Original Article

In vitro evaluation of an easy-to-remove orthodontic adhesive with photochromic property

Jiarong Yan[a]; Lingyun Cao[b]; Ting Luo[a]; Fang Hua[c]; Hong He[d]

ABSTRACT

Objectives: To develop a photochromic bracket adhesive (PCA) with modification using photochromic material and evaluate the biocompatibility, bond strength, photochromic property, and adhesive removal efficiency.

Materials and Methods: The resin-modified glass ionomer powder was mixed with the photochromic material and then blended with the liquid agent to form PCA. Biocompatibility was evaluated by CCK-8 kit, and shear bond strength (SBS) was measured. Stereoscopic microscopy and quantitative color analysis were used to assess the photochromic property. Bracket bonding and debonding procedures were performed on a head simulator with the assistance of an ultraviolet radiator. The effectiveness of adhesive removal during bonding and debonding procedures was assessed using a stereomicroscope. Removal time was recorded, and the enamel damage index after debonding was analyzed.

Results: CCK-8 assay and SBS test indicated that 5wt.% mixing ratios of the photochromic material did not compromise the biocompatibility and SBS of the adhesive (PCA5). PCA5 showed photochromic properties and could help the operator remove adhesive more thoroughly without increasing enamel damage.

Conclusions: Photochromic adhesive (PCA5) can be good for orthodontic adhesive removal and therefore has good clinical translation potential. (*Angle Orthod*. 2024;94:200–206.)

KEY WORDS: Adhesive; Ultraviolet; Photochromic; Enamel

INTRODUCTION

Fixed appliances have been widely used in orthodontics due to their good biomechanical properties.[1] After treatment, brackets are debonded from the enamel. Orthodontic bracket adhesives have similar optical properties to enamel, making them challenging to identify.[2] Excess adhesive leads to bacterial adhesion and consequently facilitates the formation of white spot lesions (WSLs) and may also be stained and affect esthetics. After debonding, due to the similar appearance, it is difficult to remove the remnant adhesive

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Table 1. Composition of the Photochromic Material

Compound	CAS No.
6'-(Indolin-1-yl)-1,3,3-trimethylspiro[indoline-2, 3'-naphtho[2,1-b][1,4]oxazine]	114747-44-3
3,3-Diphenyl-3H-naphtho[2,1-b]pyran	4222-20-2
Polyoxymethylenemelamine	9003-08-1
Styrene maleic anhydride monomethyl-maleate polymer	31959-78-1
4-(1-phenylethyl)-o-xylen	6196-95-8
Mineral oil	8012-95-1

precisely.[3] Excessive removal leads to irreversible and iatrogenic damage to the enamel,[4] while incomplete removal results in biofilm accumulation and staining.[5]

Recently, a fluorescence-aided identification technique (FIT) has been applied to distinguish the resin from teeth.[6,7] As teeth and resin have different fluorescent properties under ultraviolet (UV) light, FIT is a noninvasive and accurate method for restorative and adhesive resin detection.[8,9] Studies have explored the use of FIT for bracket adhesive removal during the debonding procedure and found that remnant adhesives can be removed more efficiently with the help of FIT.[10,11] However, FIT has shortcomings. The weak fluorescent properties of the resin make it possible to distinguish the resin from the enamel only in a dark environment,[9] and as a short-wavelength light, the UV light would cause iatrogenic damage to the eyes.[12] In addition, FIT technology cannot be used to identify excess adhesive during the bracket bonding procedure, as UV light can cause the adhesive to cure. The accurate identification and removal of bracket adhesives from the enamel is still a challenge awaiting a solution.

Photochromic materials are a family of compounds that have reversible structure that switches between two different states with different colors when excited by light.[13] They have attracted considerable attention in photoactive devices such as optical memories and switches, photosensitive glasses, and photochromic decorations.[14] Therefore, photochromic material could provide the adhesive photochromic properties to mark adhesives when needed and thus help orthodontists remove excess and remnant adhesive more efficiently.

