bond strength was observed among different groups:37% (Group III) > 15% (Group II) >self-etching primer (Group V) >2%(Gp.I)>air abrasion (Group IV). Intergroup comparisons of Shear Bond strength showed statistically significant difference ($p < 0.001$) for all the groups (Table 3).

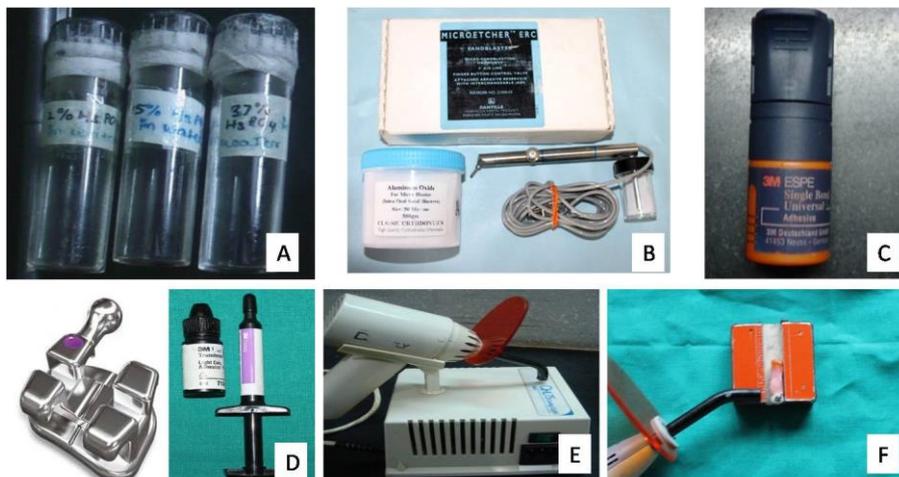


Fig-1: A Different Concentration of Phosphoric Acid (2%, 15%, 37%). Fig-1b: air abrasion (with aluminium oxide). Fig-1c: self-etching primer. Fig-1d: mbt bracket .022 slots and 3m primer and bonding material. Fig-1e: halogen unit. Fig 1f jig made for the accuracy of distance for curing

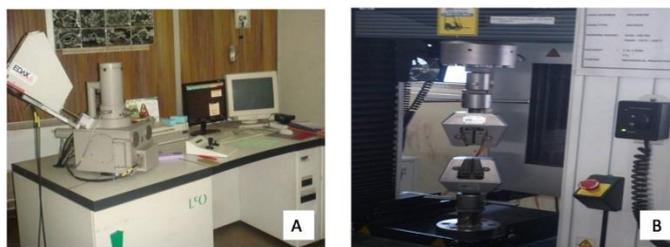


Fig-2: An electron microscope (LEO- CAMBRIDGE, ENGLAND). Fig-2b: universal testing machine

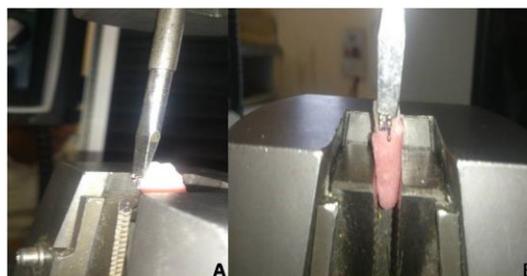


Fig-3: A side view of the sample for shear bond strength. Fig-3b: frontal view of the sample placed for shear bond strength

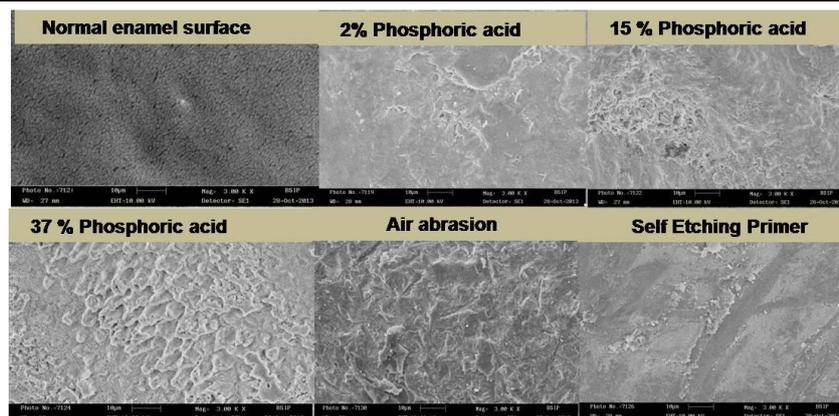


Fig-4: Evaluation of normal and etched enamel surface by SEM at 3000XT.

