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Surface Modification Methods of Self-Cured Acrylic Resin Repaired with Resin Composite Using a Universal Adhesive

Awiruth Klaisiri [1,2], Apichai Maneenacarith [1], Nantawan Krajangta [1,2], Alysha Sukkee [3], Nannita Stephannie Hardy [3], Tuksadon Wutikhun [4] and Chayaporn Supachartwong [1,*]

[1] Division of Restorative Dentistry, Faculty of Dentistry, Thammasat University, Pathum Thani 12120, Thailand; dentton@hotmail.com (A.K.); apichai.man@dome.tu.ac.th (A.M.); knantawa@gmail.com (N.K.)
[2] Thammasat University Research Unit in Restorative and Esthetic Dentistry, Thammasat University, Pathum Thani 12120, Thailand
[3] Faculty of Dentistry, Thammasat University, Pathum Thani 12120, Thailand; alyshaswork@gmail.com (A.S.); nannita.hardy@yahoo.com (N.S.H.)
[4] National Nanotechnology Center, National Science and Technology Development Agency, Pathum Thani 12120, Thailand; tuksadon@nanotec.or.th
* Correspondence: c.supachartwong@gmail.com; Tel.: +66-29869051

Abstract: This research study's purpose was to evaluate the mechanical and chemical surface treatment methods for self-cured acrylic resin repaired with a resin composite employing a universal adhesive agent. Eighty self-cured acrylic resins were built and designed into eight groups of ten specimens and surface conditioned using sandblasting (SB) and/or with methylmethacrylate monomer (MMA) and/or universal adhesive (UA) as follows: Group 1, non-surface modified; Group 2, SB; Group 3, UA; Group 4, SB + UA; Group 5, MMA; Group 6, SB + MMA; Group 7, MMA + UA; Group 8, SB + MMA + UA. A template was put on the specimen center, and the pushed resin composites. Mechanical testing machinery was used to examine the samples' shear bond strength (SBS) values. To examine failure patterns, the debonded specimen surfaces were examined using a scanning electron microscope. The one-way ANOVA method was used to evaluate these data, and Tukey's test was used to determine the significance level ($p < 0.05$). The highest SBS was obtained in Group 8 (27.47 $\pm$ 2.15 MPa); however, it was statistically equivalent to Group 7 (25.85 $\pm$ 0.34 MPa). Group 1 (4.45 $\pm$ 0.46 MPa) had the lowest SBS, but it was not statistically significant compared to Group 2 (5.26 $\pm$ 0.92 MPa). High SBS values were frequently correlated with cohesive patterns. The application of MMA prior to UA is the best method for increasing the SBS between self-cured acrylic resin and resin composite interfaces. However, the use of SB is not significantly different from not using SB.

Keywords: adhesive agent; acrylic resin; bond strength; resin composite; surface treatment

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1. Introduction

Acrylic resin is an organic substance synthesized from ethylene polymers, which are created when polymethylmethacrylate (PMMA) and methylmethacrylate (MMA) mix together. This combination leads to the formation of a polymer that can be shaped into a pliable mass through the process of polymerization. This polymerization can be achieved either through chemical or thermal methods [1]. Self-curing acrylic resins, known for their exceptional versatility, find extensive applications in restorative dentistry and prosthodontics. These materials are invaluable in dental practice due to their adaptability and user-friendly nature. Their ability to set and harden without the need for additional curing techniques makes them convenient and time-efficient for dental professionals. As a result, self-curing acrylic resins have become indispensable components in various dental procedures, creating durable and aesthetically pleasing restorations and prosthetics with remarkable ease and effectiveness. Temporary restorations, such as crowns or bridges, can

be efficiently fabricated using these resins to provide interim solutions while permanent restorations are being prepared. Additionally, self-curing acrylic resins play a pivotal role in creating complete dentures, offering patients with missing teeth a functional and aesthetic replacement. Moreover, these acrylic resins are instrumental in prosthodontic implant rehabilitations, where they are used to fabricate implant-supported prostheses that restore oral function and appearance for patients with missing teeth [2].