The objectives of this study were to develop a photochromic bracket adhesive (PCA) based on resin-modified glass ionomer (RMGI) cement, which consists of powder and liquid agents and thus easy to modify. Then, the biocompatibility, bonding strength, and adhesive removal efficiency of PCA was evaluated. The hypotheses of the study were PCA (1) would not show acceptable biocompatibility, (2) would not demonstrate acceptable bond strength, (3) could not be removed more thoroughly than RMGI, and (4) could not be removed more rapidly than RMGI.

MATERIALS AND METHODS

Preparation of PCA

RMGI cement (Fuji Ortho LC, GC, Tokyo, Japan) was used as the parent adhesive system. A photochromic material (Green by Light, Huancaibs, Shenzheng, China; Table 1) was used. The major components of the photochromic material could have their structure changed under UV radiation (Figure 1). The photochromic material was mixed into the powder of RMGI to form the PCA powder, and the mixing ratios were 1wt.% (group-1%), 2.5wt.% (group-2.5%), 5wt.% (group-5%), 10wt.% (group-10%), 15wt.% (group-15%), and 20wt.% (group-20%). The powder was then mixed with the liquid agent of the RMGI to form the PCA. RMGI cement without any addition served as the control (group-0%).

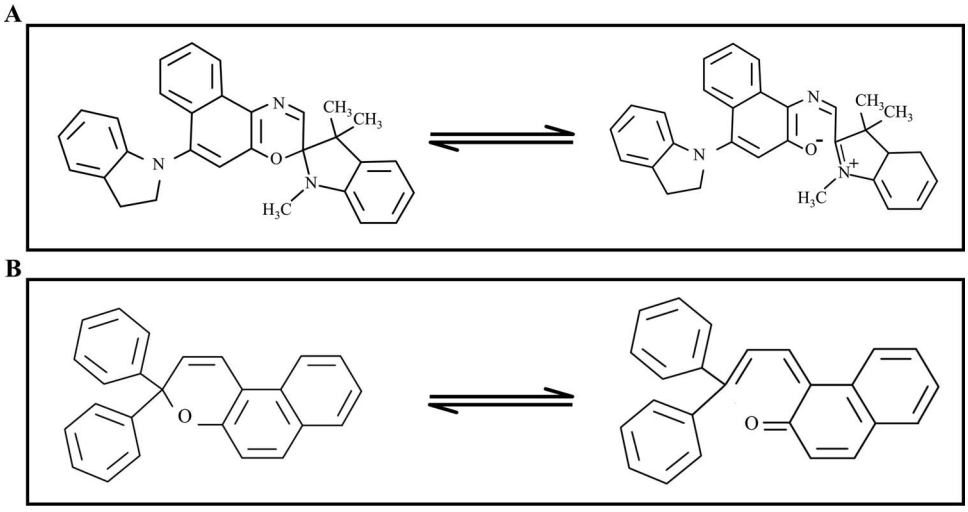

Figure 1. Photochromic reaction formulas of (A) 6'-(indolin-1-yl)-1,3,3-trimethylspiro[indoline-2,3'-naphtho[2,1-b] [1,4] oxazine] and (B) 3,3-diphenyl-3H-naphtho[2,1-b] pyran.

