Original Research Article

COMBINED EFFECT OF ACIDIC BEVERAGES AND IMMERSION TIME ON BONDING PERFORMACE OF COMPOSITE TO ENAMEL: AN IN-VITRO STUDY

UNDER PEER REVIEW

ABSTRACT

Introduction: Carbonated beverages are widely consumed over the world. The effect of acidic liquids on dental enamel has a significant impact on composite resin adhering to the enamel substrate. The purpose of this study was to see how acidic beverages affected the bonding of composite resin to exposed enamel. Methods and Materials: 128 premolars were taken and divided into four groups for both 24 hours immersion (n=40) and 15 days immersion (n=40). The samples were divided into the following categories: G1: 3x/15 mins immersion in Beverage A, G2: 3x/15 mins immersion in Beverage B, G3: 3x/15 mins immersion in Beverage C, G4: 3x/15 mins immersion in artificial saliva as control group. Prior to immersion, the buccal surfaces of the enamel were minimally ground at the layer of enamel solely using a Tungsten carbide bur, then flattened with #600 - #1200 silicon carbide paper (SiC) paper. Specimens were submerged in the media for 24 hours and 15 days, respectively. Following immersion, the specimens' prepared surfaces were repaired using composite resin. The enamel pattern was morphological analyzed using a Scanning Electron Microscope (SEM) and the shear bond strength was determined . Results: One-way ANOVA with multiple comparison tests was used to statistically evaluate the data. When compared to control media, all of the beverages tested significantly reduced shear strength. Following immersion in the test beverages, enamel's morphology changed noticeably, with Beverage A group displaying the most pronounced morphological changes. Conclusion: All acidic beverages examined had a detrimental impact on shear strength. In different periods of time, varied degrees of enamel demineralization were detected.

Keywords: [Soft drinks, composite resin, shear strength, enamel, acid]

1. INTRODUCTION

After trauma, caries, and periodontal disease, non-carious tooth tissue degeneration posed the next major threat to the function and survival of the human dentition. Erosion is the most common threat to dental surface degradation among all wear processes, according to epidemiological research and studies. Beverages containing acid or carbonated drinks are the most common culprits for this depletion. The global population consumes a lot of carbonated beverages [1]. According to a research by the Malaysian Health Ministry [2], 36% of Malaysian adolescents drank fizzy drinks at least once a day. Citric acid-based drinks [3], cola-based drinks [4], energy drinks[5], and isotonic beverages have also been shown to hasten the erosion process. F&N Beverage Marketing Sdn Bhd, Coca-cola Bottlers (M) Sdn Bhd, and Permanis Sdn Bhd were the leading suppliers of beverages in Malaysia [6].

When the intraoral pH falls below 5.5, demineralization of the enamel can occur. Most acidic beverages on the market were within this range, and drinking these soft drinks can reduce the pH in the oral environment in as little as 20 minutes [7]. Dental erosion has been recognized as a facilitated phenomenon called erosive tooth wear, in addition to mechanical abrasion processes and attrition. Each of these occurrences has its own features, and they frequently occur together, making it impossible to isolate their specific impact.

Dental erosion can result in a variety of clinical issues, including aesthetics owing to enamel fracture, which can lead to tooth shortening and loss of occlusal vertical dimension. Some individuals may be uncomfortable as a result of the alterations, necessitating restorative treatment. Many clinicians have urged patients to seek treatment as soon as symptoms appear, in order to avoid long-term structural damage to hard tissue [1]. Basic treatments for carious teeth include composite restorative treatment using adhesives. However, there is a paucity of evidence and research on how enamels exposed to carbonated drinks may react with composite and adhesive bonding materials [1]. Therefore, the goal of this study is to evaluate the combined effect of time and acidic beverages on strength of composite resin to enamel. The null hypothesis was that neither immersion time nor the types of acidic content would affect the bond strength of composite resin to enamel.