Furthermore, acrylic resins are tasteless, ensuring patients experience minimal discomfort or undesirable taste sensations when using dental restorations or prostheses made from these materials. Additionally, acrylic resins exhibit excellent polishability, allowing dental professionals to achieve smooth and aesthetically pleasing surface finishes on restorations and prostheses. This polishability is particularly important for achieving natural-looking results and enhancing the appearance of dental restorations. Moreover, the acceptable thermal properties of acrylic resins contribute to their functional performance in the oral cavity. These resins can withstand the temperature variations experienced during eating and drinking without significant degradation, ensuring the longevity and stability of dental restorations and prostheses. Lastly, the ease of working with acrylic resins simplifies the fabrication process, making them highly convenient for dental professionals. Their ease of handling and manipulation allows for efficient and precise procedures, reducing chairside time and enhancing workflow in dental practice [1].

The temporary restoration could potentially experience fractures if the acrylic resin used for its fabrication lacks sufficient thickness. Intraoral fractures may also occur during dental appointments. In any event, when considering both cost and efficiency factors, the repair approach is favored over the refabrication of materials. Repairing the damaged restoration is the more economically viable and time-efficient option compared to completely refabricating it from scratch. However, for the repaired restoration to attain a high level of quality and longevity, it is imperative to achieve a strong and reliable bond between the two resin materials involved in the repair process. The success of this bonding interaction is critical for ensuring the restored structure's structural integrity and functionality, preventing any compromised performance or premature failure [3]. Two strategies have been provided to describe the adhesion of acrylic resin repaired with resin composites: (a) The first strategy involves the infiltration of monomers into the acrylic resin surface, which has been mechanically treated. This process results in the creation of micromechanical adhesion, wherein the monomer molecules penetrate into the surface irregularities and microstructures of the acrylic resin. This intimate interlocking of the monomers with the mechanically treated surface fosters a strong bonding interaction. (b) The second strategy revolves around the chemical attachment of monomers to both the acrylic resin and the resin material via reactive methacrylate groups. In this approach, the monomer molecules form covalent bonds with specific methacrylate sites present in both the acrylic resin and the resin composite. This chemical linkage enhances the adhesion between the two materials, generating a durable and stable interface. Such fractured acrylic resin could be repaired with composite materials [4,5]. The success of the adhesion bonds in this repair process is contingent upon a range of influencing factors. These factors encompass the particular type of acrylic resin employed, the surface treatment method applied to the acrylic resin, and the inherent properties of the resin composite material chosen for the repair [6].

The success of the repair process primarily relies on establishing a strong bond between the resin composite and the surface-treated acrylic resin [7]. Various protocols have been employed to enhance the bondability of acrylic resin and resin composites during repair procedures, encompassing different approaches such as mechanical and chemical surface treatments [5,8,9]. Despite numerous investigations and diligent efforts in this field, a clear and definitive consensus on the most effective approach has not yet been reached. Therefore, the purpose of this research study was to evaluate and compare the effectiveness of mechanical and chemical surface treatment methods for self-cured acrylic resin repaired with a resin composite employing a universal adhesive agent. The null hypothesis of this research is that there would be no significant difference in the shear bond strength (SBS)

values among the different surface treatment methods utilized to repair self-cured acrylic resin with a resin composite.

2. Materials and Methods

2.1. Preparation of Self-Cured Acrylic Resin Specimens

A total of eighty pieces of self-cured acrylic resin (Shofu Inc., Kyoto, Japan) were utilized in the investigation. These acrylic resin specimens were fabricated using a silicone mold, ensuring uniformity, with each specimen having a diameter of 6.0 mm and a thickness of 4.0 mm. The acrylic resins were handled with care as they were poured into the silicone molds, and then a self-curing procedure was allowed to take place for a period of ten minutes. After the curing procedure was complete, the acrylic resin rods in their solid form were obtained by removing the silicone molds. In order to prevent the rods of acrylic resin from becoming damaged during subsequent testing, type IV gypsum was used to attach them inside polyvinyl chloride tubes. The acrylic resin surfaces were sanded with 600-grit silicon carbide sandpaper (3M abrasive sheet, 3M, Saint Paul, MN, USA) for regularizing and standardizing the surface roughness. In order to clean the samples thoroughly and eliminate any contaminants, all specimens underwent a 10 min ultrasonic cleaning process in distilled water using an ultrasonic cleaner (WUC-D22H, DKSH, Singapore Pte Ltd., Singapore). Table 1 indicates the materials characterization that were utilized for this research.

