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Does Applying Morpholine to Saliva-Contaminated Acrylic Resin Improve the Repair Bond Strength?

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Abstract: The current study evaluates the effect of morpholine on saliva-contaminated acrylic resin repaired with light-cured resin composites. Sixty rods of self-curing acrylic resin were fabricated and assigned into four groups of fifteen specimens and surface-treated with saliva, phosphoric acid (PH), morpholine (MR), liquid MMA monomer, and a universal adhesive agent (UA, Singlebond Universal) based on the following techniques: group 1, saliva; group 2, saliva + PH + MMA + UA; group 3, saliva + MMA + UA; and group 4, saliva + MR + MMA + UA. An Ultradent model was placed at the center of the specimen, and then the resin composite was pressed and light-cured for 20 s. A mechanical testing device was used to evaluate the samples' shear bond strength (SBS) scores. The debonded specimen areas were inspected under a stereomicroscope to identify their failure mechanisms. The data were assessed by employing the one-way ANOVA approach, and the significance level ($p < 0.05$) was established with Tukey's test. The greatest SBS scores for group 2 (30.46 ± 2.26 MPa) and group 4 (32.10 ± 2.72 MPa) did not differ statistically significantly from one another. The lowest SBS recorded for group 1 was 1.38 ± 0.87 MPa. All of the fractured samples in group 1 had an adhesive failure profile. Groups 2 and 4 had the greatest percentages of cohesive failures. This study concluded that applying phosphoric acid and morpholine to sandblasted self-curing acrylic resin contaminated with saliva before MMA and universal adhesive agents are applied is the most efficient protocol for stimulating SBS when it is repaired with light-cured resin composites.

Keywords: adhesive agent; MMA; morpholine; surface treatment; universal adhesive

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1. Introduction

The dental material known as self-curing acrylic resin is an organic substance produced by combining ethyl polymers derived from a combination of polymethyl methacrylate (PMMA) and methyl methacrylate (MMA) [1], which are polymerized through chemical processes without the need for heat or light. There are two primary components to this kind of resin: liquid and powder. While polymers and initiators are present in the powder component, monomers and activators are present in the liquid component [2]. This substance is widely utilized in prosthetic and restorative dentistry because of its great versatility. Because it is esthetically pleasing and long-lasting, self-curing acrylic resin is indispensable in many dental applications [3]. In the interim between permanent restorations like crowns or bridges, it acts as a temporary restorative material. Using an insufficient thickness of acrylic resin to make temporary restorations might lead to fractures. During visits, fractures may occur. Repairing is frequently a more desirable and time-saving solution than new restoration when efficiency and economic considerations are taken into consideration. However, the repair pieces must be well bonded to the original resin in order to guarantee their quality and long-term endurance. For the restored creation to be structurally sound and function well, the old and new materials must successfully bind together [4,5]. Light-cured resin composite materials can be used for the repair of destroyed acrylic resin [4,6].

The development of light-cured resin composites and their application in restorative procedures contrast with the use of acrylic resin in temporary restoration and denture bases [7,8]. Additional restorative materials were created after resin composites were established; however, light-cured resin composites still have several advantages over other restorative materials. The esthetically pleasing appearance of light-cured resin composites is one of their most crucial characteristics. Resin composites consist of many components, including a resin matrix, inorganic fillers, silane coupling agents, pigments, initiators, and accelerators. The resin matrix is a function of the polymerization process [9]. Filler is used due to its mechanical properties. There are fundamentally two categories of filler particles: macrofill particles and microfill particles. Hybrids are a combination of macrofill and microfill particles and have demonstrated excellent mechanical and physical properties, as well as good potential thermal results [10,11]. Moreover, advanced resin composites of the future have been designed specifically for bio-interactive systems, effectively addressing problems like secondary caries and mechanical breakdowns, hence extending the lifetime of dental restorations [12]. The pigments utilized in their creation provide them with a range of hues that closely resemble enamel, enabling resin composite restorations that are nearly identical to real teeth [13]. The applications of resin composites in dentistry are numerous and include, but are not limited to, filling materials, repair materials, crowns, inlays, onlays, core build-ups, orthodontics, cement for single or multiple prosthetics, and root canal posts [14,15]. It is conceivable that these materials will be used more frequently in the future.