Table-1: Distribution of sample on the basis of etchants

S.no	Group	No. Of specimens	etchants	Etching time
1.	Group I	20	2% Phosphoric Acid	15 seconds
2.	Group II	20	15% Phosphoric Acid	15seconds
3.	Group III	20	37% Phosphoric Acid	15 seconds
4.	Group IV	20	Air Abrasion	15 seconds
5.	Group V	20	Self- etching primer	15 seconds

Table-2: Shear bond strength obtained for different groups (MPa)

Group	N	Minimum	Maximum	Mean	Std. Deviation	Median
Group I	20	7.87	8.73	8.31	0.26	8.32
Group II	20	11.30	12.16	11.74	0.27	11.78
Group III	20	13.28	14.13	13.73	0.25	13.74
Group IV	20	7.01	7.84	7.40	0.26	7.41
Group V	20	9.88	10.69	10.29	0.24	10.28

Table-3: Intergroup comparisons of Shear Bond strength (Tukey HSD Test)

Comparison	Mean Difference	SE	"p"
I vs II	-3.43	0.08	<0.001
I vs III	-5.42	0.08	<0.001
I vs IV	0.90	0.08	<0.001
I vs V	-1.98	0.08	<0.001
II vs III	-1.99	0.08	<0.001
II vs IV	4.34	0.08	<0.001
II vs V	1.45	0.08	<0.001
III vs IV	6.33	0.08	<0.001
III vs V	3.44	0.08	<0.001
IV vs V	-2.89	0.08	<0.001

Level of significance: "p" is level of significance- $p > 0.05$ = Not significant, $p < 0.05$ = Significant, $p < 0.01$ = highly significant, $p < 0.001$ = Very highly significant

DISCUSSION

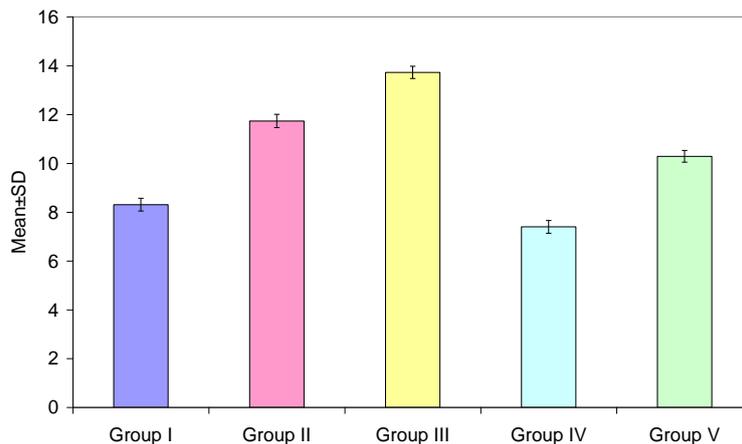
The development of directly bonding orthodontic attachments to dental enamel is among the most significant advances in orthodontics that replaced the long and arduous task of banding all the teeth to be moved during orthodontic treatment. Enamel preparation plays vital role in the strength of bonded orthodontic attachments.

Acid Etching increases the porosity of exposed surfaces through selective dissolution of crystals which provide a better bonding surface for adhesive material.

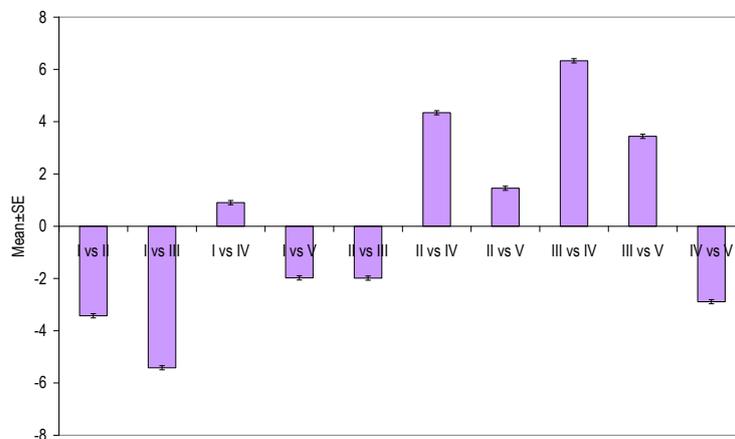
The acid etch technique was introduced by [8]. Various investigators have evaluated the technique to determine the factors that might affect [9-10] the strength of the mechanical bond including the type of enamel conditioner, acid concentration, length of etching time and using other materials to obtain the optimal conditions for bonding and decreasing the damage to enamel [9-12] Past studies [13] used phosphoric acid with many concentrations (5%, 15%, 37%) with different application times (15sec, 30sec and 60 sec) [12] and concluded that the acid concentration can be reduced significantly without significant increase in the