Table 1. Materials characterization.

Material	Composition
Self-cured acrylic resin (Shofu Inc., Kyoto, Japan) Lot: 022264 (powder) Lot: 032299 (liquid)	Powder: MMA-EMA copolymer, pigments and others Liquid: MMA and others
Optibond universal adhesive (Kerr Corporation, Brea, CA, USA) Lot: 9208786	GPDM, GDM, HEMA, dimethacrylate, acetone, ethanol
Resin composite (Harmonize A4E shade, Kerr Corporation, Brea, CA, USA) Lot: 8609390	Bis-GMA, TEGDMA, EBPADMA, zirconia/silica cluster filler (2–3 µm) comprised of 20 nm spherical fumed silica and 5 nm zirconia particles, prepolymerized filler.

Abbreviations: MMA-EMA, methylmethacrylate-ethylmethacrylate; MMA, methylmethacrylate: GPDM, glycerol phosphate dimethacrylate; GDM, 1,3-glycerol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; Bis-GMA, bisphenol A-glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; EBPADMA, Ethoxylated bisphenol A dimethacrylate.

2.2. The Specimen Grouping

The acrylic resin specimens were distributed randomly into eight groups (n = 10 per group) and surface conditioned using sandblasting (SB), MMA monomer liquid, and/or universal adhesive agent (UA) according to the following protocols (Table 2).

2.3. Sandblast Technique

The specimens were sandblasted with 50-micron Al_2O_3 particles. This sandblasting procedure involved directing the Al_2O_3 particles toward the specimens from a distance of 10 mm. The sandblasting was carried out for a precisely timed duration of 10 s, and the process was conducted at a pressure of 2.8 bar [10]. Following the sandblasting process, these specimens were subjected to a sequential cleaning procedure. Initially, they were cleaned for 10 s using a combination of water and air, effectively eliminating any residual particles or contaminants. Subsequently, a 10 s air-drying step was performed using a triple syringe, ensuring a dry and pristine surface for further analysis or testing.

Table 2. Indicates the surface treatment protocols of the group of specimens.

Group	Surface Treatment Protocol
1	Non-surface-treated of self-cured acrylic resin
2	Self-cured acrylic resin treated only with sandblast (SB)
3	Self-cured acrylic resin treated only with universal adhesive agent (UA)
4	Self-cured acrylic resin treated with SB prior to application with UA (SB + UA)
5	Self-cured acrylic resin treated only with MMA monomer liquid (MMA)
6	Self-cured acrylic resin treated with SB prior to application with MMA monomer liquid (SB + MMA)
7	Self-cured acrylic resin treated with MMA monomer liquid prior to application with UA (MMA + UA)
8	Self-cured acrylic resin treated with SB prior to application with MMA monomer liquid and UA (SB + MMA + UA)

2.4. MMA Monomer Liquid Conditioning

The specimens were conditioned with MMA monomer liquid (Shofu Inc., Kyoto, Japan). The application of the monomer liquid was performed carefully for a duration of 1 min, using a single-use microbrush. After that, the specimens were allowed to undergo evaporation, ensuring the removal of any excess monomer and leaving them ready for subsequent procedures.

2.5. Universal Adhesive Conditioning

The specimens were conditioned with Optibond universal adhesive agent (Kerr Corporation, Brea, CA, USA). The application of the adhesive was carefully carried out using a single-use microbrush. After applying the adhesive, the excess universal adhesive was eliminated by another new single-use microbrush. Subsequently, the adhesive-coated specimens were subjected to an air-drying process for a duration of 10 s by a triple syringe and then light-polymerized for 20 s by an LED light curing instrument (Elipar Freelight 2, 3M ESPE, Saint Paul, MN, USA).

