



## Effect of 38% hydrogen peroxide agent on color change of composite resins stained with tea and coffee beverages (An in vitro study)

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### Abstract

**Background:** The esthetic application of bleaching materials has gained popularity, with consequences for teeth and restorative materials. The purpose of this in vitro study was to investigate the effect of in-office bleaching agent (38% hydrogen peroxide) on the color change of three composite resins after staining with tea and coffee beverages.

**Materials and Methods:** Sixty disc-shaped samples from three different composite resins (Tetric Ceram, Tetric Flow and Tetric Evo Ceram) were used in this study. The samples of each group were randomly divided into two subgroups according to type of beverages (tea and coffee) used for staining of samples. After staining of composite resins the total color change ( $\Delta E_1$ ) was calculated using spectrophotometer, then the samples immersed in WHITEsmile® office bleaching agent for 15 min x 3 times with a 7 days interval followed by  $\Delta E_2$  calculation. Data were subjected to the ANOVA, LSD and student t-test at 0.05 significance level.

**Results:** The results revealed that, there was clinically significant color change  $\Delta E > 3.3$  with a statistically highly significant differences ( $P=0.000$ ) for all the groups. Coffee causes more discoloration than tea. After bleaching, the samples became whiter in color and only appreciable by skillful operator but considered clinically acceptable  $\Delta E_1 - \Delta E_2 < 3.3$ .

**Conclusion:** Tea and coffee beverages causes discoloration in composite but this effect was depended on the type of beverages and composite resins. The most of color change occurred with Tetric Flow composite resin after immersion in coffee beverage. The bleaching agent (38% hydrogen peroxide) can remove the staining of composite resins and make its color clinically acceptable.

**Keywords:** Hydrogen peroxide, color change, composite resins, spectrophotometer.

### Introduction

Concerns about aesthetic lead the people to choose tooth-colored dental restoration especially the one that can match similarly the color of the tooth.

Composite resins are one of these that were widely used because of their excellent aesthetic properties, which can match the color of natural teeth.

However, a major disadvantage is their discoloration after prolonged exposure to the oral environment <sup>(1)</sup>. Discoloration of tooth-colored resin-based materials may be caused by intrinsic or extrinsic factors. <sup>(2)</sup>

The intrinsic factors involve the discoloration of the resin material itself, such as the alteration of the resin matrix and the interface of the matrix and the fillers. Extrinsic factors for discoloration include staining by adsorption or absorption of colorants as a result of contamination from exogenous sources. The degree of discoloration from exogenous sources varies according to the oral hygiene, the eating-drinking and smoking habits of the patients. <sup>(3)</sup>

The test commonly applied for determining the color stability of resinous materials used in dentistry as specified by ADA specification no. 12 and bureau of Indian Standards IS: 12181-1987 is based on visual comparison of the color of a specimen exposed to the radiation of a Xenon lamp or a radiation source of equivalent energy and then comparing the color of the specimen with the color of an unexposed sample. <sup>(4)</sup> The most common types of equipment for measurement of color were spectrophotometers and tristimulus colorimeters. <sup>(3)</sup>

The use of bleaching techniques is a relatively new approach to lighten stained teeth and composite resin restorations. The efficacy of bleaching agents depends on the type of stain, its etiology and the exposure time of the bleaching agent used. <sup>(5)</sup>

Materials with different chemical compositions such as glass ionomers, composites, compomers and ormocers may respond differently to the same bleaching agent <sup>(6)</sup>

So this study was conducted to assess the effect of commonly used types of beverages (coffee and tea) on

discoloration of composite resin (microhybrid, flowable and nanohybrid), and measured the efficacy of in office bleaching agent (38% hydrogen peroxide) to remove the discoloration caused by these beverages.