2. MATERIAL AND METHODS

2.1 Preparation of samples

128 human molars were gathered from various dental clinics and kept in a 0.2% thymol solution. Only the buccal surface of enamel was revealed after the teeth were cleaned and immersed in self-curing, fast-setting acrylic resin (Rapid Repair, DeguDent GmbH, Hanau, Germany). The buccal site of the premolars was then minimally ground at the enamel layer only using a Tungsten carbide bur, and then polished with silicon carbide sheets in a circular motion starting with grade #600, then #800, #1000, and #1200 until a flat surface was achieved. A calibrated pH meter (Horiba Laqua-PH1100) was used to determine the pH of each beverage/media. Tables I and II reveal the pH and content of the beverages/media used.

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Table 1: pH of immersion media

Immersion media	pH

Artificial saliva	8.81
Beverage A	2.57
Beverage B	3.10
Beverage C	3.09

Immersion Media	**Composition**
Artificial Saliva	400 mg NaCl, 1.21 mg KCl, 780mg NaH2PO42H2O, 5mg Na2S.9H2O, 1000mg CO(NH2)2 and 1000 ml of distilled water
Beverage A	Carbonated water, high fructose corn syrup, caramel color, phosphoric acid, natural flavor, and caffeine
Beverage B	Carbonated water, color (caramel E150d), acids (phosphoric acid, citric acid), flavorings (including caffein), sweeteners (Aspartame, Acesulfame K), acidity regulator (sodium citrate), preservative (potassium sorbate). Contains a source of phenylalanine.
Beverage C	Orange juice from concentrate, sugar, orange pulp cells, orange cell sacs, pure squeezed orange juice, citric acid, stabilizer, flavoring, sodium benzoate, vitamin (C, E, A), sodium metabisulphite, coloring, sulphur dioxide

Table 2: Content of artificial saliva and other media

**Based on manufacturer's information*

2.2 Erosive challenges

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A summary of the procedure is schematically illustrated in Figure I and 2. All samples were divided into four groups at random, with immersion periods of 24 hours (n=40) and 15 days (*n*=40) (Table 2). Every 8 hours, each group was immersed three times each day for 15 minutes per cycle. The samples were re-immersed in artificial saliva after each cycle. The control samples were kept in artificial saliva for the duration of the experiment (24 hours and 15 days), with the solution being changed daily. All the three acidic beverages were stored at a temperature of 5°C, while the samples in artificial saliva was kept at room temperature.