2.6. Application of Resin Composite

An ultradent mold with specific dimensions of 2.0 mm thickness and 2.0 mm diameter was positioned in the center of the sample that had undergone surface treatment. To fabricate the test specimen, a resin composite with an A4E shade (Harmonize, Kerr Corporation, Brea, CA, USA) was pushed into the mold and light-polymerized for 40 s by an LED light curing instrument. An ultradent mold was carefully removed from the specimen. To further enhance the polymerization process and ensure complete curing, the specimens were subjected to another 40 s light-polymerization cycle using the same LED light curing instrument (Figure 1). All specimens performed a one-day incubation process in 37-degree Celsius distilled water in an incubator (DI-150, Human Lab Inc., Gyeonggi-Do, Korea).

2.7. Shear Bond Strength (SBS) Value and Fracture Mode Pattern Examination

The SBS was measured with a universal testing tool (AGS-X 500 N, Shimadzu Corporation, Kyoto, Japan) and knife-edge shearing blade instrument at a test speed of 0.5 mm per minute (Figure 2). The cross-head loading force was placed close and parallel to the acrylic resin and resin composite interface until debonding. The SBS value was obtained by dividing the adhesion zone by the bond fracture force and recording it in megapascals (MPa).

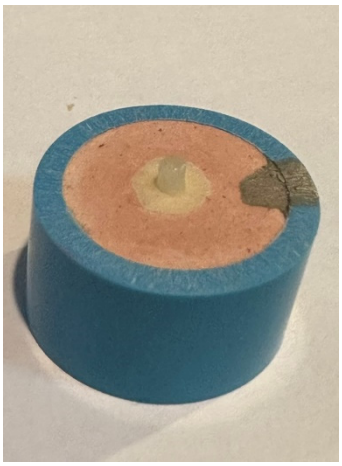

Figure 1. The bonded specimen.

Figure 2. The knife-edge SBS test.

The fracture failure mode patterns of both self-cured acrylic resin and resin composites were subjected to detailed examination using a scanning electron microscope (SEM) with a magnification of ×1000 (Versa 3D, FEI Company, Hillsboro, OR, USA). Three patterns were used to classify the fracture failure modes [11–14]:

(a) An adhesive failure mode pattern when broken at the junction between acrylic resin and resin composite. This happens when there are no resin composite residues on the acrylic resin surface.
(b) A cohesive failure mode pattern when broken within acrylic resin or resin composite
(c) A mixed failure mode pattern when combined with adhesive failure mode patterns and cohesive failure mode patterns.

2.8. Data Analysis

The normality of collected data was assessed using the Kolmogorov–Smirnov test. To evaluate the homogeneity of variance within each group, Levene's test was employed, specifically for data that exhibited a normal distribution. These collected data were subjected to thorough analysis using the one-way analysis of variance (ANOVA) method. To determine the statistical significance, a predetermined significance level of $p < 0.05$ was applied, and a further pairwise comparison of means was conducted using a post hoc Tukey test.

3. Results

Table 3 provides the mean SBS and standard deviation (SD). The significantly highest shear bond strength values were exhibited in Groups 7 (25.85 ± 0.34 MPa) and 8 (27.47 ± 2.15 MPa). Meanwhile, Groups 1 (4.45 ± 0.46 MPa) and 2 (5.26 ± 0.92 MPa) found the significantly lowest shear bond strength values. Group 3 (18.11 ± 1.11 MPa) and Group 4 (15.63 ± 1.67 MPa) exhibited no significant difference in shear bond strength values. In the same way, Group 5 (11.20 ± 1.30 MPa) and Group 6 (12.24 ± 1.96 MPa) showed no significant difference in shear bond strength values. The SB groups are not significantly different when they are used compared to when they are not.

Table 3. The mean SBS ± SD and percentage of failure mode pattern.