## Materials and Methods

### Samples Preparation

Sixty specimens (shade A2) were constructed from three composite materials (20 from each). The three resin-based composite materials used in this study are shown in (Table 1). Plastic disc shape molds were used for the composite samples construction (10 mm in diameter and 2 mm thickness). The resin materials were inserted into the mold. The mold was placed on flat microscopic glass slide on top of a celluloid strip (Odus, Produits Dentaires SA CH-1800 Vevey/Switzerland) and covered with other strip and glass slide. Every sample was light cured using a halogen curing light (YDL, Hangzhou Yinya New Materials CO., China) with a light intensity 400-450 mW/cm<sup>2</sup>, for 20 sec. of exposure time to top and bottom surfaces, respectively. The output of the curing units was checked with a radiometer. <sup>(2)</sup>

The samples were finished and polished using soflex composite finishing and polishing system (EDENTA, Polstfach 183, FL-9490 Vaduz/Liechtenstein). Water resistant marker was used for numbering the specimens on one side to distinguish the specimen before and after immersion <sup>(3)</sup>. One surface of sample was marked; this surface will be in front of spectrophotometer's light source during measurement.

Then all prepared samples were stored in distilled water at 37°C for 24 hours using incubator, for rehydration and completion of the polymerization

to mimic the first day of service in the oral environment, then subjected to

baseline color values measurement ( $L_0$ ,  $a_0$ ,  $b_0$ ).<sup>(2)</sup>

### Sample grouping

Sixty samples (shade A2) were prepared from the three composite materials (20 samples from each)

**Group TC:** 20 samples were made from **Tetric Ceram**.

**Group TF:** 20 samples were made from **Tetric Flow**.

**Group TE:** 20 samples were made from **Tetric Evo ceram**.

Then the samples of each group were further randomly subdivided into two equal subgroups (n=10)

**Subgroup Te :** Immersion of composite sample in tea

- TCTe (n=10)
- TFTe (n=10)
- TETe (n=10)

**Subgroup Co:** Immersion of composite samples in coffee

- TCCo (n=10)
- TFCo (n=10)
- TECo (n=10)

Then **Te** and **Co** subgroups subjected to bleaching agent and subdivided according to composite materials used:

**BTe:** bleaching of **Te** subgroup

- TCBTe (n=10)
- TFBTe (n=10)
- TEBTe (n=10)

**BCo:** bleaching of **Co** subgroup

- TCBCo (n=10)
- TFBCo (n=10)
- TEBCo (n=10)

### Preparation of Staining Solution (beverages).

The solutions used for staining (tea & coffee) were presented in Table 2. Tea solution was prepared by adding one pre-measured tea bag into 200 mL of boiling distilled water. Coffee solution was prepared by adding 5 grams of coffee powder to 200 mL of boiling distilled water. Both solutions were stirred every 30 minutes for 10 seconds until they cooled down to 37°C then filtered through a filter paper.<sup>(3,7)</sup>

### Immersion of samples in staining solutions

After baseline color measurements were made, the prepared solutions were poured into dishes and the samples immersed in them for 15 days. However, according to the

manufacturer of the coffee and tea, the average consumption time for one cup of coffee is 15 minutes, and among coffee drinkers the average consumption quantity is 3.2 cups per day. Therefore, a 24 hour storage time simulates about one month of coffee consumption, so 15 days immersion times equal to 15 months of coffee and tea consumption<sup>(8)</sup>. The solutions were kept at a constant temperature of  $50 \pm 1$  °C in an incubator. The staining solutions were changed every 7<sup>th</sup> day and to reduce the precipitation of particles in solutions, solutions were stirred once a day.<sup>(7,9)</sup>

After completion of immersion period (15 days) the samples were cleaned using an electric toothbrush (Oral-B, Colgate-Palmolive, NY, USA) with toothpaste (Colgate,

Thailand) for 10 seconds on each side of the specimens. This was followed with gentle rinsing with distilled water, blotted dry with tissue papers then color values ( $L_1, a_1, b_1$ ) and total color change were measured ( $\Delta E_1$ ).<sup>(2)</sup>