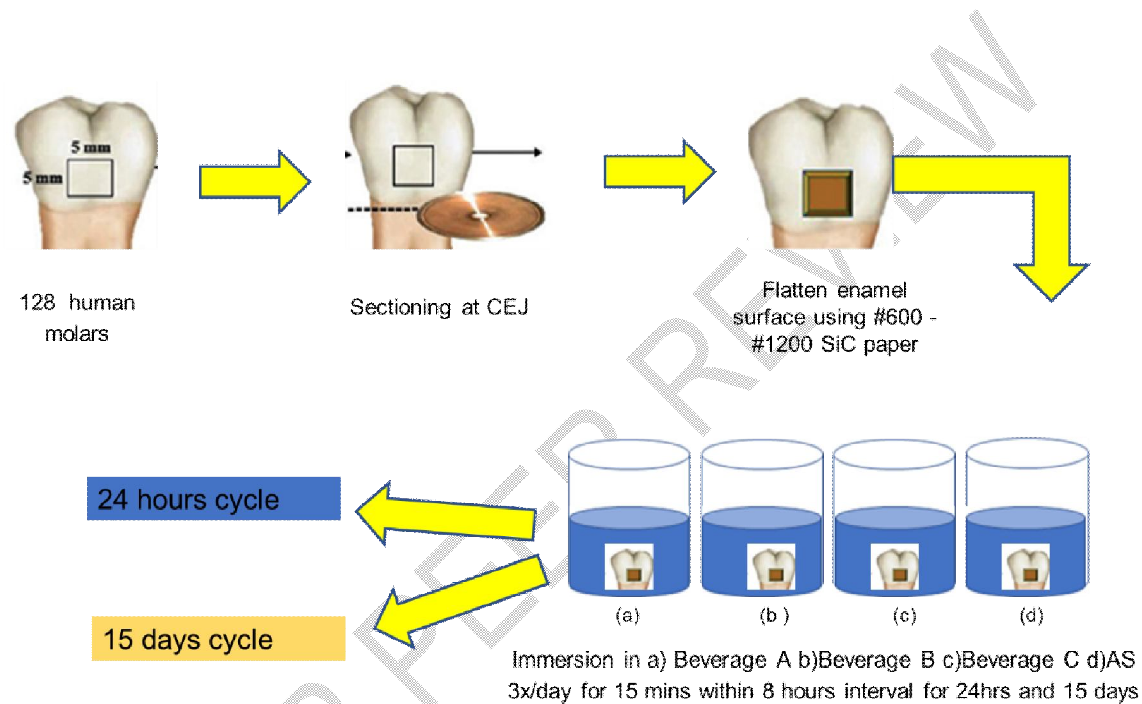

Figure 1 shows the technique for preparing the specimen and immersion procedure.

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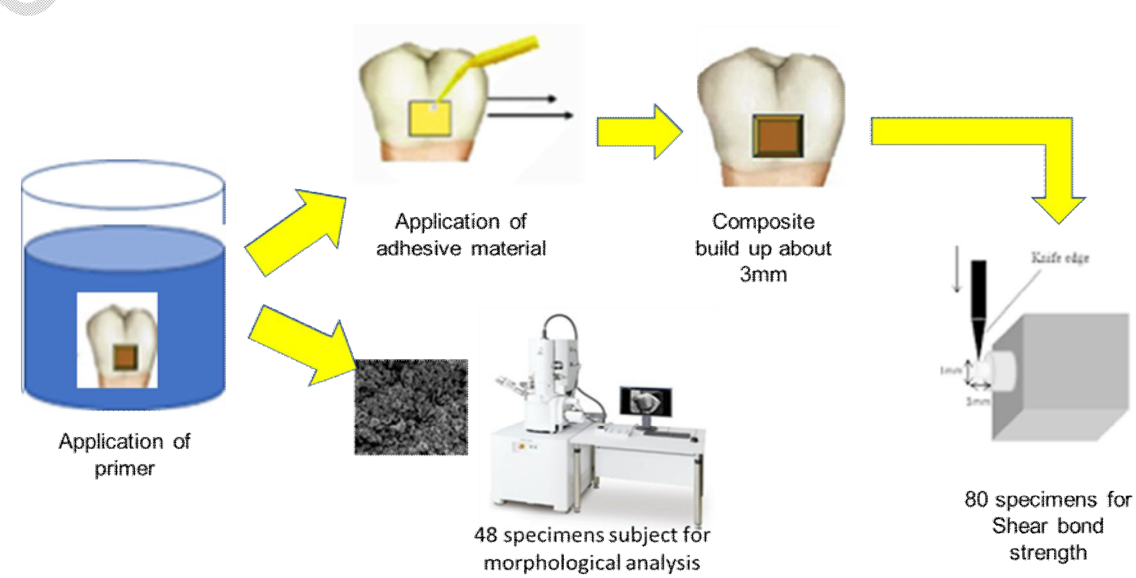

Figure 2: Composite build up for shear strength testing and micromorphological analysis under SEM.

2.3 Restoration procedure

All samples were cleansed with 0.9 % normal saline after the erosive challenge. Bonding was done with a self-etching, two-component universal adhesive (Kerr OptiBond^{TM} eXTRa Universal), which began with the application of primer with a disposable microbrush for 5 seconds and gentle air-drying for 5 seconds, followed by the application of the adhesive and gentle air-drying for 5 seconds to allow the solvent to evaporate. Light curing was performed for 15 seconds using a light curing equipment (MiniLed, SatelecActeon) with a 1000mW/cm2 light intensity. Following the bonding method, a composite build-up (Diafil, DiaDent) with a 4mm height of composite resin, cylindrical shape, was applied to all samples, with each 1mm increment being light-cured for 40 seconds. Components and the mode of application of the materials obtained from manufacturer's data sheets were shown in Table 3.