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Cytocompatibility Evaluation

The adhesive was filled into the caps of 0.5 mL Eppendorf tubes and cured for 30s on each side using a light-cure system (Bluephase 800 mW/cm^2 430–490 nm, Ivoclar-Vivadent Amherst, NY). Adhesive disks of all groups (n = 6) were incubated in alpha-Modified Eagle's Medium (Gibco, Grand Island, NY) with 10% fetal bovine serum (Gibco) for 24 hours at 37°C, 5% CO_2 to obtain extracts.[15] The ratio of extraction was set as 3 cm^2/mL, according to the ISO standard 10993-12:2012. Human gingival fibroblasts (HGFBs; ScienCell, Carlsbad, Calif) were seeded in a 96-well plate and extracts of all groups were dropped (100μL per well). HGFBs with pure culture medium served as the natural culture group (NC group). After 24h, 10μL CCK-8 solution (CCK-8 kit, Beyotime, Shanghai, China) was dropped into each well of the 96-well plate and incubated for another 2 hours.[16] Absorbance of wells at 450 nm were obtained by a spectrophotometer (Powerwave-340, Bio-Tek Instruments, Winooski, Vt).

Shear Bond Strength Test

Intact human premolars were collected after obtaining ethics approval (No. 2019-A65, Ethics Committee for Human Studies of the School and Hospital of Stomatology, Wuhan University). Premolars were randomly divided into immediate (n = 105) and long-term groups (n = 60). Brackets (metal bracket of upper first premolar, GAC, Greensboro, NC) were bonded onto the center of the clinical crown with the adhesive in all groups (n = 15 for each group) following the instructions. After incubating teeth in deionized water at 37°C for 24 hours, a universal testing machine (E1000, Instron, Norwood, Mass) was used to measure the shear bond strength (SBS) with parameters set to 0.5 mm/min measurement speed, in the occlusogingival direction. The maximum shear force was recorded to calculate the SBS using the following formula: $Shear\ bond\ strength = F_{MAX}/S_{bracket}$.

Based on the immediate SBS results, a long-term SBS test was conducted on group-1%, group-2.5%, and group-5% (n = 15). A thermocycler method was used for aging. Samples were thermally cycled from 5°C to 55°C for 5000 cycles, with a 30-second immersion time.[17] After aging, the SBS of each sample was measured.

Adhesive Remnant Index Analysis

After SBS tests, the enamel surface of each sample was observed using a stereomicroscope (2× magnification; Stereozoom S9D, Leica, Weztlar, Germany) with a light source (Leica LED3000 SLI), and the Adhesive Remnant Index (ARI) was recorded according to the following scores:

0 = No adhesive remaining on the enamel
1 = Less than half of the adhesive remaining on the enamel
2 = More than half of the adhesive remaining on the enamel
3 = All adhesive remaining on the enamel.

Photochromic Performance Evaluation

According to the cytocompatibility and SBS results, group-5% was selected as the final experimental photochromic adhesive (PCA5). Bonded tooth samples of PCA5 and RMGI were observed. After debonding the brackets, remnant adhesive on the enamel was observed under natural light, mixed light (natural light mixed with UV light), and after UV irradiation. The bonded tooth sample was irradiated by UV light for 3 seconds from a distance of 5 mm and videotaped. Quantitative color analysis was performed using the *Lab* color mode of Photoshop software on the images of the adhesive before and after irradiation. The difference between the color of PCA5 at different points and its base color was quantified using the following formula[18]: $\triangle E = ((\triangle L)^2 + (\triangle a)^2 + (\triangle b)^2)^{1/2}$.

Excess Adhesive Analysis

Forty premolars were collected and randomly divided into RMGI and PCA5 groups (n = 20).[10] The morphology of enamel was obtained by the stereomicroscope. The premolar was then mounted into the left upper first premolar socket of a head simulator (JG-C5, Jinglemed, Guangdong, China).[10] Brackets were bonded onto the center of the clinical crown of the premolars by the same operator (YJR), and the time taken to remove the excess adhesive was recorded. After curing, photographs of the bonded tooth samples were taken. Image J was used to measure the uncleaned area of the excess adhesive.