### Measurement of color and calculation of the color difference

Color of the specimens was measured using spectrophotometer (FGI-SST-1103, China) against a white background. Values were recorded using the Commission International de l'Éclairage (CIE)  $L^*a^*b^*$  color system and relative to CIE standard illuminant  $D_{65}$ . Color of samples before and after immersion in the different types of the staining solutions was measured according to the following formula<sup>(2)</sup>:

$$\Delta E_1^* = [(L_1 - L_0)^2 + (a_1 - a_0)^2 + (b_1 - b_0)^2]^{1/2}$$

$$\Delta E^* = \text{total color change } \Delta E^* = (\Delta L^2 + \Delta a^2 + \Delta b^2)^{1/2}$$

$L^*$  characterizes the lightness of the color and can be ranged between 0 (dark) and 100 (light) luminance reflectance

$a^*$  red-green color coordinate. Positive  $a^*$  values represent red hues and negative  $a^*$  values represent green hues.

$b^*$  yellow-blue color coordinate. Positive  $b^*$  values represent yellow hues and negative  $b^*$  values represent blue hues

$L_0, a_0, b_0$  (baseline measurements before immersion in staining solutions)

$L_1, a_1, b_1$  (measurements after immersion of the same sample in staining solutions)

Three different intervals were used for distinguishing color differences because the ability of the human eye to appreciate differences in color differs from individual to individual (as it is a combination of eye characteristics and skill of the operator). No Perceptible color change by the human eye occurred if  $\Delta E < 1$ , but If  $3.3 > \Delta E > 1$ ,

this color difference is appreciable by skillful operator but considered clinically acceptable. Whilst values of  $\Delta E > 3.3$  are appreciable by non skilled persons and considered clinically unacceptable and statistically significant.<sup>(10,11)</sup>

### Method of Bleaching

The tested samples were subjected to in office bleaching process by applying WHITEsmile® gel (38% hydrogen peroxide) using cotton applicator on the marked surface of the sample. The time of application is 15 min. After bleaching, the specimens were rinsed under running water for 1 minute to remove the bleaching agent, blotted dry and stored in distilled water at 37°C till the next application. This step was repeated for 3 times for each sample as recommended by the manufacturer; so the total exposure time of bleaching agent for each sample was 45 min with 7 days interval.<sup>(12)</sup> After bleaching the samples subjected again to color testing measurement  $\Delta E_2^* = [(L_2 - L_1)^2 + (a_2 - a_1)^2 + (b_2 - b_1)^2]^{1/2}$ .

### Statistical Analysis

All statistical analyses were carried out using SPSS statistical software (version 19.0, SPSS, Chicago, IL, USA). After data collection, mean values and standard deviations were calculated for all groups and subgroups. The total color change  $\Delta E_1$  (after immersion in tea and coffee) and  $\Delta E_2$  (color change after bleaching) of each tested materials (Tetric ceram (TC), Tetric flow (TF) and Tetric EvoCeram (TE)) were calculated according to equation  $\Delta E = (\Delta L^2 + \Delta a^2 + \Delta b^2)^{1/2}$ . One way analysis of variance (ANOVA) test was performed among the experimental groups to determine where there any statically significant differences under various conditions.

When a significant difference was found, Least significant difference (**LSD**) test was done to find where the significance occur. Also student t-test were used to compare between  $\Delta E_1$  and  $\Delta E_2$ . The mean difference is significant at the 0.05 level.

## Results

The mean and standard deviation values of  $\Delta E_1$  are summarized in Table 3 and graphically

represented in Figure 1. The result found that both beverages causes' clinically significant discoloration ( $\Delta E > 3.3$ ) in three types of composite resin tested with a statistically high significant differences among the groups revealed by one way ANOVA test ( $P = 0.000$ ).