Groups	Mean SBS ± SD	Percentage of Failure Mode		
		Adhesive	Mixed	Cohesive
1. No treatment	4.45 ± 0.46 [a]	100	0	0
2. SB	5.26 ± 0.92 [a]	100	0	0
3. UA	18.11 ± 1.11 [b]	10	40	50
4. SB + UA	15.63 ± 1.67 [b]	10	40	50
5. MMA	11.20 ± 1.30 [c]	30	60	10
6. SB + MMA	12.24 ± 1.96 [c]	20	60	20
7. MMA + UA	25.85 ± 0.34 [d]	0	30	70
8. SB + MMA + UA	27.47 ± 2.15 [d]	0	40	60

The value with identical letters indicates no statistically significant difference.

The pattern of the distribution of failure modes observed in the study is shown in Table 3. After being debonded, all debonded samples in Groups 1 and 2 were classified as having an adhesive failure pattern. In addition, in Groups 3 to 8, mixed and cohesive failure mode patterns increased. In Groups 5 and 6, the most common failure pattern is mixed failure at 60% and 60%, respectively. In Groups 3, 4, 7, and 8, the most frequent failure pattern observed was the cohesive failure, constituting 50%, 50%, 70%, and 60% of the respective cases.

In this part of the SEM analysis, SEM pictures of examples of adhesive, mixed, and cohesive failure mode patterns are shown in Figures 3–6. Group 1 and Group 2 show an adhesive failure pattern (Figure 3). Group 3 and Group 4 show a cohesive failure pattern in the acrylic resin (Figure 4). Group 5 and Group 6 show a mixed failure pattern (Figure 5). Group 7 and Group 8 show a cohesive failure pattern in the resin composite (Figure 6).

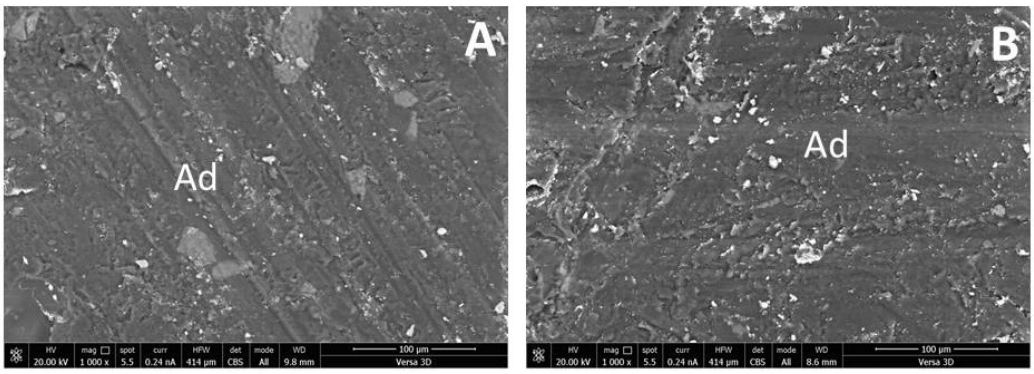

Figure 3. SEM pictures showing adhesive failure: (**A**) Group 1 (no treatment); (**B**) Group 2 (SB). (Ad, adhesive failure).