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Materials	Composition	Mode of application
	BiS-GMA,TEGDMA,Barium glass,silica,nanohybrid inorganic filler particles.	Composite build up curing time 20 seconds.
Kerr optibond XTR Universal	Primer:	Apply self-etching primer

Table 3. Chemical formulations of adhesive system and resin composite used in this study.

2.4 Shear bond strength test

The samples were mounted to the universal testing equipment (Instron E3000 Canton, MA) and a chisel-shaped rod was used to provide a shear stress parallel to the adhesive contact in the base of the cylinders. The maximal force (N) required to debond the composite was measured using a crosshead speed of 0.5 mm/min. By dividing the force values by the base area, the SBS was computed (MPa).

2.5 SEM (Scanning Electron Microscopy)

Another 48 human molars was selected and underwent immersion with the same manner as stated in previos . After the cycle was completed, the samples were immersed for 10 minutes in distil water, dehydrated in ascending ethanol manner (25 %, 50 %, 75 %, 95 %, and 100 %); mounted on stubs; gold sputtered, and analyzed in a scanning electron microscope (SEM-ZEISS Model SEM EVO50) operating at 20 kV. At magnification of 1000x, the whole buccal surface of each tooth was scanned and the best representative pictures were recorded. The purpose of the SEM analysis was to offer just a visual and illustrative comparison of the specimens, therefore no statistical analysis was performed.

2.6 Statistical Analysis

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The period and immersion media were used as variables in a One-way ANOVA to determine the mean shear bond strength. At a significance level of 0.05, Tukey's HSD multiple comparisons statistical test was utilized. SPSS Statistical Software version 16 was used for all statistical studies (SPSS INC, Chicago, IL, USA).

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3. RESULTS

The shear bond strength of the control group (artificial saliva) was substantially higher ($p<0.05$) when compared to the other three drinks as shown in Table 4. The shear bond strength of the three acidic beverages was significantly lower than that of the control group ($p<0.05$), although there was no statistical significance difference between them ($p>0.05$).

Groups	Period	
	24 hours	15 days
Artificial saliva	15.1+4.32[a]	13.4+3.19[a]
Beverage A	7.3+4.21[b]	5.5+4.3[b]
Beverage B	8.8+4.11[b]	7.9+3.20[b]
Beverage C	10.5+3.30[b]	10.3+3.38[b]

Table 4 :Means and standard deviations (MPa) of shear bond strength for experimental groups.

**Groups identified by different lowercase superscript letters indicate statistical significance ($p< 0.05$).*

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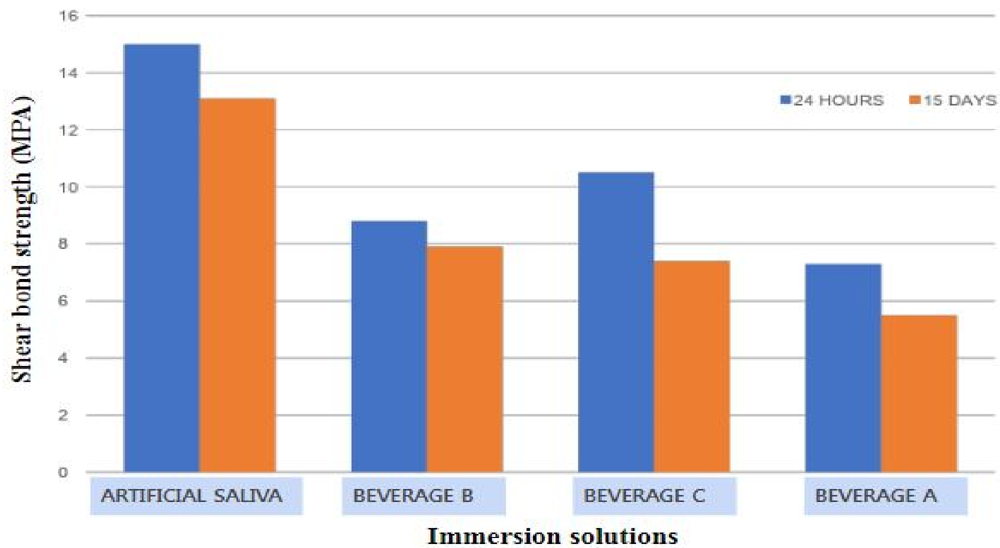