After bleaching, the color change  $\Delta E_2$  for the three tested composite resins was calculated. The composite resins showed clinically significant color change ( $\Delta E > 3.3$ ), with highly significant differences between the groups revealed by one way ANOVA test ( $P = 0.000$ ) as shown in Table 4 and graphically represented in Figure 1. More discoloration of composite resins occurred with coffee.

Further investigation using **LSD** test showed that there was a statistically high significant differences ( $P < 0.05$ ) between all the subgroups before and after bleaching (table 6, 7).

After bleaching, the color of all the three materials almost returned to the baseline, and was below clinically perceptible limit ( $\Delta E_1, \Delta E_2 < 3.3$ ). **t-test** was done to compare between  $\Delta E_1$  and  $\Delta E_2$  for tea and coffee subgroups and show a highly significant difference s shown in Table 5.

## Discussion

Color plays an important role in obtaining optimum aesthetics. The

major disadvantage of resin composites is their color instability which may be a major cause for replacement of restorations. Discoloration can be evaluated visually and by using specified instruments such as spectrophotometer, which can potentially eliminate subjective errors in color assessment<sup>(13)</sup>.

**Staining of composite resins with tea and coffee.** The results found that all types of composite resins tested discolored after subjected to staining agent (tea and coffee) with highly significant differences among the groups ( $P = 0.000$ ). The most discoloration occurred with **Tetric Flow** composite, followed by **Tetric Ceram** and less color change occurs with **Tetric Evo Ceram** (Table 3). The resin matrix used in the materials play an important role in staining susceptibility of composite resins. UDMA seems to be more stain-resistant than bis-GMA because of its low water absorption and solubility characteristics. It was reported that the water uptake in bis-GMA based resins increased from 3 to 6 % while that in TEGDMA increased only from 0 to 1%. The resin systems of **Tetric Ceram** and **Tetric Flow** consist of three major components: bis-GMA, UDMA and TEGDMA. TEGDMA consider as hydrophilic resin, that responsible for the high water absorption and discoloration rates in tested composites<sup>(14)</sup>. **Tetric Evo Ceram** containing dimethacrylates resins that have low water absorption rates than TEGDMA containing composite resin, so consider as staining resistance composite resin. According to **Bagheri et al.**,<sup>(14)</sup> if the composite is able to absorb water, then it is also capable of absorbing other fluids, resulting in its discoloration and in the reduction of its mechanical properties due to polymer matrix degradation<sup>(10)</sup>. The glass filler

particles will not absorb water into the bulk of the material, but can adsorb water onto the surface. Extra water sorption may decrease the life of resin composites by expanding and plasticizing the resin component, hydrolyzing the silane and causing microcrack formation<sup>(15)</sup> Therefore, the microcracks or the interfacial gaps at the interface between filler and matrix allow stain penetration and discoloration<sup>(14)</sup> This theory could be confirmed in this study, as can be observed in the alterations of L, a and b values and causes  $\Delta E > 3.3$ . In the present study, difference in filler size between composites might have allowed the nanohybrid materials (**Tetric Evo Ceram**) to attain a lower surface roughness value than the microhybrid (**Tetric Ceram**) and flowable (**Tetric Flow**) composites. During finishing and polishing operations, filler particles might be plucked out leaving voids. It has been reported that in nanohybrids, smaller particles were shaved off and smaller voids were left on the surface as compared to the microhybrids<sup>(16)</sup>, and causes discoloration in composite resin. But the differences in filler content are small between the three types of composite resins used in this study, so higher color changes were showed with tetric flow composite resin that has lower filler loading than other composite resins. Bayne and Taylor, 1995 stated that increasing the filler contents of composite resins generally improve the physical, chemical and mechanical properties such as water absorption, color stability, hardness and wear resistance<sup>(17)</sup>