failure of bonded attachments. A study by Carstensen [10, 14], showed that reducing the acid concentration from 37% to 15% H₃PO₄ and applying it for 60 seconds had no significant difference. SEM showed that the 15 seconds application of 37% phosphoric acid concentration was better than other durations and any increase in the duration could lead to increase in the damage of the enamel prism. While many studies [15] demonstrated that the optimal length of time for applying 37% phosphoric acid to the orthodontic bonding area of mandibular premolars was 30 seconds,

reducing the time have been suggested because acid conditioning causes superficial tooth loss [16]. reported that the difference in the shear bond strength of an orthodontic bracket adhesive to enamel etched with 50% phosphoric acid for 5,15 and 60 seconds were not significant. In contrast Mardaga and Shanonn [17] determined the bond strength of an orthodontic bonding system to enamel surface etched with 37% phosphoric acid for 15, 30 and 60 seconds. They reported a stepwise increase in bond strength with an increase in etching time.



Graph-1: Intergroup comparison between median of all groups (MPa)



Graph-2: Intergroup Mean differences (MPa)

The calculated etch depth ranged from 27.1µm by etching with 37% H₃PO₄ for 60 seconds to 3.5 µm by etching with 5 % H₃PO₄ for 15 seconds [18]. Acid etching technique is clinically effective and reliable. However, there are few drawbacks associated with this technique i.e. irritation of acid to oral soft tissues and time required to obtain the desired dissolution. Air abrasion, on the other hand, possesses neither of these drawbacks, while having minimal effect on oral soft tissues, with typical tooth surface preparation time ranging from 0.5 to 3 seconds, without additional step of rinsing or drying. Various studies have been done to evaluate the potential of air abrasion technique [19].

Air abrasion technology introduced by Dr. Robert Black [20] was examined for its potential application in dentistry. This technique uses a high speed stream of aluminum oxide particles propelled by air pressure. Zachrisson and Büyükyilmaz [21] found that sandblasting improves the strength of bonds to gold, porcelain, and amalgam. Further, Faltermeier and Behr [22] reported that the process of sandblasting improves the shear bond strength (SBS) of stainless steel brackets. Chung *et al.* [23] found sandblasting to be a more viable alternative to chemical etching techniques in terms of bond strength, while Berket *et al.* [24] and Canayet *et al.* [25] reported that sandblasting

the enamel surface does not provide adequate SBS for bracket bonding.

Use of the Self Etching Primer in adhesive system for enamel conditioning has become popular among orthodontists because it produces a gentler etch pattern as compared to other methods and because the combination of the etchant and primer in this method simplifies the clinical procedure. So a self-etching primer system has been introduced for the bonding of orthodontic brackets. Bishara *et al.* [26]. reported that the use of a self-etching primer system resulted in a clinically acceptable bond strength.

So in the present study, different concentrations of phosphoric acid i.e. 2%, 15% and 37% ,air abrasion &Self Etching Primer with 15 seconds application were evaluated to determine the most appropriate etchant for preparing the enamel surface(Graph I).

The fundamental organizational pattern of Human enamel (as given by Ten Cate [27] and Orbans[28]) consist of rod (prism) and inter rod enamel (interprismatic substance). Enamel is built from closely packed long and ribbon like carbonatoapatite crystals measuring 60 -70 nm in width and 25-30 nm in thickness. The unit cell has hexagonal symmetry which is clearly seen in cross-sectional profile of maturing enamel. However, fully mature enamel crystals exhibit an irregular outline due to compression against each other during final part of growth. The boundary between rod and interrod enamel is delimited by a narrow space containing organic material known as rod sheath.

The etching pattern depends on crystal orientation i.e. crystal dissolves more readily at the ends than on their sides due to different inclinations in the rods as compared to interrod area. As tooth is mainly etched for bonding the brackets, only the buccally oriented smooth surface is considered and occlusal surface is left out. Since in 90% of the cases bonding is mainly performed at the center of clinical crown, the pattern of the etched surface will not change because of different inclination of rods present at cervical and coronal areas.