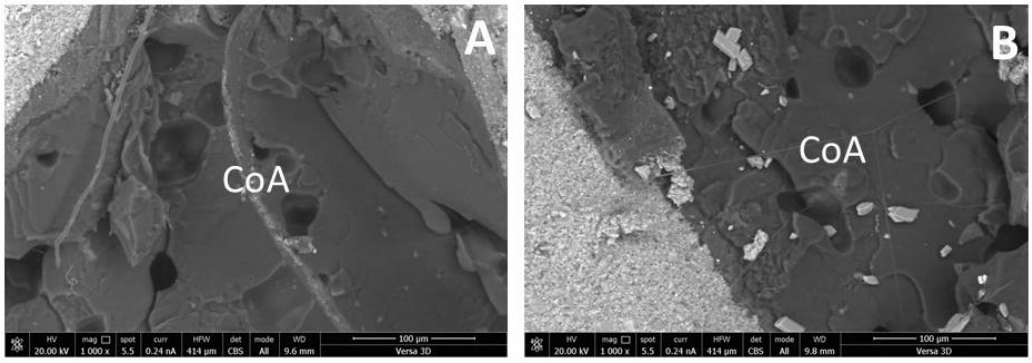

Figure 4. SEM pictures showing cohesive failure in acrylic resin: (**A**) Group 3 (UA); (**B**) Group 4 (SB + UA). (CoA, cohesive failure in acrylic resin).

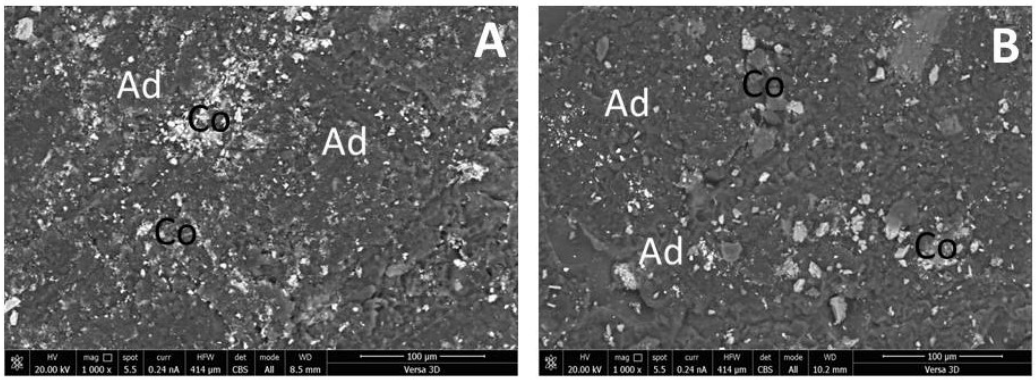

Figure 5. SEM pictures showing mixed failure: (**A**) Group 5 (MMA); (**B**) Group 6 (SB + MMA). (Ad, adhesive failure; Co, cohesive failure).

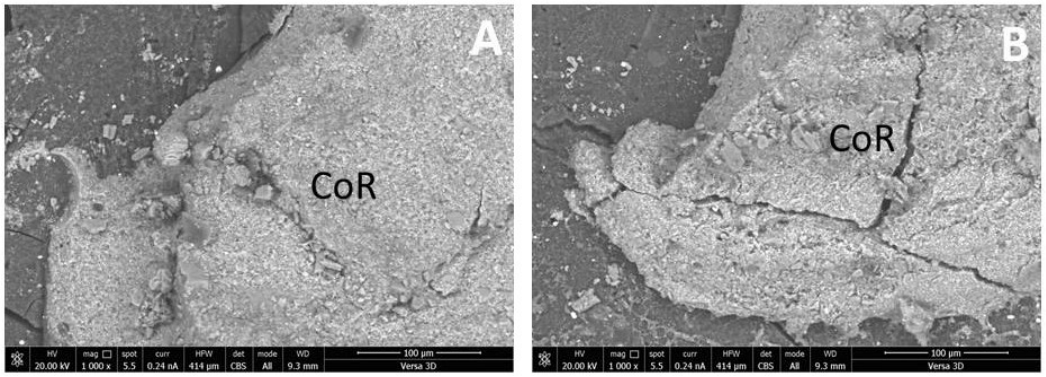

Figure 6. SEM pictures showing cohesive failure in resin composite: (**A**) Group 7 (MMA + UA); (**B**) Group 8 (SB + MMA + UA). (CoR, cohesive failure in resin composite).

4. Discussion

The primary objective of this research was to assess the efficacy of mechanical and chemical surface modification techniques on self-cured acrylic resin, specifically when it is repaired using resin composite materials. The obtained results of the present in vitro investigation highlighted the benefits of universal adhesive agents in promoting mechanical and chemical adhesion between self-cured acrylic resin and resin composites combined with MMA monomer surface treatment. The findings from this study demonstrate that the SBS values of all the experimental groups exhibited significant variations. As a consequence, the null hypothesis, which assumed no significant differences between the groups, was convincingly rejected.