In **Table 3**, we can see that  $\Delta E$  of composite resins was higher with coffee than tea; this differences could be related to the differences in pH of beverages. The pH of coffee is about (5), while the pH of tea (5.6) the lowest

pH causes surface disintegration of composite resins and increase wear and surface roughness. Increase in surface roughness of composite resins causes staining from exogenous sources<sup>(17)</sup>. Also the type of colorants in beverages plays important roles in discoloration of composite resins. Both tea and coffee contained yellow colorants which had different polarities. Higher polarity components like those in tea were eluted first, while lower polarity components like those in coffee were eluted at a later time. It is widely accepted that the susceptibility to extrinsic staining as well as the degradation of composite can be determinate by its degree of fluid sorption<sup>(19)</sup>. Discoloration by tea due to adsorption of polar colorants onto the surface of resin composite materials could be removed by tooth brushing; where as discoloration by coffee was due to both absorption and adsorption of polar colorants onto the surface of materials. This adsorption and penetration of colorants into the organic phase of the materials were explained by the authors as probably due to compatibility of the polymer phase with the yellow colorants of coffee.<sup>(1)</sup> There are many studies confirmed this study, **Gupta et al.**<sup>(3)</sup> found that higher color change occurred in **Filtek Z 250 & Tetric ceram** after immersion in coffee followed by tea and less color change occurred with coca cola.

**Effect of bleaching agent on color change:** The efficacy of bleaching agents depends on the type of stain, its etiology and the exposure time of the bleaching agent used<sup>(4)</sup>. The statistical difference among the composite resins in bleaching action was high significant (**P=0.000**) as shown in Table 4; differences in total color change between different composite materials might be a result of different resin, filler content, initiation

components and different degrees of conversion of resin matrix<sup>(6)</sup>.

Both flowable and microhybrid composites contain **UDMA** and **TEGDMA** with lower filler content where as Nano hybrid contains **dimethacrylates** with higher filler content (**82-83 %wt.**) which may be less resistant to the bleaching agent. This finding is in agreement with the results of other studies which attributed the higher discoloration of certain composites to their lower filler concentration, and lower resistance to oxidation attributed to the higher resin content of the material<sup>(4, 6, 19)</sup>. While measurement of total color change ( $\Delta E_2$ ) after bleaching revealed, perceptible color change of all composite resins used ( $\Delta E_2 > 3.3$ ), this mean that bleaching agent was effective in reducing the staining in the groups to be within the clinically acceptable value ( $\Delta E_1 - \Delta E_2 < 3.3$ ) with highly significant differences between  $\Delta E_1$  &  $\Delta E_2$  revealed by **t-test** as shown in **Table 5**, and highly significant differences in color change values among the composite materials used revealed by **LSD** test (table 7).

Hydrogen peroxide is an aggressive oxidant capable of degrading the polymer matrix of resin-rich composite materials. It breaks down into oxygen and water as well as free radicals which result in oxidation of the pigments or amine compounds within the structure. In addition to its reactivity, hydrogen peroxide demonstrates an extensive ability for diffusion. Oxidation of the pigments may occur as a result of direct interaction with hydrogen peroxide on the resin surface. Peroxides might induce oxidative cleavage of polymer chains. Therefore, any unreacted double bonds are expected to be the most vulnerable parts of the polymers. Furthermore, free radicals induced by peroxides may impact the resin filler

interface and cause filler-matrix debonding. Microscopic cracks are formed, resulting in surface roughness and leading to diffusion of agent<sup>(19)</sup>.

The results of bleaching subgroups were agreed with many studies<sup>(6, 19)</sup> but the difference was, most of study used home bleaching agent for many days. **Hubbezoglu I. et al.**<sup>(19)</sup>, found that bleaching of microfill, microhybrid and ormocer-based resin composites with **16%** and **37%** of carbamide peroxides and **35%** hydrogen peroxide led to increase whiteness in these composite resins with highly significant difference among the resin composites (**p<0.05**). Higher color change occurred by hydrogen peroxide agent with ormocer-based resin composites.

One of the limitations of Our in vitro study is the lack of saliva; Saliva has washing action that decreases the action of staining solutions, so in vivo study needed to investigate the effect of these beverages on color of composite resins with presence of saliva.