Remnant Adhesive Analysis

For the remnant adhesive analysis, the bracket was debonded and the tooth was mounted back into the socket. The procedure to remove the remnant adhesive was performed by the same operator (YJR) to maintain uniform adhesive removal forces using a tungsten-carbide bur (CBR1S 016, Germany DENT, Tautenhain, Germany), and the removal time was recorded. For the RMGI group, the remnant adhesives were removed under conventional lighting from a dental chair unit. For the PCA5 group, the enamel surface was exposed to a UV light (AB-H100, Desert Star 15W, Guangdong, China) for 3 seconds, and then the removal procedure was performed under conventional lighting. A new tungsten-carbide bur was used after every 10 samples. After the removal procedure, enamel surfaces

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were observed using the stereomicroscope, and the unremoved area of the remnant adhesive was measured by Image J software.

Enamel Damage Index Analysis

Enamel damage was evaluated through the stereomicroscope observation, and the Enamel Damage Index of each enamel surface before the bonding procedure and after removing the remnant adhesive was recorded according to the following:

0 = Smooth surface without scratches, visible perikymata
1 = Acceptable surface with fine scratches spread out
2 = Rough surface with several rough scratches or visible minor grooves
3 = Surface with rough scratches, large grooves, and enamel damage visible to the naked eye

The enamel damage caused by the removal procedure for remnant adhesive was calculated by subtracting the before-bonding score from the after-removal score.

Statistical Analysis

CCK-8 assay, SBS, adhesive residual area, and adhesive removal time were shown in mean ± SD and analyzed using one-way analysis of variance and Tukey's multiple comparison tests. The significance level for all tests was set at $\alpha = .05$.

RESULTS

Cytocompatibility

The CCK-8 assay (Figure 2) showed that, except for the group-20%, the absorbances of all other adhesive groups were not statistically different from that of the NC group ($P > .05$ for all groups). The group-20% had significantly lower absorbance compared with the NC group ($P < .05$).

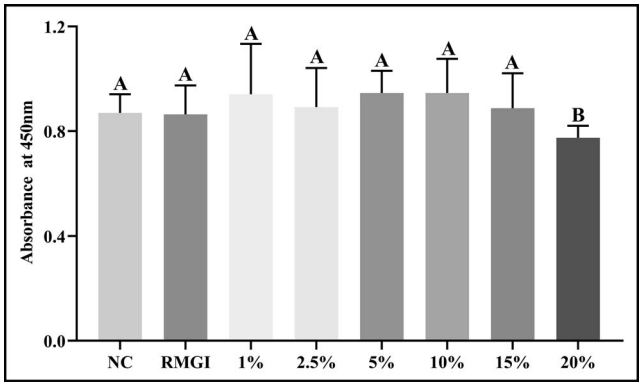

Figure 2. Cytocompatibility evaluation of adhesives. Bars with the same letter are not significantly different ($P > .05$).

SBS and ARI

Group-1%, group-2.5%, and group-5% showed no statistically significant difference in the immediate SBS compared with the RMGI group ($P > .05$ for all groups), while group-10%, group-15%, and group-20% had lower SBS ($P < .05$ for all groups) (Figure 3A). After aging, the SBS of all PCA groups was still comparable with that of the RMGI group ($P > .05$ for all groups; Figure 3B). PCA5 had similar adhesive remnant profiles as RMGI after immediate and long-term SBS tests (Figure 3C).

Photochromic Performance

During the bonding stage, RMGI had an appearance similar to the enamel, while PCA5 appeared green and could be easily distinguished from enamel (Figure 4A). After curing, both RMGI and PCA5 had an appearance similar to enamel. Under UV irradiation, the RMGI showed a faint purple fluorescence, while the PCA5 turned green. After UV irradiation, the fluorescence of RMGI disappeared, while PCA5 maintained its green appearance. After UV irradiation, the color of PCA5 gradually returned (Figure 4B). The results of the quantitative color analysis showed a consistent trend: the value of △E decreased continuously and, at the time point of 60 seconds, the value was only slightly above the limit of the color difference that can be distinguished by the naked eye.

