

## Evaluation of Colour Stability of Selected Adhesive Systems with Different Staining Solutions

Hazlina Abdul Ghani<sup>1\*</sup>, Nur Alaa' Nasuha Abd Aziz<sup>1</sup>, Nur Aisyah Roslan<sup>1</sup>, Amar M. Thiyab<sup>2</sup>,  
Mohammed Gh. Abd Ali Al-Naser<sup>1</sup>

1. Centre for Restorative Dentistry Studies, Faculty of Dentistry, Universiti Teknologi MARA, Sungai Buloh Campus, Jalan Hospital, 47000 Sungai Buloh, Selangor, Malaysia.
2. Pembroke Dental Center, Ireland.

### Abstract

The purpose of this study is to evaluate the color stability of selected adhesive systems after immersion in different staining solutions, namely coke, instant coffee, and distilled water. The three adhesive systems used in this study were Maxcem Elite Chroma, Multilink N, and Rely X U200. A total of 102 veneers were prepared. For the experiment, two veneers were bonded using one of the adhesive systems, which produced 51 bonded specimens. The baseline color values ( $L^*$ ,  $a^*$ ,  $b^*$ ) for each specimen were measured with a spectrophotometer after immersion in distilled water for 24 hours. All 51 specimens were divided into three groups of adhesive systems. Each group was immersed in coke, instant coffee, or distilled water. The measurement was recorded every three days for 30 days. The recorded color difference  $\Delta E$  values were analyzed using IBM SPSS (IBM, Armonk, New York). The color difference  $\Delta E$  values for all adhesive systems were significantly affected after immersion in staining solutions ( $p < 0.001$ ). All adhesive systems showed the highest changes after immersion in instant coffee ( $p < 0.001$ ). However, the adhesive systems exhibited no significant changes after immersion in coke ( $p = 0.441$ ). All adhesive systems exhibited changes in color after immersion in the staining solutions. The use of instant coffee showed the highest staining for all adhesive systems. Maxcem Elite Chroma showed the highest staining among the adhesive systems used in this study after immersion in coke.

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### Introduction

Veneers have become a valuable treatment option for patients who seek better aesthetics, as veneers represent a more conservative approach and allow superior translucency.<sup>1</sup> Thus, it is very crucial for veneers to have long-term color stability, which is determined by their underlying adhesive cement. Dental cements are paramount for veneers as they occupy the gap between the restorative material and tooth structure and prevent the dislodgment of restoration during mastication or speech. In general, dental cements can be

divided into 2 categories, namely provisional dental cement and definitive dental cement.

Definitive cement, which is basically permanent cementation, is further divided into zinc phosphate cement, zinc polycarboxylate cement, glass ionomer cement, resin-modified glass-ionomer cement, and adhesive resin cement.<sup>2</sup> Maxcem Elite Chroma, Multilink N, and RelyX U200 belong to the adhesive resin-based systems. Table 1 summarizes the characteristics and chemical compositions of the aforementioned resin-based cements.

However, due to the high degree of translucency and changes in color over time, the discoloration of the materials in the resin cement used for cementation may become more visible, which affects the final aesthetic appearance of the restoration.<sup>3</sup> The causes of the discoloration of restorative materials, including the dental adhesive systems, are divided into extrinsic and intrinsic factors.<sup>4-8</sup> Extrinsic factors include the accumulation of plaque and surface stains by

#### \*Corresponding author:

Dr. Hazlina Abdul Ghani,  
Centre for Restorative Dentistry Studies, Faculty of Dentistry,  
Universiti Teknologi MARA, Sungai Buloh Campus, Jalan  
Hospital, 47000 Sungai Buloh, Selangor, Malaysia.  
E-mail: hazlina796@uitm.edu.my

staining agents.<sup>4</sup> Slight exposure to the staining agents can cause discoloration on the surface or subsurface of the resin restoration. Meanwhile, the intrinsic factors include the physio-chemical reactions within the material itself, which causes changes in color.<sup>4</sup>

commercially available dental adhesive systems after exposure to extrinsic factors, which specifically involved coke, instant coffee, and distilled water.

## Materials and methods

### Pilot study

For the pilot study, 4 pairs of Ceramage veneers (Shofu) and 1 dental adhesive system were used (Rely X U200, 3MESPE). The veneers used were standardized in terms of the dimension (10 mm x 8 mm x 1 mm) and manipulated according to the manufacturers' instructions.