Artificial saliva is a synthetic substance that mimics the composition and characteristics of natural saliva. Water and additional ingredients, including glycerin, carboxymethylcellulose, and other kinds of electrolytes, are usually included. Occasionally, xylitol and other additions are included. Artificial saliva relieves irritation to the oral tissues, protects the mucosa in the mouth, makes eating simpler, and hydrates the oral tissues [16]. The operational field of dentistry can be affected significantly by saliva contamination [17–19]. Strong adhesive binding properties can only be achieved when using a clean, high-energy material surface. A restorative surface etched with phosphoric acid can absorb salivary contents rapidly, resulting in a decrease in the surface free energy and making their adhesion impossible [20]. Therefore, in this study, we used saliva contamination as a negative control when repairing self-curing acrylic resin with light-cured resin composites.

Morpholine is a heterocyclic organic substance. There are ether and amine functional groups in this heterocyclic group. Because of its amine group, morpholine is categorized as a base [21]. Morpholine is a structure that is interesting in medicinal chemistry since it is found in many medicines and physiologically active compounds. Because of its low price and low polarity, it is frequently employed in the synthesis of organic molecules and in the chemical industry as a solvent [22]. In previous studies in dentistry, morpholine was used as a decontaminant for saliva in resin composite repair [23] and as an agent for the surface treatment of fiber posts [24]. Additionally, Ghosh and Pal reported the use of morpholine as a photo-initiator in the photopolymerization of MMA, and it was discovered that morpholine may cause the polymerization of MMA [25].

However, there are still insufficient data on the use of morpholine as a surface treatment for promoting the bond ability between saliva-contaminated acrylic resin and light-cured resin composites; for this reason, this study was begun. In the present investigation, we purposely determine the effect of morpholine on saliva-contaminated self-curing acrylic resin repaired with light-cured resin composites by examining the shear bond strengths

(SBSs). The null hypothesis states that morpholine does not significantly affect the SBS between saliva-contaminated acrylic resin and light-cured resin composites.

2. Materials and Methods

2.1. Acrylic Resin Preparation

G*Power 3.1 software was utilized to calculate the sample size, with 0.05 serving as the significance level and 0.95 as the power. The experiment used sixty rods of self-curing acrylic resin (Shofu Inc., Kyoto, Japan). Each of these 6.0 mm diameter and 4.0 mm thick acrylic resin specimens was created with a silicone mold to ensure accuracy. Following the curing process, the silicone molds were removed to reveal the solid acrylic resin rods, which were then attached to polyvinyl chloride tubes using type IV gypsum. To normalize and uniformize the surface texture of the acrylic resin surfaces, a 600-grit silicon carbide abrasive sheet (RS Component, Bangkok, Thailand) was used to sand them. Every specimen was subjected to a 10 min ultrasonic washing protocol in distilled water using an ultrasonic cleaning device (Misumi Corporation, Tokyo, Japan) in order to completely clean the specimens and remove any impurities.

2.2. The Sandblast Method

Fifty-micron aluminum oxide particles were used to blast the specimens. During this sandblasting process, the aluminum oxide particles were targeted 10 mm away from the specimens. Sandblasting was undertaken at an applied pressure of 2.8 bar for an attentively timed period of 10 s [4]. These samples went through a series of cleaning procedures after being sandblasted. They were first thoroughly washed for 10 s with a mixture of water and air, which removed any last bits of dirt or debris. Then, via a triple syringe, a 10 s air-drying process was carried out to guarantee a clean, dry surface for further procedures.

Figure 1 shows a schematic of the methodology.