Figure 3: Effect of types of solution and immersion period on shear bond strength.

The enamel was altered to various degrees when observed using a scanning electron microscope. Figure 4 shows a representative pictomicrograph of specimens immersed in four distinct types of medium for a total of 24 hours at 1000x magnification. In 4A, there was a large area of significant demineralization on the enamel surface. In figure 4B, there was a partial demineralization area on the enamel surface (in a circle). Changes in enamel morphology may be seen in Figure 4C, which are defined by areas of shallow demineralization. In 4D, the surface appears to be homogeneous with no visible alterations in the enamel morphology.

Figure 5 depicted a representative pictomicrograph of specimens immersed in four distinct types of medium for 15 days under 1000x magnification. In 5A, there was a continuous area of demineralization on the enamel surface where the prisms looked to be missing and some zones were dissolving. Crystals with fuzzy outlines (arrow) were seen inside the prisms in 5B, indicating some degree of demineralization. In Figure 5C, we saw a region of compartmented enamel demineralization (arrowhead). However, as demonstrated in Figure 5D, the surface remains smooth with no porosities or demineralization activity.

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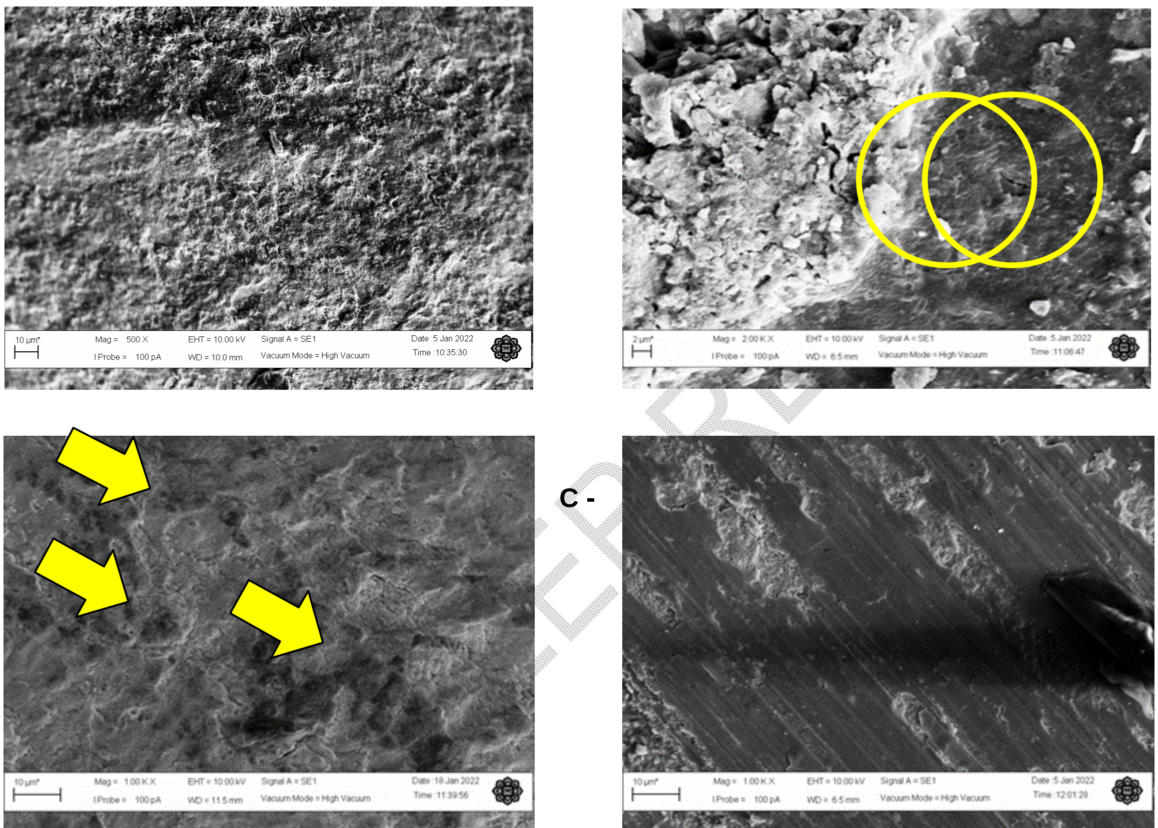
24 hours duration:

A - Beverage A B - Beverage B



C - Beverage C D - Artificial saliva

FIGURE 4

Figure 4A showed extensive demineralization, while Figure 4B revealed partial demineralization on the enamel surface. Figure 4C showed changes in enamel morphology marked by area of shallow demineralization. Surface appears as homogeneity without obvious changes in the enamel morphology as shown on Figure 4D.

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15 days duration:

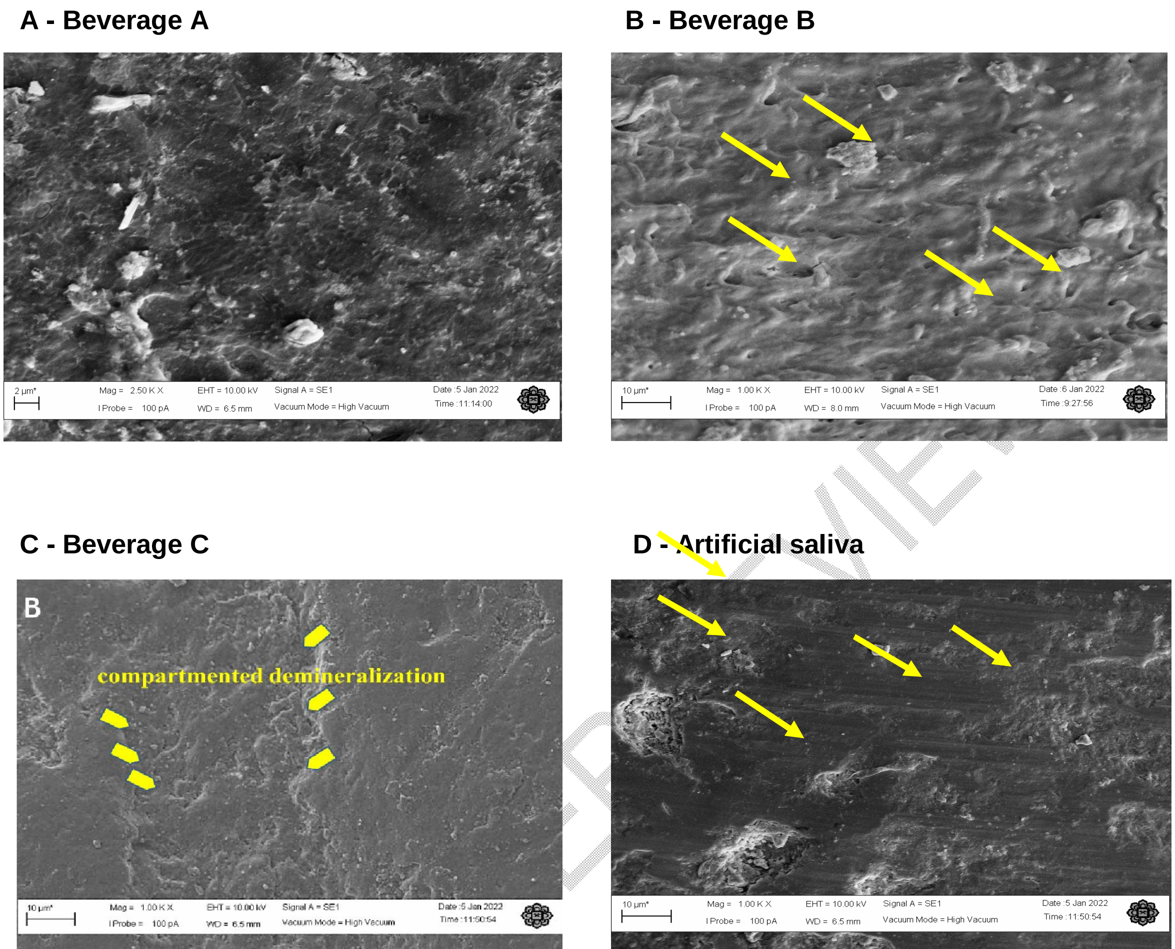

Figure 5A-area of continuous demineralization on the enamel surface. In Figure 5B crystals with faded contours (arrow)-increased degree of demineralization. Areas of compartmented demineralization (arrowhead) of the enamel were evident in Figure 5C. The surface remains smooth with no porosities or demineralization in Figure 5D.

4. DISCUSSION

Previous research on the bonding of composite to enamel exposed to acidic liquids are still inconclusive. Enamel is the outermost layer of the tooth that serves as a substrate for composite resin bonding. Any changes in enamel microstructure will impair resin tag [8] adherence, bond strength, and formation.

The null hypothesis is rejected based on the findings of this study, as there was no significant variation in the shear bond strength of composite between all acidic drinks. Their shear bond strength values, however, were significantly lower when compared to the control group. This is due to the acid content; however, the types of acid utilized in this study, phosphoric acid and citric acid, revealed varying degrees of erosive enamel pattern. The acidic characteristics, titratable acidity, which is the acidic content, and pKa of acid, which

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reflects the concentration of H+ ions, all influence the depth of erosion. Acidic soft beverages with a low pH value lower the pH of the oral cavity, eroding the enamel more effectively [9].