### Sample size

Four pairs of cemented composite resins (n=4) were used and each was immersed in 4 different staining solutions, which included distilled water, coke, pepsi and instant coffee. The cemented sample that was immersed in distilled water acted as a control.

### Mold construction

Non-precious alloy Dura-Bond metal mold with the dimension of 10 mm x 8 mm x 1 mm was prepared with the help of a laboratory technician. Considering the difficulty of removing veneers from the first mold, another mold was prepared (Figure 1), and was successfully made after its sharp edges were trimmed into rounded edges, which enabled the ease of sample removal.

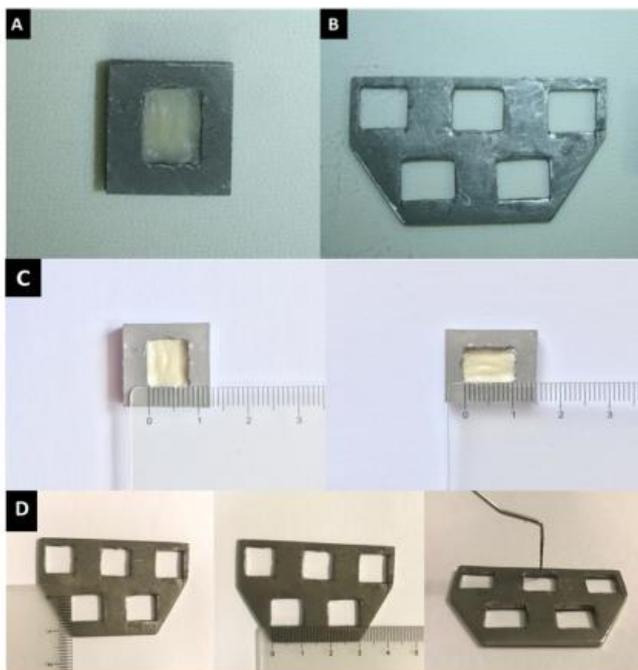
### Polymerization distance

Moore et al. (2008) proposed the distance of 1 mm between the light curing tip and composite resin as the lowest distance to be achieved in a clinical setting.<sup>9</sup> The thickness of the modelling wax (1mm) was used as a guidance for the standardization of the distance of light curing tip to the samples (Figure 2). An increase in the distance between the light curing tip and composite resin would significantly diminish the light intensity.<sup>10</sup> The distance of 1 mm between the light curing tip and composite resin has been recognised as the most commonly used and recommended for clinical setting,<sup>10-12</sup> which was also considered for this study.

Product	Company	Type	Mode	Curing	Composition
Maxcem Elite Chroma	Kerr	Self-etch / Self-Adhesive	paste dual syringe; auto mix	Dual	GPDM (glycerol dimethacrylate dihydrogen phosphate), comonomers (mono-, di-, and tri-functional methacrylate monomers), stabilizer, barium glass fillers, fluoroaluminosilicate glass filler, fumed silica (filler load 67% weight, particle size 3.6 mm)
Multilink N	Ivoclar/Vivadent	Primer A + B	Paste/paste dual syringe; auto mix	Self	Dimethacrylate and HEMA, barium glass filler, silicon dioxide filler, ytterbium trifluoride, catalysts and stabilizers and pigments
Rely X U200	3M ESPE	Self-Adhesive	Paste; clicker/auto mix	Dual	Base paste: Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives Catalyst paste: methacrylate monomers, alkaline fillers, silanated fillers, pigments, rheological additives

**Table 1.** The resin-based cement used in the present study.

The degree of changes in color varies according to the oral hygiene status, nutritional habits, cigarette smoking, consumed beverages, and manufacturers' formulations. Furthermore, the degree of discoloration may also directly depends on the regularity of carbonated beverage consumption and the quantity of intake.<sup>7</sup> Therefore, patients can overcome these problems by avoiding or reducing their consumption of preservatives-laden foods or beverages. Patients are also advised to avoid smoking. Adding to that, dental practitioners should perform sufficient light curing, as unconverted camphorquinone would cause yellowish discoloration.<sup>5</sup> The polishing of the composite resin should also be properly performed, as rough surfaces facilitate retention of staining agents which could lead to the discoloration of the composite resin. Besides that, manufacturers should design products that take longer time to stain given the public concern about their aesthetics. In view of the above, this study aimed to evaluate the color stability of 3



**Figure 1.** (A) The first metal mold could not be used due to difficulty upon removal of samples (B) Metal mold used in this study (C)(D) The measurement of the molds (10 mm x 8 mm x 1 mm).