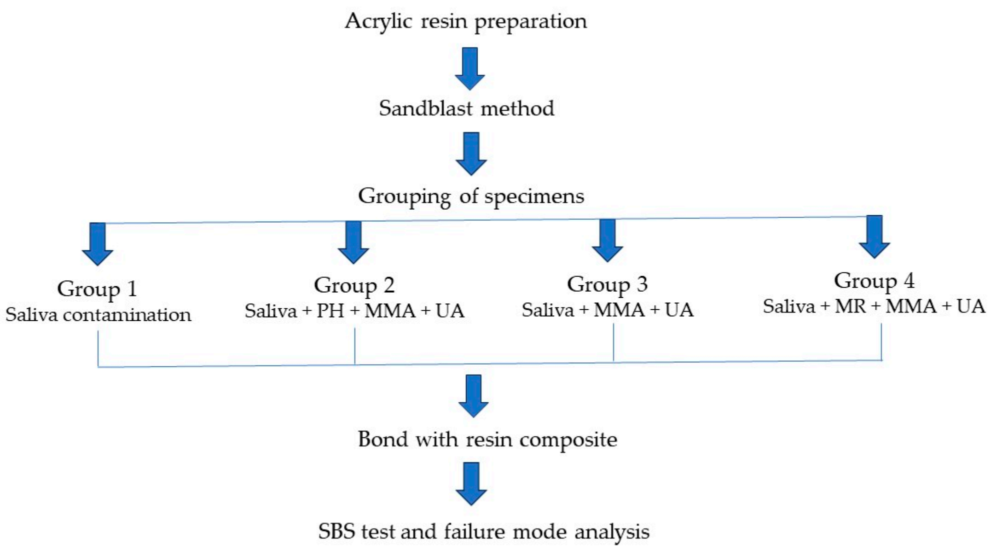

Figure 1. Schematic of the methodology. Abbreviations: PH, phosphoric acid; MMA, methyl methacrylate; UA, universal adhesive; MR, morpholine; SBS, shear bond strength.

2.3. Grouping of the Specimens

Table 1 lists the materials that were implemented in the current research. The sandblasted acrylic resin samples were separated randomly into four groups (n = 15 per group) and surface-treated employing saliva, phosphoric acid, morpholine, liquid MMA monomer, and a universal adhesive agent (Singlebond Universal, 3M, Neuss, Germany) based on the following techniques (Table 2).

Table 1. Lists the materials that were implemented in the current research.

Material	Manufacturer	Chemical Composition
Self-curing acrylic resin	Shofu Inc., Kyoto, Japan	Powder: MMA-EMA copolymer, pigments, and others Liquid: MMA and others
Morpholine	Loba Chemie PVT Ltd., Mumbai, India	98% extra pure $O(CH_2CH_2)_2NH$
Singlebond Universal	3M, Neuss, Germany	10-MDP, Bis-GMA, HEMA, DMA, methacrylate functional copolymer, silane, filler, initiators, ethanol, water
Resin composite, Harmonize A2E	Kerr Corporation, Orange, CA, USA	Bis-GMA, TEGDMA, EBPADMA, zirconia/silica cluster filler (2–3 m) comprising 20 nm spherical fumed silica and 5 nm zirconia particles, and prepolymerized filler (filler loading; 81.5% by weight)

Abbreviations: MMA-EMA, methyl methacrylate–ethyl methacrylate; MMA, methyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol A-glycidyl methac-rylate; TEGDMA, triethylene glycol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; DMA, dimethacrylate; EBPADMA, ethoxylated bisphenol A dimethacrylate.

Table 2. Shows the groups of surface-treated specimens.

Groups	Surface Treatment
1	Saliva-contaminated acrylic resin (saliva)
2	Saliva-contaminated acrylic resin treated with phosphoric acid prior to the application of MMA monomer liquid and universal adhesive (saliva + PH + MMA + UA)
3	Saliva-contaminated acrylic resin treated with MMA monomer liquid and universal adhesive (saliva + MMA + UA)
4	Saliva-contaminated acrylic resin treated with morpholine prior to the application of MMA monomer liquid and universal adhesive (saliva + MR + MMA + UA)

Abbreviations: PH, phosphoric acid; MMA, methyl methacrylate; UA, universal adhesive; MR, morpholine.

2.4. Artificial Saliva Contamination

The acrylic resin surface was treated with 100 microliters of artificial saliva using a microbrush (Dental vision, Bangkok, Thailand), and it was then thoroughly dried for 20 s using an air syringe.

2.5. Phosphoric Acid Etching Protocol

The specimen was exposed to 37% phosphoric acid solution for 30 s (Kerr Corporation, CA, USA). It was cleaned with water and then dried for ten seconds with a triple-syringe spray.