Adhesive Removal Effect

During the bonding procedure, the uncleaned area of excess PCA5 was significantly smaller than that of RMGI ($P < .05$). After debonding and removing adhesive, the unremoved area of remnant PCA5 on the enamel surface was significantly smaller than that of RMGI ($P < .05$; Figure 5A). PCA5 took a longer time to remove for both excess and remnant adhesives ($P < .05$ for excess and remnant adhesives; Figure 5B). The enamel damage of the PCA5 group was similar to that of the RMGI group (Figure 5C).

DISCUSSION

In this study, a photochromic bracket adhesive (PCA5) was constructed to make excess and remnant adhesives recognizable when needed. PCA5 had acceptable cytocompatibility and SBS. The photochromic ability of PCA5 allowed excess and remnant adhesive to be removed more thoroughly, while the time for removal was longer compared with RMGI. Therefore, the first, second, and third hypotheses were rejected, and the fourth hypothesis could not be rejected.

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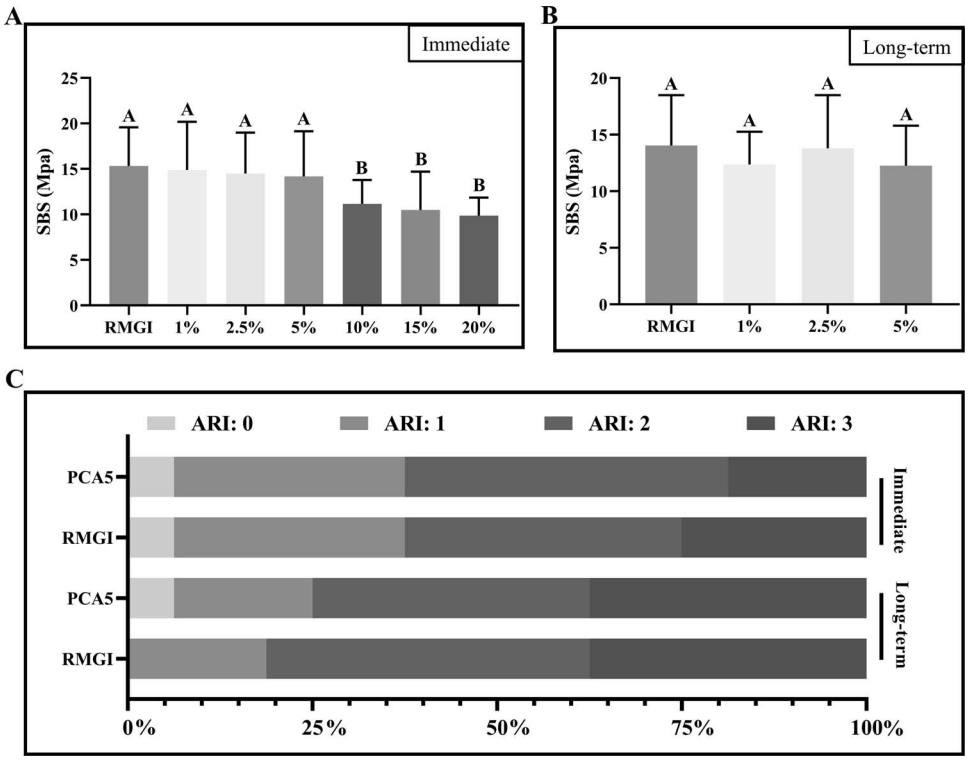

Figure 3. Immediate adhesive SBS (A), long-term SBS (B), and ARI (C) results. Bars with the same letter are not significantly different ($P > .05$).

HGFBs were chosen, and the adhesive extracts were obtained for biocompatibility evaluation as the adhesive is most likely to come into indirect contact with the gingival tissue through saliva. The absorbance at 450 nm of the CCK-8 assay represents the vitality of the cells.[19] The CCK-8 evaluation showed that all of the PCAs had no cytotoxicity to HGFBs, except for the group-20%.