When compared to the other three beverage groups, the Beverage A group had the lowest mean shearing forces for both the 24 hours and 15 days immersion periods. Beverage A, which included only phosphoric acid, showed a greater erosive effect within the first 24 hours of exposure, whereas Beverage B (phosphoric acid plus citric acid) and Beverage C (citric acid) demineralized the enamel after prolonged exposures (15 days). Saliva rich in ions such as calcium ions, hydroxide ions and phosphate ions surround the outer enamel surfaces. As a result, under normal conditions, the enamel crystals will not demineralize by losing Ca^{2+}, PO_4^{3-}, or OH^- to the environment, but when these ions are depleted in the surrounding saliva, combined with high concentrations of H^+ ions found in most erosive substances, including acidic drinks, there will be a loss of equilibrium between enamel and its surrounding fluid, causing the H^+ ions from the acid to react on the enamel while other acids such as carbonic, phosphoric, acetic, lactic, malic, oxalic, tartaric, and citric acid [10] provide H^+ ions that contribute to dental erosion[11].

The erosive solution will only affect the near-surface enamel with little dissolution during initial exposure, and then it will cause partial breakdown of enamel crystal by diffusion into enamel, resulting in a softened layer at the enamel surface [11,12]. Citric acid in carbonated drinks has the potential to chelate calcium ions, causing rapid calcium loss in the tooth structure and, as a result, demineralization of the enamel. Citric acid (sodium citrate) reduces the acidogenicity of dental plaque and is indicated as a technique to lessen the cariogenicity of non-alcoholic soft drinks [13]. Citric acid may serve as a chelating agent, binding calcium from enamel and causing the solution to become undersaturated, facilitating demineralization [14,15].