2.6. Morpholine Surface Treatment

To make the 9.8% morpholine solution (Morpholine, medical grade, Loba Chemie PVT Ltd., Mumbai, India), 98% morpholine was diluted from 10 mL to 100 mL with distilled water. Ten microliters of the 9.8% morpholine solution was applied thinly to the surface using a disposable micro-applicator. The covered surface was then gently dried for 20 s with air from the triple syringe [23].

2.7. MMA Monomer Treatment

The liquid MMA monomer (Shofu Inc., Kyoto, Japan) was used to condition the specimens. Applying the monomer liquid with a single-use microbrush took one minute of careful application [4]. Subsequently, the specimens were left to evaporate in order to guarantee that any extra monomer disappeared and prepare them for further operations.

2.8. The Universal Adhesive Protocol

Universal adhesive was used to treat the specimens. Applying the universal adhesive with a single-use microbrush required one minute of careful application. Subsequently, the specimens were left to evaporate in order to guarantee that any extra monomer disappeared and the surface looked shiny. Subsequently, light activation was achieved within around 20 s (Acteon, Merignac, France).

2.9. Bonding with Light-Cured Resin Composites

An Ultradent mold that measured precisely 2.0 mm in diameter and thickness was placed at the center of the surface-modified specimen. A resin composite (Harmonize, Kerr Corporation, Orange, CA, USA) of the shade A2E was pressed into the model to create the test specimen, and an LED light-curing system light-polymerized it for 40 s. The Ultradent mold was carefully removed from the specimen. The samples underwent an additional 40 s light activation cycle utilizing the same LED light device system in order to optimize the polymerization process and guarantee thorough curing. For every specimen, a one-day incubation protocol was conducted in an incubator (Scitek Global Inc., Houston, TX, USA) containing distilled water at 37 degrees Celsius.

2.10. Evaluation of SBS and Investigation of Failure Characteristics

The SBS data were generated with a knife-edge cutting blade at a laboratory speed of 0.5 mm per minute using the AGS-X 500N universal measuring equipment (Shimadzu Corporation, Kyoto, Japan) (Figure 2). The shear bond score was established by dividing the bond breakdown strength by the total area of the bonding location.

Figure 2. The SBS test configuration.

The acrylic resin and light-cured resin composites' fracture mode profiles were obtained using a stereomicroscope featuring 40× magnification. Three prototypes were created to determine the processes causing the fractures [26–28]: (A) an adhesive form (debonding at the junction between the acrylic resin and the resin composite), (B) a cohesive form (a fracture inside the acrylic resin or the resin composite), and (C) a mixed form (fractures including a mixture of the adhesive and cohesive modes).

2.11. Statistical Assessment of the Data

The Kolmogorov–Smirnov test was employed to decide whether the shear bond value pattern was normal. The SBS of each group was assessed by one-way analysis of variance

(ANOVA) to determine any significant differences between the light-cured resin composite and acrylic resin surface treatment groups. Tukey's HSD assessments were utilized to assess the SBS data in megapascals (MPa). For every analysis, a confidence level of 95% and a significance level of 0.05 were considered.

3. Results

3.1. SBS Data

Table 3 reports the means and standard deviations (SD) of the SBS data (MPa). There was no statistically significant difference between group 2 (30.46 ± 2.26 MPa) and group 4 (32.10 ± 2.72 MPa) in terms of their maximum SBS values. The minimum SBS value was determined for group 1 (1.38 ± 0.87 MPa).

Table 3. The mean SBS (SD) and failure pattern mode (%).

Group	Mean SBS (SD)	Failure Pattern		
		Adhesive	Mixed	Cohesive
1. Saliva	1.38 ± 0.87^{a}	100	0	0
2. Saliva + PH + MMA + UA	30.46 ± 2.26^{b}	0	20	80
3. Saliva + MMA + UA	17.36 ± 2.81^{c}	0	33.33	66.67
4. Saliva + MR + MMA + UA	32.10 ± 2.72^{b}	0	6.67	93.33

Abbreviations: PH, phosphoric acid; MMA, methyl methacrylate; UA, universal adhesive; MR, morpholine. Values with the same letters do not differ statistically significantly.