Bracket adhesives require sufficient bond strength to resist masticatory loads during daily life. The immediate SBS of PCAs limited the mixing ratio of photochromic materials to 5%. For the long-term SBS test, the thermocycling aging method was chosen to simulate aging of the adhesive in the oral environment.[20] After aging, all three PCA groups exhibited satisfactory SBS. Based on the results of biocompatibility and SBS, PCA5 was chosen as the final experimental adhesive for the follow-up evaluations.

Photochromic compounds can undergo structural changes under the excitation of light, and the change in structure leads to a significant change in its absorption spectrum, resulting in a different appearance.[21] 6'-(Indolin-1-yl)-1,3,3-trimethylspiro[indoline-2,3'-naphtho[2,1-b][1,4]oxazine] and 3,3-diphenyl-3H-naphtho[2,1-b]pyran are spirooxazine compounds[22] and pyranoid compounds,[23] respectively. Under the excitation of UV light, both compounds undergo bond breaking and recombination (Figure 1), thus showing different colors. The former changes from white to blue, and the latter changes from white to yellow; therefore, the joint action of these two chemicals eventually achieves the green photochromic effect. After UV irradiation, the compounds gradually regain their original structures under the effect of temperature, and their colors also return. During the blending process, PCA5 gradually changed from white to green (Figure 4A). This phenomenon may be due to the chemical reaction between the photochromic material and the liquid agent of RMGI cement, similar to the photochromic reaction. The light excitation during the light curing process causes the photochromic material to regain its structure of white, thus allowing PCA5 to regain its original color (Figure 4A). After removing the bracket, the remnant PAC5 could turn green when excited by UV light and thus could be distinguished from the enamel. Since photochromic compounds are short-chain organics and the mixing ratios are low, combined with the SBS results, it was thought that PCA5 will not affect the property of the enamel and bond strength. However, the effect of the photochromic compound on the release of fluoride from RMGI requires further investigation.

A ΔE value superior to 3.3 is appreciable by the naked eye.[24] Quantitative color analysis showed that PCA5 still exhibited naked eye–visible color change at 60 seconds after UV excitation. However, in practice, it became difficult to distinguish PCA5 from the enamel at about 30 seconds after UV irradiation. Therefore, irradiation of the adhesive with UV light would be required several times during the remnant adhesive removal procedure to ensure complete removal. This may have been one of

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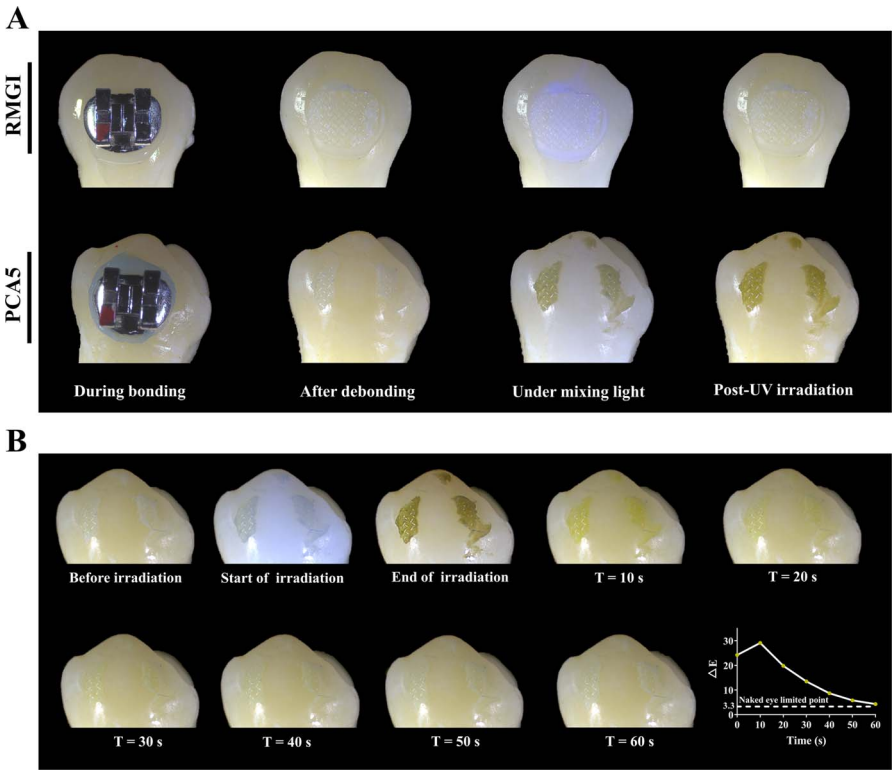