It is essential to know how different surface modifications influence how these materials interact in order to create a strong and durable connection between acrylic resin and resin composites [5,8,9]. The acrylic resin and resin composite must be bonded to one another in a reliable and powerful method for clinical efficacy [6,15]. To increase mechanical adhesion, the surface roughness of acrylic resin must be improved. In comparison to no surface treatment, the sandblasting method raised the SBS values [16]. To enhance chemical bonding between the acrylic resin and resin composite, apply an adhesive agent for conditioning the acrylic resin surface that is efficient enough [9,16]. It has been suggested that a variety of micromechanical surface modification techniques and chemical surface treatment through adhesive systems can improve the ability to repair bonds in acrylic resin repaired using resin composite [16–18]. Additionally, surface treatment of acrylic resin with MMA (methyl methacrylate) has been shown to enhance the bonding ability of acrylic resin when repaired with resin composite [5,19–23].

Based on the obtained results, it was observed that the sandblasting protocol (5.26 $\pm$ 0.92 MPa) did not lead to a statistically significant increase in the shear bond strength (SBS) values compared to the non-sandblasted surface (4.45 $\pm$ 0.46 MPa). The findings from this investigation clearly demonstrated that sandblasting alone, without any chemical surface conditioning, resulted in poor adhesion between the self-cured acrylic resin and resin composites. This highlights the fact that achieving a strong and reliable bond between self-cured acrylic resin and resin composites requires more than just relying on micromechanical retention provided by sandblasting. In view of these findings, it is clear that additional approaches are required to improve the SBS between self-cured acrylic resin and resin composites. Notably, the incorporation of MMA monomers and/or adhesives appears promising for enhancing bond strength [19].

Polymerized acrylic resins are mainly composed of PMMA. The MMA monomer appears to promote acrylic resin polymer swelling and improve monomer dispersion within the bulk of polymerized acrylic resin [19]. The underlying principle behind this phenomenon lies in the ability of the MMA monomer to dissolve the surface of PMMA present in the polymerized acrylic resin. This dissolution process leads to an improved connection between the various resin components, creating a more cohesive and homogenous structure [19–21]. Additionally, Qaw et al. [6] found that an MMA-based agent demonstrated chemical adhesion to the active carbon double bond present on the surface of the resin substrate [24]. This research exhibited that the use of MMA monomer liquid applied to a self-cured acrylic resin surface (11.20 $\pm$ 1.30 MPa) prior to repair with resin composite increased the SBS when compared to no surface treatment (4.45 $\pm$ 0.46 MPa). A study by Vergani et al. [20] also supported these findings; treating the acrylic resin surface with MMA liquid monomer will lead to the dissolution of the PMMA and improve the bonding capacity of repair materials. This is in accordance with previous studies [5,19–23], which consistently reported higher SBS values following the application of MMA monomer compared to non-surface-treated specimens.

The quality of the bond between the acrylic resin and the repair or restoration is significantly improved after the surface of the acrylic resin has been pretreated with adhesive agents. Because adhesive agents are used, the surface of the self-curing acrylic resin is made more wettable. This is because the adhesive successfully infiltrates and polymerizes inside the surface porosity. Micromechanical adhesion is established as a result of this process, which further strengthens the overall strength and stability of the repaired interface [5]. Additionally, it is possible for the adhesive's dimethacrylate monomers to chemically connect with the PMMA acrylic resin through the action of reactive methacrylate groups, thereby forming a covalent bond between the two materials. [25]. Muhsin [5] discovered that adding a bonding agent to acrylic resin gave it enough active sites to interact with the resin composite. Furthermore, Alshali et al. [26] conducted a study revealing that the utilization of a UA enhanced the ability to repair bonds between self-cured acrylic resin and resin composites. According to these outcomes, SBS between self-cured acrylic resin and resin composite was affected by the application of universal adhesive (18.11 $\pm$ 1.11 MPa)

prior to repair with resin composite; consequently, SBS was enhanced compared with no surface treatment (4.45 ± 0.46 MPa). A universal adhesive agent worked better than MMA monomer surface treatment as an agent to modify the surface of acrylic resin. The application of the universal adhesive agent is an essential step in resin composite repairs of self-cured acrylic resins.