3.2. Failure-Type Patterns

The failure-type incidence is explained in detail in Table 3. Every sample with fractures in group 1 presented an adhesive failure profile after breaking. Additionally, the majority of the cohesive failure scenarios were covered by groups 2 to 4. The groups with the highest proportion of cohesive failures were 2 and 4, accounting for 80–93.33 percent of the total.

3.3. Stereomicroscope Images

This section of the study used stereomicroscope images to explain the adhesive, mixed, and cohesive failure mode configurations. The stereomicroscope images are displayed in Figures 3–6. The adhesive failure structure for group 1 is displayed in Figure 3. A cohesive failure structure and a mixed failure structure are presented for groups 4, 5, and 6 (Figures 4–6).

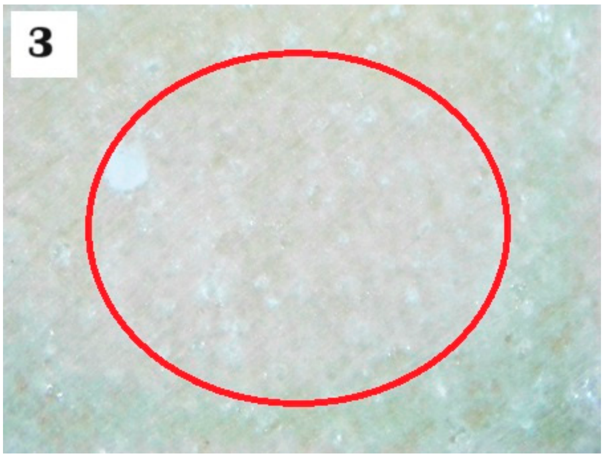

Figure 3. The stereomicroscope picture for group 1; all adhesive failures.

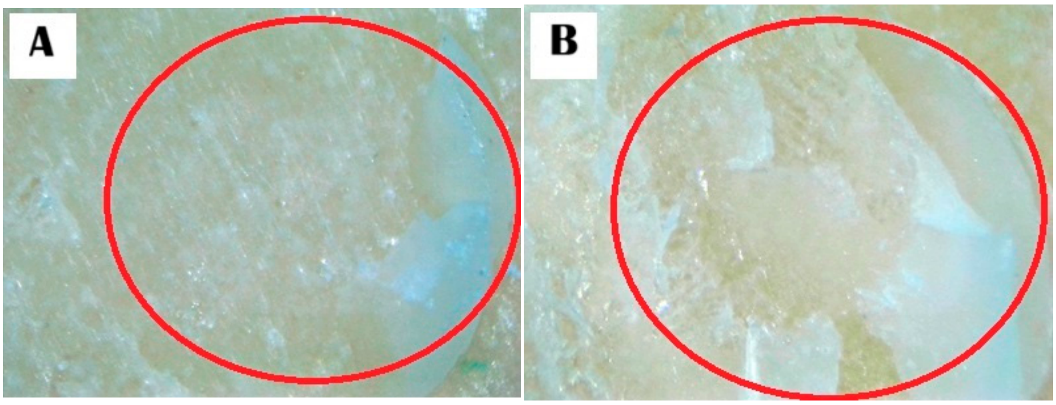

Figure 4. The stereomicroscope pictures for group 2: (**A**), mixed failure; (**B**), cohesive failure.