Figure 4. Color appearance of RMGI and PCA5 under different light conditions (A) and photochromic performance of PCA5 (B).

the reasons that the PCA5 removal time was longer during debonding. Another reason was that the photochromic effect caused a more careful and thorough removal of PAC5 compared with the RMGI. PCA5 can be removed more completely during the bracket bonding procedure and the remnant adhesive removal procedure, thus reducing plaque adhesion on the enamel and avoiding the adhesive staining problem.

Due to having a similar hardness to enamel, the tungsten-carbide bur is a suitable tool for removing adhesive

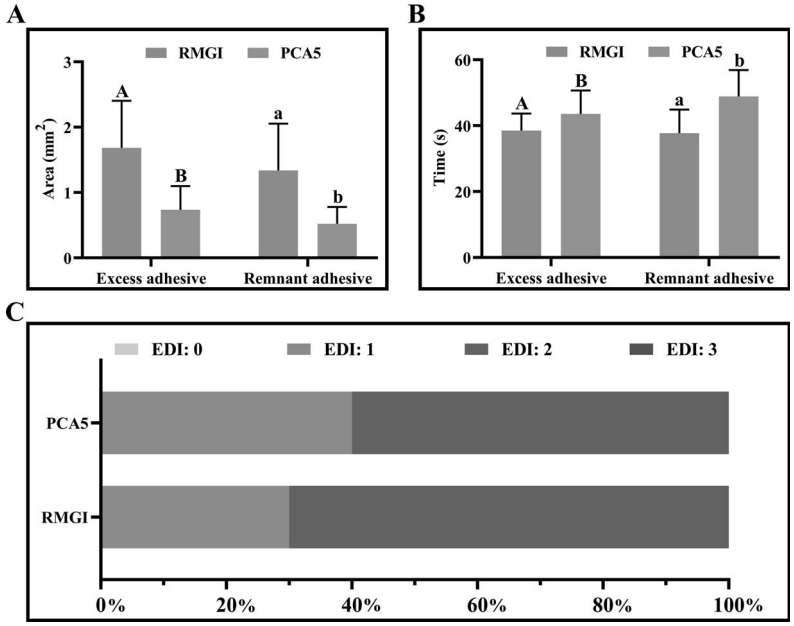

Figure 5. Adhesive removal effectiveness (A) and removal time (B); EDI of RMGI and PCA5 (C). Bars with the same letter are not significantly different ($P > .05$).

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from enamel surfaces without causing additional damage.[25] However, it has been shown that the tungsten-carbide bur can still leave a rough enamel surface.[6] In this study, the enamel surfaces of both RMGI and PCA5 groups showed slight damage after removing the remnant adhesive.

There were limitations of this study. First, the stability of the photochromic performance needs to be investigated over a longer time period, in a more realistic environment, due to the long duration of orthodontic treatment. The specific structural changes of the photochromic material within PCA5 under the blending process require further investigation. Finally, clinical studies are needed to confirm the effectiveness of the PCA5.

CONCLUSIONS

- PCA5 had reliable SBS and biocompatibility.
- PCA5 had excellent photochromic properties and could be removed more thoroughly than the RMGI, thus providing a new strategy for bracket adhesive removal.

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