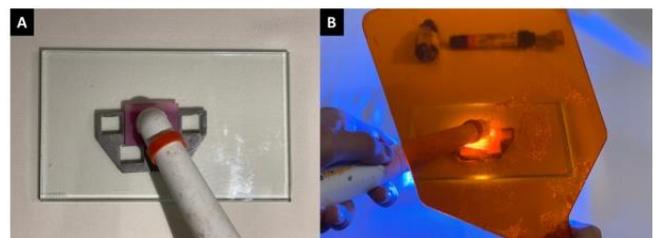


**Figure 2.** The modelling wax with 1 mm thickness was used to standardize the distant between the light cure and sample.

#### Preparation of samples

For the preparation of veneer samples, composite resin shades A2B were dispensed, manipulated, and polymerized in the customized metal mold, according to the manufacturers' instructions. All samples were light-cured with FLASHlite Magna LED Dental Curing Light (DenMat, Lompoc, CA, USA; light intensity of 1500 mW/cm<sup>2</sup>) for 20 seconds, at 1mm distance as recommended by previous studies (Figure 3).

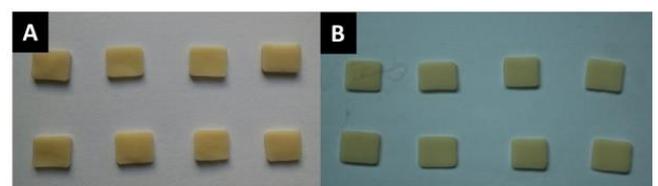
<sup>9-12</sup>



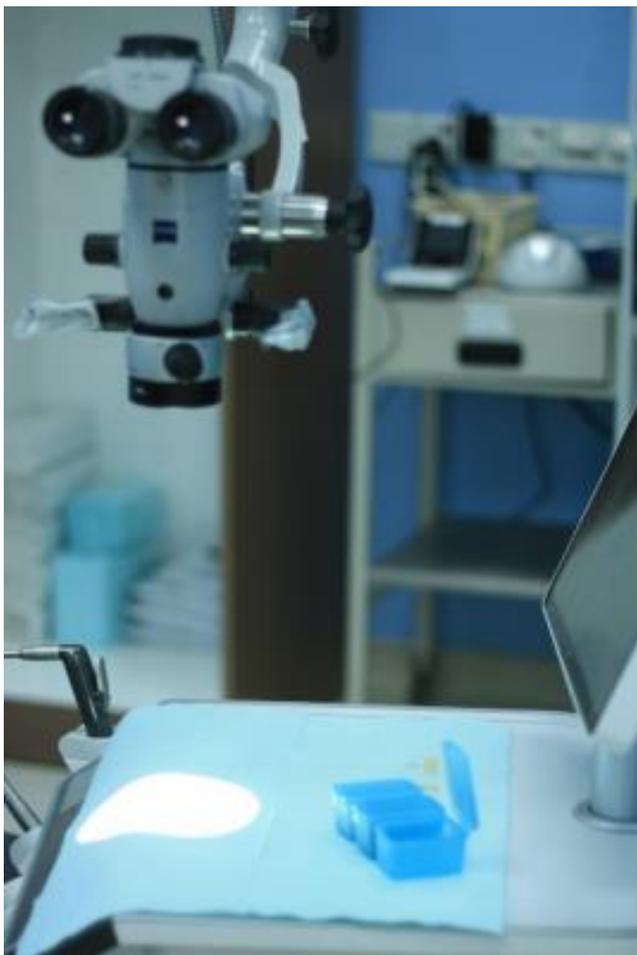
**Figure 3.** (A) The distant between curing light and the sample was 1 mm (B) The sample was polymerized by using (light cure) FLASHlite Magna LED Dental Curing Light (DenMat, Lompoc, CA, USA; average output of 1500 mW/cm<sup>2</sup>) for 20 seconds with the light tip approximately 1 mm away from the specimen.