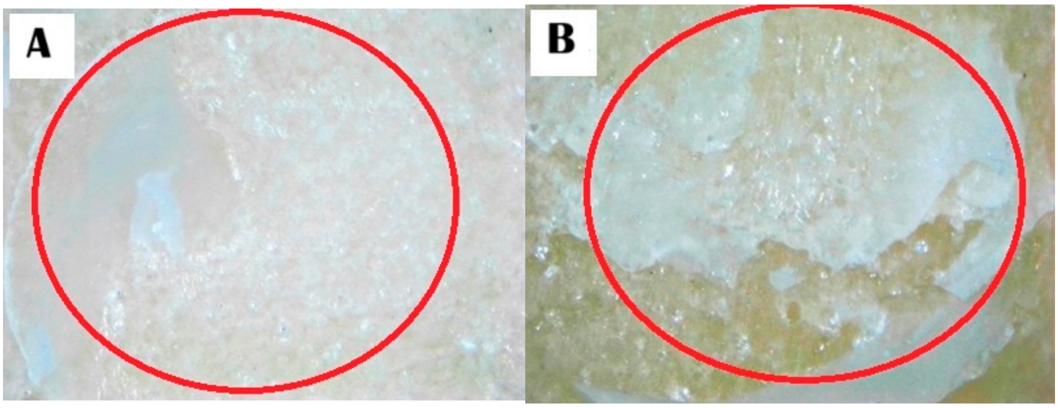

Figure 5. The stereomicroscope pictures for group 3: (**A**), mixed failure; (**B**), cohesive failure.

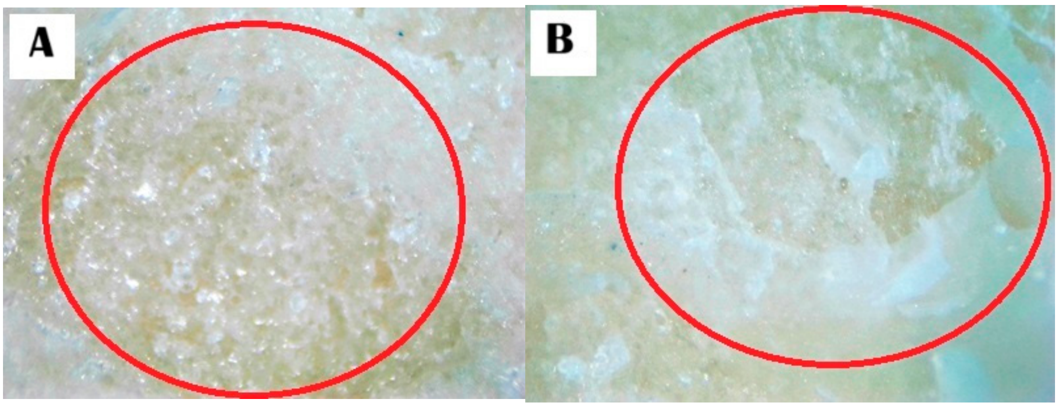

Figure 6. The stereomicroscope pictures for group 4; (**A**), mixed failure; (**B**), cohesive failure.

4. Discussion

The current study examines the effect of morpholine on saliva-contaminated acrylic resin repaired with light-cured resin composites by measuring the SBS. Significant differences in each group's SBS score were shown in the results. It has thus been established that the null hypothesis was false.

To establish effective and long-lasting adhesion between self-curing acrylic resin and resin composites, it is important to recognize the way different surface adjustments affect the manner in which these materials interact [4,6,29]. For clinical effectiveness, a powerful and dependable interaction between the self-curing acrylic resin and the resin composite is required [4,30]. The acrylic resin's surface roughness has to be increased in order to improve the mechanical adhesion. The SBS scores were higher after the sandblasting procedure than after the non-sandblasted surface treatment [31]. Conversely, Klaisiri et al. reported that when compared with non-sandblasted surfaces, no statistically significant increase in the bonding ability was seen after sandblasting treatment [4]. Furthermore, the surface roughness remained uniform throughout all groups since any deviations might have potentially affected the SBS results. A sufficiently effective MMA monomer and/or bonding agent must be used to prepare the sandblasted acrylic resin surface in order to boost the chemical bonds between the resin composite and the acrylic resin [4,32]. The potential repair bond ability of acrylic resin utilizing resin composites has been reported to be enhanced by a sandblasting method for micromechanical surface preparation and chemical surface alterations using MMA monomer and/or bonding agent systems [32,33]. Furthermore, Klaisiri et al. observed that the best repair bond ability is achieved when the MMA monomer is applied prior to the universal adhesive on sandblasted self-curing acrylic resin surfaces [4]. For this reason, the MMA monomer and universal adhesive were used to prepare the sandblasted self-curing acrylic resin surfaces in this experiment as the standard protocol.