The other reason could be that as the acidic content is absorbed into the enamel, the dissolution process consumes H+ ions, resulting in an increase in OH- ions concentration as pH rises, while the Ca^{2+} and PO_4^{3-} ions released by enamel saturate the solution, causing demineralization to stop within a short distance from the surface [11]. Beverage A (phosphoric acid content) has the lowest pH of all the groups, and because it has the lowest pH, it may be a contributing factor in higher enamel surface degradation. However, it is vital not to estimate the erosive potential of beverages and meals only on the basis of pH, as there is no "critical pH" associated with erosion[10]. Although pH is critical in enamel demineralization, buffering capacity is also important; the greater buffering capacity a substance has, the more erosive effect it will occur [10,11].

For both cycles, all eroded specimens had uneven surfaces and demineralization of the enamels (24 hours and 15 days). During the 15-days immersion, the enamel characteristics in Beverage A were more extensive and visible than those in Beverage B and C. We speculated that the presence of phosphoric acid in Beverage A was the cause of this effect. Although beverage B and C were shown to be less erosive than beverage A, there was still some erosion when compared to the control group. The interaction of pH, acid concentration, and the presence of calcium[16] influences the degradation of enamel by dietary acid solutions. Despite the fact that beverage B contains two forms of acid (phosphoric and citric acid), it did not demonstrate a significant difference in enamel demineralization when compared to beverages A and C, which only contained one type of acid, due to citric acid. This explains why beverages containing citric acid did not have effect as severe on enamel deformation as other beverages.

Early detection of erosion and identification of risk factors are critical since they increase the likelihood of a successful treatment. Brushing teeth shortly after consuming acidic drinks,

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according to studies, hastens tooth loss. It is obvious that prolong use of acidic drinks may enhance the risk of rapid dentin demineralization rate and caries[17]. The adherence of restorations to degraded enamel caused by soft beverages has also been a topic of debate among researchers. When comparing enamel submerged in soft drinks to enamel immersed in artificial saliva, the shear strength data showed that enamel immersed in soft drinks had a lower shear strength value.

Clinically, this might be a major problem for dentists when it comes to bracket attachment for fixed appliances, as well as any restoration instances using enamel/dentin as the substrate. Excellent fixed device-to-dental-surface adhesion is a critical component of any orthodontic treatment's clinical success[18]. Another key factor that needs to be considered is the quality and long-term durability of the enamel restoration used to address the defect caused by soft drink erosion. Other studies have found that erosion weakens the bond between the enamel and the dentin[19], but there has been no research on the bond between the enamel and the dentin. As a result, the findings of this study revealed important information about how erosion caused by soft drink affects the bonding efficacy of enamel surfaces. As a result, dentists should be cautious when using resin-based materials to restore enamel erosion lesions in patients. Although there is a lack of evidence on the impact of surface pretreatment on damaged enamel, further study should be carried out to evaluate on how surface pretreatment methods can increase bond strength to eroded dentin[19].

5. CONCLUSION

In this study, the combined effect of immersion time and acidic contents had negatively affected the bond strength and enamel morphology in samples immersed in Beverage A, but there was no statistically significant difference when compare between the other two acidic beverages. The study discovered areas of demineralization on the enamel surface near the adhesive produced by soft acidic liquids. Patients undergoing any restoration requiring enamel substrate or fixed orthodontic bracket bonding treatments should be counselled to avoid soft acidic drinks, which might increase the risk of erosion.

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7. APPENDIX

Contribution Details :

	Author A	Author B	Author C	Author D
Concepts	√	√	√	√
Design	√			√
Definition of intellectual content	√	√	√	
Literature search		√	√	
Clinical studies	-	-	-	-
Experimental studies	-	-	-	-
Data acquisition	√	√	√	√
Data analysis	√			√
Statistical analysis	√			√
Manuscript preparation	√	√	√	√
Manuscript editing	√			
Manuscript review	√			√
Guarantor	-	-	-	-

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