Based on the obtained results, the use of MMA monomer and UA-treated acrylic resin surfaces (25.85 ± 0.34 MPa) had the highest SBS compared with MMA monomer-treated only (11.20 ± 1.30 MPa) and UA-treated only (18.11 ± 1.11 MPa) resins. This result was supported by SEM examination of the failure mode pattern, which revealed cohesive failure to be more prevalent than adhesive failure, which was seen on the non-treated surface of the specimen. According to Muhsin [5], Bähr et al. [19], and Vergani et al. [20], superior bonding strength was achieved on acrylic resin surfaces treated with MMA monomers and adhesives. The strongest SBS between self-cured acrylic resin and resin composite was achieved by first applying MMA monomer to the surface of the self-cured acrylic resin, followed by the utilization of a universal adhesive agent. The MMA monomer liquid and universal adhesive combination appear to exhibit superior linking bonding ability. The MMA monomer might be copolymerized with the amphiphilic monomer of a universal adhesive agent. This might be created by the diffusing and polymerizing MMA monomer and universal adhesive over the self-cured acrylic resin and resin composite interface to generate an interpenetrating polymer set of linkages [21].

In certain aspects of the observed failure pattern. Three patterns were used to classify the fracture failure modes [11–14]: (a) an adhesive failure mode pattern when broken at the junction between acrylic resin and resin composite, (b) a cohesive failure mode pattern when broken within the acrylic resin or resin composite, and (c) a mixed failure mode pattern when combined with adhesive failure mode patterns and cohesive failure mode patterns. It was noted that all specimens belonging to Groups 1 and 2 exhibited adhesive failure. Additionally, in Groups 3 to 8, there was a noticeable increase in the occurrence of mixed and cohesive failure mode patterns. In Groups 3, 4, 7, and 8, cohesive failure patterns were frequently correlated with high SBS. The total number of cohesive patterns and bond strength were directly correlated; as bond strength increased, so did the frequency of cohesive patterns [27]. The repair will be more effective the closer the bond strength value is to the cohesive failure repair strength of the resin material [26].

In the clinical application of this research, an alternate technique in clinical practice is the use of MMA to modify the surface of the self-cured acrylic resin before using the universal adhesive agent to repair it with resin composite. The SBS of resin composites and self-cured acrylic resin repairs were successfully increased by this technique.

This research study's design, which focused on the use of just one universal adhesive, meant that it was limited in that it could not be applied to other universal and conventional adhesives. Only 24 h after bonding could the incubated specimen determine the acrylic resin and resin composites' SBSs. In the future, thermocycling may be employed to evaluate the durability of acrylic resin and resin composites. The SBS is just one factor that affects how well an adhesion technique performs in a clinical situation. Therefore, it is important to carefully analyze the results of our inquiry.

5. Conclusions

Under the scope of this research's conditions, the outcomes of this investigation clearly demonstrated that the micro-mechanical retention of the sandblasting alone, without any chemical surface conditioning, resulted in poor adhesion between the self-cured acrylic resin and resin composites. The use of MMA monomer liquid applied prior to UA is the best method for excellently increasing the SBS of self-cured acrylic resin repaired with resin composite. However, the use of SB is not significantly different from not using SB.

Author Contributions: A.K., A.M., N.K. and C.S. conceived and designed the study; A.K., A.M., A.S., N.S.H. and T.W. performed the experiments and drafted the manuscript; A.K., A.M., N.K. and C.S. interpreted the experimental results; A.K., A.M., A.S., N.S.H. and T.W. revised the manuscript. All authors have read and agreed to the published version of the manuscript.

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