In this investigation, surface contamination of sandblasted self-curing acrylic resin with saliva was conducted with artificial saliva. Many studies have reported that contaminated saliva causes a decrease in adhesive bonding ability [34,35]. However, some authors have indicated that the mucins in saliva have no effect on bonding ability [36]. Bolme et al. reported that decontaminating saliva with water and air spraying did not improve the bond strength when compared to that seen with saliva contamination [19]. According to Yin et al., phosphoric acid etching may be recommended for eliminating oral substances such as saliva and gingival fluid that contaminate restorative materials [37]. In the current study, the saliva on the sandblasted surfaces of the self-curing acrylic resin was cleaned using phosphoric acid, with phosphoric conditioning used prior to application of the bonding agent. Moreover, chemical agents such as phosphoric acid are used for salivary cleansing; the application period for salivary cleaning is usually around 30 s [38]. For this reason, phosphoric acid with a 30 s etching time was used for saliva decontamination as a positive control group (saliva + PH + MMA + UA), which had the statistically highest SBS values.

In our findings, group 4 (saliva + MR + MMA + UA) had the highest SBS values, but there was no significant difference in group 2 (saliva + PH + MMA + UA). Group 3 (saliva + MMA + UA) had lower SBS values when compared to groups 2 and 4. It was implied that group 3, comprising the sandblasted self-curing acrylic resin surfaces contaminated with saliva, showed a decreased bond strength when applying MMA and a universal adhesive agent, while group 4, with morpholine modification, showed increased bond strength on the saliva-contaminated surfaces. Additionally, when the sandblasted self-curing acrylic resin contaminated with saliva was conditioned with morpholine before the application of MMA and the universal adhesive agent, it was observed that the bond strength was high and equal to that in the positive control group, having had phosphoric conditioning prior to application of the MMA and universal adhesive agent. The three possible mechanisms by which morpholine enhances the SBS are as follows: (i) on the surfaces of self-curing acrylic resin that have been sandblasted, morpholine might be used to clean the surface, eliminate debris, and promote surface free energy [23]; (ii) morpholine has the potential to cause swelling and partial dissolution of the self-curing acrylic resin matrix [23]; and (iii) morpholine has been used as an initiator in the polymerization of MMA, and it may enhance the polymerization of MMA [25]. This study concluded that applying morpholine to the sandblasted self-curing acrylic resin that had been contaminated with saliva and repaired with light-cured resin composites improved its SBS before MMA and universal adhesive agents were applied.

The conclusions obtained from the examination of the debonded samples' breakdown processes and the outcomes of the SBS tests were in agreement. In this study, group 1 had adhesive fractures. The MMA- and universal-adhesive-treated groups, 2 through 4, revealed cohesive and mixed breakdowns and had higher SBS values. Furthermore, the cohesive failure rates increased in groups 2 and 4. A low bonding ability is frequently caused by adhesive failure, whereas mixed and cohesive failures point to greater adherence [4,23,28].

In clinical applications in dental clinics, when sandblasted self-curing acrylic resin surfaces are contaminated with saliva, morpholine is an alternative agent to phosphoric acid that could be used to decontaminate saliva before applying the MMA and bonding agent when repairing with light-cured resin composites. However, Klaisiri et al. found that the shear bonding ability did not show a statistically significant increase following sandblasting treatment as compared to the non-sandblasted surfaces [4].

This research study's focus on the use of self-curing acrylic resin limits its application to other dental materials, such as the heat-cured resins commonly used for denture bases. Determination of the self-curing acrylic resin and resin composites' SBSs could only be made by examining the specimens after 24 h of incubation after bonding. Future research could potentially apply thermocycling durability to assess the durability of the repairs made using resin composites and self-curing acrylic resin.

5. Conclusions

The present in vitro study's findings suggest that applying both phosphoric acid and morpholine to sandblasted self-curing acrylic resin contaminated with saliva before MMA and universal adhesive agents are applied is the most efficient protocol to stimulate its SBS when repairing with light-cured resin composites.

Author Contributions: A.K., N.K., N.T. and T.S. (i) conceived and designed the study; A.K., K.A., J.S., W.P., N.T. and T.S. (ii) performed the experiments and interpreted the results; A.K., N.K., K.A., J.S., W.P., N.T. and T.S. (iii) drafted the manuscript; and A.K., N.K. and T.S. (iv) revised the manuscript. All authors have read and agreed to the published version of the manuscript.

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