A wax carver was used to remove the polymerized samples from the mold. All samples were polished using a polishing kit (Ceramage, Shofu) for 5 seconds for surface standardization. Figure 4 shows the samples before and after the polishing procedure. Each was analyzed under the microscope for any deformity (Figure 5). Figure 6 shows the image of samples under the microscope. The dental adhesive system from Rely X U200, 3M ESPE was used to cement the prepared samples in pairs to resemble the veneer-cement-tooth complex. The adhesive systems were dispensed, manipulated, and polymerized according to the manufacturers' instructions. Any material excess was removed and cleaned.

Following cementation, all cemented samples were stored in distilled water at room temperature of 27°C for 24 hours. Subsequently, the determination of baseline values involved measuring each value of L\*, a\* and b\* for each sample using Konica Minolta spectrophotometer CM-5 (Figure 7).



**Figure 4.** (A) The samples before being polished (B) The samples after being polished.



**Figure 5.** The samples were analyzed under the microscope.



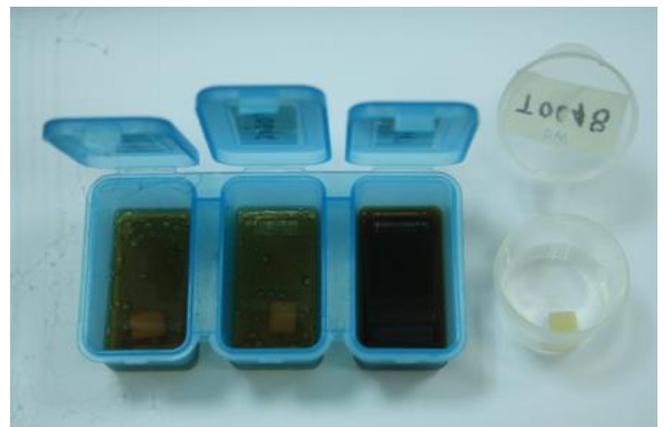
**Figure 6.** The sample's image under the microscope.

L\* indicates lightness—the color of the sample is lighter if the difference in  $\Delta L$  is positive; otherwise, the color of the specimen is darker if the difference in  $\Delta L$  is negative. Meanwhile, the value of a\* is related to the red/green coordinate, where positive difference in  $\Delta a$  means redder

whereas negative difference in  $\Delta a$  means greener. On the other hand, the value of b\* is related to the yellow/blue coordinate, where positive difference in  $\Delta b$  means yellower whereas negative difference in  $\Delta b$  means bluer. Deltas for L\* ( $\Delta L^*$ ), a\* ( $\Delta a^*$ ), and b\* ( $\Delta b^*$ ) may be positive (+) or negative (-). However, the total difference, Delta E ( $\Delta E^*$ ), is always positive. The L\*a\*b\* color space data from the spectrophotometer was scientifically employed to express color in numerical terms and to measure the differences in color between samples.



**Figure 7.** Konica Minolta spectrophotometer CM-5.



**Figure 8.** The samples were immersed in different containers consist of distilled water, Coca Cola, Pepsi and instant coffee.

Each cemented sample were then transferred to 4 different containers that contained distilled water, coke, pepsi and instant coffee (Figure 8). The samples were measured every 3 days for 30 days. The recorded values for the samples in distilled water acted as the control. Based on the final findings of this pilot

study, there is a significant change of  $\Delta E$  value (color difference) of the samples after the immersion in coke, pepsi and instant coffee. We decided to eliminate pepsi as a staining solution in the major study since pepsi shows no difference with coke in their  $\Delta E$  value from the pilot study.

### Major study

#### Sample size

51 pairs of Ceramage veneers (Shofu) were prepared (n=51). The sample size for this study was prepared according to the mathematical equation obtained from previous study.<sup>13</sup> Three different brands of adhesive system were used (Maxcem Elite Chroma, Kerr; Multilink N, Ivoclar/Vivadent; and Rely X U200, 3M ESPE).

#### Mold construction

In the major study, the same trimmed mold from the pilot study was used with 10 mm x 8 mm x 1 mm dimensions (Figure 1).

#### Polymerization distance

The thickness of the modelling wax (1mm) was used as a guidance for the standardization of the distance of light curing tip to the samples as shown in Figure 2.

#### Preparation of samples

The sample preparation items were set up (Figure 9). The Ceramage veneers were prepared using the custom-made metal mold. The composite resins were dispensed, manipulated, and polymerized according to the manufacturers' instructions (Figure 10). Polymerization was carried out using FLASHlite Magna LED Dental Curing Light (DenMat, Lompoc, CA, USA; average output of 1500 mW/cm<sup>2</sup>) for 20 seconds with the light curing tip of approximately 1 mm away from the composite resin (Figure 3).