Study comprised of therapeutically extracted premolars between age group of 12 to 17 years (mandibular/maxillary). As it was observed that younger teeth which often contained perikymata and imbrications lines, displayed a slightly different etching pattern on the cervical region as stated by Galil and Wright [4]. Perikymata showed a Type 1 pattern and appeared to etch better than the imbrication lines, which exhibited a Type 4 pattern.

Shear bond strength in Group I ranged between 7.87 MPa and 8.73 MPa. It demonstrated that

phosphoric acid concentration of 2% can be sufficient for the direct bonding of metal brackets to anterior teeth. The results are in accordance with that of Soetopo *et al.* [29] and Zidan and Hill [30]. However, these in vitro tests cannot be compared directly to the clinical situations, where bonded attachments are subjected to a variety of intraoral forces. Furthermore, in vitro bond strength measurements are carried out usually on flat ground surfaces which will produce values different from those of ungrounded teeth. So clinically, the use of low phosphoric acid concentration seems to be appropriate for bonding ceramic brackets where bond strength is higher than with that of metal brackets. 2% phosphoric acid was found to create considerably less roughening of the enamel in comparison to 37% solution. Denys and Retief [31] concluded that adequate superficial roughness and increased wettability of the etched enamel surface is more important for the orthodontic bonding mechanism than resin penetration into the deeper porous zone.

15% and 37% phosphoric acid showed mean shear bond strength 11.74 MPa and 13.73 MPa respectively which are nearly comparable to each other. Legler[12] showed that if the concentration of phosphoric acid solution that was applied to the enamel was greater than 27%, it resulted in the formation of monocalcium phosphate monohydrate whereas with weaker phosphoric acid solutions the main reaction product was di calcium phosphate dehydrate. The mono calcium phosphate monohydrate is more soluble than di calcium phosphate di hydrate as shown by Carstensen [10] and the bond strength observed was greater, when the concentration of acid increases because the penetration of enamel was increased. These findings are in agreement with those of Cehreli and Bishara *et al.* who determined that phosphoric acid caused damage to the prism at a concentration greater than 37% and therefore it was not ideally suitable for bonding the bracket. So concentration between 27% -37% phosphoric acid is preferred for bonding the enamel surface.

Air abrasion with 50 μ aluminum oxide was used for enamel surface preparation in group IV. Finer aluminum particle size cause smoother surface and resulted in less mechanical retention. The mean SBS was 7.4 MPa which is slightly below the optimal bond strength as suggested by Reynolds [32] to withstand normal orthodontic force. In contrast Roeder [33] observed that aluminum oxide particle size had no influence on the bond strength. Though earlier reports by Wiltshire [34] and Zachrisson [35] showed optimum or increased bond strength when the air pressure was 100 psi, it stood in contradiction to the present study which also employed 100 psi pressure to propel the aluminum oxide particles.

The use of the self-etching primer in adhesive systems for enamel conditioning has become popular

among orthodontists because it produces a gentler etch pattern as compared to other methods and because the combination of the etchant and primer in this method simplifies the clinical procedure. Self-etching primer treatment in Group V gives the mean SBS 10.29 MPa. That is clinically acceptable. Scougall Vilchis [36] *et al.* Ozer *et al.* and Bishara *et al.* [20] also found adequate shear bond strength levels when 4 types of self-etching primer systems was tested for orthodontic bonding. Bishara *et al.* [26] & Yamada *et al.* [37] reported unsatisfactory shear bond strength values of a self-etching primer as compared to the traditional phosphoric acid etching system (Graph II).

Concluding, we can state that 37% phosphoric acid shows the highest shear bond strength with cobblestone appearance of the enamel surface which nearly comparable to 15% phosphoric acid and self-etching primer. Whereas air abrasion with aluminum oxide shows the least shear bond strength. As related to the SEM self-etching primer shows a more regular pattern with spurs or arrow heads visible in contrast to 15% phosphoric acid which shows quite irregular pattern with cobblestone appearance.

CONCLUSION

Enamel preparation plays vital role in the strength of bonded orthodontic attachments so different modes of etching will have different impact on the shear bond strength of the composite material.

- Phosphoric acid concentration 37% shows the highest shear bond strength followed by 15% phosphoric acid but the SEM pattern shows more variations in this group.
- Shear bond strength by self-etching primer shows comparable results to that of the 15% phosphoric acid along with the more regular uniform SEM pattern.
- Phosphoric acid concentration of 2% and air abrasion with aluminum oxide shows the least shear bond strength among all the present groups with varying pattern of enamel surface (SEM).

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