Any irregularities of the Ceramage veneers were removed and trimmed using a straight handpiece tungsten carbide bur (Figure 11). All samples were polished with a polishing kit (Ceramage, Shofu) for 5 seconds for surface standardization (Figure 12). Figure 13 shows the samples before and after the polishing procedure. Following that, each sample was analyzed under the microscope for any deformity (Figure 14). In

this study, 51 pairs of veneers were divided into 3 groups of adhesive system and cemented with the selected adhesive system accordingly. They were placed in 3 different containers (17 cemented veneers in each container) containing distilled water and stored at room temperature of 27°C for 24 hours. Subsequently, the baseline values of L\*, a\* and b\* for all samples were measured using Konica Minolta spectrophotometer CM-5 (Figure 7).



Figure 9. The setup for sample preparation.

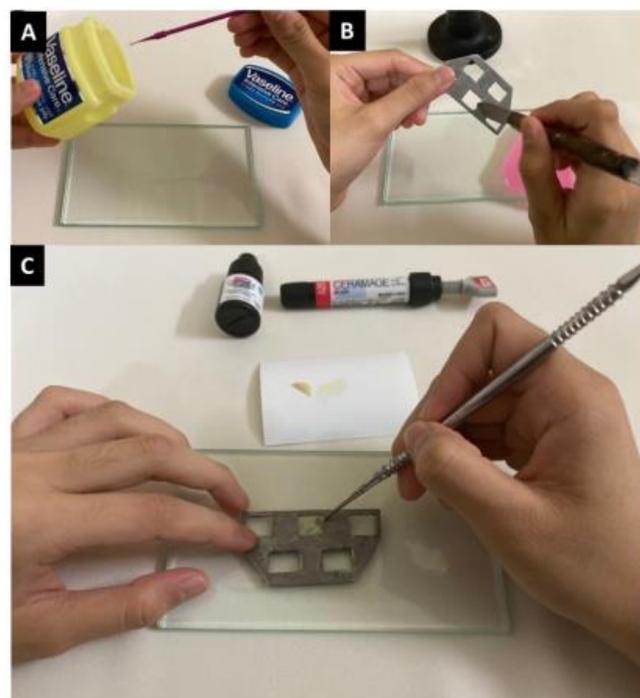


Figure 10. (A) The petroleum jelly (Vaseline) was applied on glass slab for easy removal of the sample (B) The wax was applied on the edge of the mold (C) The resin composite was dispensed in the mold.



**Figure 11.** The sample was trimmed by using straight handpiece tungsten carbide bur.



**Figure 12.** The polishing kit (Ceramage, Shofu) was used in this study.



**Figure 13.** (A) The sample before polished (B) The sample after polished.



**Figure 14.** The sample's image under the microscope.

*Preparation of staining solutions*

For the preparation of staining solution from instant coffee, as per manufacturers' recommendations, approximately 1.3 g of instant coffee was added into 150 ml of boiling water for 10 minutes and then filtered through filter paper.<sup>14</sup> Staining solutions from carbonated drink, coke was prepared. Following that, the cemented veneers of each container were further divided into 3 subgroups based on selected staining solutions with the ratio of 1:8:8 (distilled water: coke: instant coffee) (Figure 15).

Adhesive systems	Maxcem Elite Chroma, Kerr	Multilink N, Ivoclar/Vivadent	Rely X U200, 3M ESPE
Staining solutions			
Distilled water			
Coke			
Instant coffee			

**Figure 15.** Distribution of cemented veneer samples in 3 different adhesive systems and in 3 different staining solutions.

*Immersion of samples in staining solutions*

The samples were immersed in 3 different staining solutions and the color stability of dental adhesive systems were measured every 3 days for 30 days.

Subgroup 1: Distilled water (control)

Subgroup 2: Coke

Subgroup 3: Instant coffee



**Figure 16.** The mean values of L\*, a\*, and b\* of the sample were calculated.

*Color change measurement*

Table 2 presents the Konica Minolta spectrophotometer CM-5 calibration for the color change measurement. All samples of 3 subgroups were placed on the measuring head of spectrophotometer (one at a time) and covered with a black cover. The 3 variables L\*a\*b\* measurements were performed 3 times. The mean values were automatically calculated and recorded by the spectrophotometer (Figure 16). The same procedure was repeated every 3 days for 30 days. The color difference ΔE was calculated from the mean values of ΔL\*, Δa\*, Δb\* for the 3 subgroups using the following formula:

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

where ΔE\* represents the color difference; ΔL\* represents the change in lightness; Δa\* represents the red-green coordinate; Δb\* represents the yellow-blue coordinate.

Measurement type selection	Reflectance
Measurement area selection	5 mm, with specific target mask size
Specular component selection	SCE excluded
Zero and White calibration	Set the zero calibration boxes and process, then remove box and process white calibration with automatic selection

**Table 2.** Calibration setting steps for Konica Minolta spectrophotometer CM-5.

*Data collection and statistical analysis*

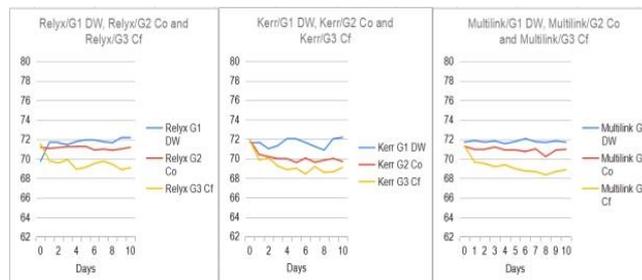
The color difference ΔE values were analyzed using IBM SPSS (IBM, Armonk, New York). The mean and standard deviation values that were estimated for the samples from each subgroup were statistically analyzed. The mean values of different groups were compared using one-way ANOVA and the statistical significance of the differences between the groups were determined in t-test. As for the present study, p < 0.05 was considered as the level of significance.

**Results**

Figure 17 shows the changes of color difference ΔE values for all dental adhesive systems in 3 subgroups of staining solutions throughout the study. A similar pattern in color changes was observed for all adhesive systems groups' veneers, with instant coffee causing the most color changes for all adhesive groups.

Apart from that, the results of the one-way ANOVA test in Table 3 showed significant differences between the average mean values of color difference ΔE for the 3 solutions (p < 0.001).

Meanwhile, the results of t-test in Table 4 revealed significant differences between the average mean values of color difference ΔE within the solution groups.



**Figure 17:** DW, Distilled water; Co, Coke; Cf, Coffee, Color change values, ΔE of each adhesive system in different solutions.

Test Statistic F	Critical Value @5% C	Std. Error	Significant F > C	P-value
103.365	3.091	0.3	Yes	< 0.001

**Table 3.** Results of one-way ANOVA test for the changes of color difference ΔE values for the solutions.

		Test Statistic t	Critical Value 5% C	Std. Error	Significant F > C	P-value
DW	Co	7.32	1.998	0.3	Yes	<0.001
	CF	13.745	1.998	0.3	Yes	<0.001
Co	DW	7.320	1.998	0.3	Yes	<0.001
	CF	7.460	1.998	0.3	Yes	<0.001
CF	DW	13.745	1.998	0.3	Yes	<0.001
	Co	7.460	1.998	0.3	Yes	<0.001

**Table 4.** Results of t-test for the changes of color difference ΔE values within the solution groups.

As for the dental adhesive systems, the results of the one-way ANOVA test in Table 5 showed no significant differences between the average mean values of color difference ΔE (p = 0.316). The results of t-test in Table 6 also revealed no significant differences between the average mean values of color difference ΔE within the adhesive system groups.

Test Statistic F	Critical Value @5% C	Std. Error	Significant F > C	P-value
1.167	3.091	0.3	No	0.316

**Table 5.** Results of one-way ANOVA test for the changes of color difference ΔE values of the adhesive systems.

		Test Statistic t	Critical Value @5% C	Std. Error	Significant F > C	P-value
Kerr	Multilink	0.968	1.998	0.3	No	0.337
	Rely-X	1.53	1.998	0.3	No	0.131
Multilink	Kerr	0.968	1.998	0.3	No	0.337
	Rely-X	0.502	1.998	0.3	No	0.617
Rely-X	Kerr	1.530	1.998	0.3	No	0.131
	Multilink	0.502	1.998	0.3	No	0.617

**Table 6.** Results of t-test for the changes of color difference  $\Delta E$  values within the adhesive system groups.

## Discussion

Due to the increasing demand for aesthetic dentistry, it is important to select reliable restorative materials for maximum satisfaction. However, according to Jain et al. (2003), all restorative materials would be discolored over time, which was a major disadvantage.<sup>15</sup> The factors that potentially influence the color stability of these materials include resin matrix, dimension of filler particles, depth of polymerization, and staining agents.<sup>16, 17</sup>

For the past few years, numerous studies on the color stability of dental materials after immersion in staining solutions were conducted. Most of these past studies were performed on composite resins or veneers. There has been lack of studies on adhesive systems. Therefore, this study evaluated the color stability of dental adhesive systems after immersion in different staining solutions. In order to reproduce a clinical scenario, the veneer-cement-tooth complex were resembled by using the adhesive systems to cement veneer to veneer.

In this study, the adhesive systems were immersed in 3 staining solutions, which were distilled water, coke and instant coffee. Distilled water acted as control solution. For beverages, coke and instant coffee are identified as staining solutions.<sup>18</sup> The changes in color for the materials after exposure to these 2 staining solutions have been reported in various studies.<sup>5, 18-20</sup> For instance, a study in 2016 on regular beverages among Malaysians revealed coffee as the most consumed beverage, with 60.83% of the total respondents indicated that they drink coffee daily.<sup>21</sup>

There are 2 ways to evaluate the changes in color, which are visual or instrumental techniques.<sup>16</sup> A spectrophotometer was used in this study, instead of visual examination, to produce precise, reproducible, and statistically

practical results.<sup>22</sup> The CIE L\*a\*b\* color system, which characterises color based on human perception and clinical significance,<sup>16, 23</sup> was used in this study. In particular, the values of b\*, which indicates the yellow/blue coordinate, increased for both coke and instant coffee throughout the experiment. This explains the yellowish stain produced. The values of \*L, which indicates the change in lightness, recorded a slight reduction for both staining solutions. These results coincided with the results of b\* values. The values of a\*, which indicates the red/green coordinate, did not demonstrate substantial difference throughout the experiment, as there was no such color present in the study.

Besides that, the color stability of 3 adhesive systems were also tested in this study, namely (1) Maxcem Elite Chroma, Kerr, (2) Multilink N, Ivoclar/Vivadent, and (3) Rely X U200, 3M ESPE. The comparison of all 3 adhesive systems revealed no significant differences. There were differences between the initial color difference  $\Delta E$  value and final color difference  $\Delta E$  value but the values were rather similar, which explain the insignificant outcome.

In this study, the immersion period for all samples was a total of 30 days, as this time interval was commonly used in the past colorimetric studies.<sup>6</sup> The selected adhesive systems showed clinically perceptible color changes after immersion in coke and instant coffee, which were denoted by the significant differences of  $\Delta E$ . The samples were mostly darker and yellowish. Instant coffee caused the most discoloration. This finding was found to be consistent with a study performed by Bagheri et al. (2015), where coffee was also found to cause significant color changes, as compared to coke. The discoloration is attributed to the adsorption and absorption of the colorant particles of coffee onto the surface materials.<sup>24</sup> According to Um and Ruyter (1991), coke exhibits small color changes, as compared to coffee and tea, which is possibly due to the low yellow colorant content although it has the lowest pH and potentially damages the surface integrity of the composite resin materials.<sup>25</sup> This can be linked to the results of this study, where coke caused significant color changes to all adhesive system groups but the changes appeared less significant, as compared to instant coffee.

## Conclusions

In conclusion, the immersion of all selected adhesive systems in both coke and instant coffee caused changes in color. Instant coffee exhibited more prominent staining, as compared to coke. This is believed to be due to the higher yellow colorant content in coffee in comparison to coke.<sup>25</sup> This study also demonstrated the color changes of all 3 adhesive systems when these adhesive systems were subjected to different staining solutions. Patients should be aware of the consequences of consuming coke and coffee, as these beverages, particularly coffee, can potentially cause prominent staining to the restorative materials in their teeth. It is also important for clinical practitioners to consider the staining susceptibility of the adhesive systems used for the veneers.

This *in vitro* study presented a limited simulation of the intraoral condition where the discoloration of the materials can only be evaluated in different single solution. Therefore, it is recommended for future research to further explore these changes *in vivo* to better reflect a clinical setting.

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## Declaration of Interest

The authors declare that there is no conflict of interest in this study.

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