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Table (1) Types of composite resin materials used in this study.

Types	Category	Manufacturer	Composition
Tetric Ceram	Microhybrid	Ivoclar Vivadent Schaan, Liechtenstein	<b>Organic part:</b> Bis-GMA, UDMA, TEGDMA (20.2% wt). <b>Inorganic part:</b> Ba, Al, Yt, Ba-Al-Fl glass, dispersed SiO <sub>2</sub> , spheroid mixed oxide (79 % wt). <b>Filler size:</b> 0.04 - 3.0 µm
Tetric Flow	flowable	Ivoclar Vivadent Schaan, Liechtenstein	<b>Organic part:</b> Bis-GMA, UDMA, TEGDMA (35% wt) <b>Inorganic part:</b> Ba, Al, Yt, Ba-Al-Fl glass, dispersed SiO <sub>2</sub> , spheroid mixed oxide (68.1 % wt.) <b>Fillers size:</b> 0.04-3 µm
Tetric Evo Ceram	nano-hybrid	Ivoclar Vivadent Schaan, Liechtenstein	<b>Organic part:</b> Dimethacrylates (17-18% wt.) <b>Inorganic part:</b> Ba-glass, Ba-Al-silicate glass, YbF <sub>3</sub> , mixed oxide prepolymers, (82-84 % wt.) <b>Filler size:</b> 40-3000 nm



Table (2) Types of staining solutions and bleaching agent used in study.

Material	Type	Composition	Manufacturer	PH of solution
Tea	Lipton Yellow Label Tea	Fine black tea from Ceylon India and Kenya	Unilever, Istanbul, Turkey	5.6
Coffee	Nescafé Classic	5 g coffee (without sugar and milk)	Nestlé Switzerland	5
In-office Bleaching agent	Power whitening WHITEsmile®	38% Hydrogen Peroxide	GmbH · Birkenau, Germany	4.2

Table (3) Mean and standard deviation of total color change  $\Delta E_1$  for all tested composite resins after immersion in tea and coffee beverages. And one way ANOVA comparing among the tested composite resins.

Groups	Subgroups $\Delta E_1$							
	Tea (Te)				Coffee (Co)			
	Mean	SD	F. value	P. values	Mean	SD	F. value	P.value
TC	5.46	0.19	348.16	0.000 HS	6.65	0.34	151.41	0.000 HS
TF	7.27	0.25			8.35	0.5		
TE	4.75	0.21			5.31	0.31		

HS: highly significant.

Table (4) Mean and standard deviation of total color change  $\Delta E_2$  for all tested composite resins after bleaching of the tea and coffee subgroups. And one way ANOVA comparing among the tested composite resins

Groups	Subgroups $\Delta E_2$							
	BTe				BCo			
	Mean	SD	F. value	P. values	Mean	SD	F. value	P. values
TC	3.59	0.24	984.25	0.000	4	0.35	587.57	0.000 HS
TF	6.78	0.24			7.73	0.32		
TE	2.72	0.17			3.35	0.25		

Table (5) Differences between  $\Delta E_1$  and  $\Delta E_2$  for each composite group and student t-test comparing between  $\Delta E_1$  and  $\Delta E_2$ 

Subgroups	$\Delta E_1 - \Delta E_2$	t-test	P-value	Sig.
TCTe vs. TCBTe	1.87	19.34	0.000	HS
TFTe vs. TFBTe	0.49	4.51	0.000	HS
TETe vs. TEBTe	2.03	23.85	0.000	HS
TCCo vs. TCBCo	2.65	17.32	0.000	HS
TFCo vs. TFBCo	0.62	3.24	0.005	HS
TECo vs. TEBCo	1.96	15.8	0.000	HS

Table (6) LSD comparing between composite resins used in  $\Delta E_1$  values.

Subgroups	LSD values	Sig.
TCTe vs. TFTe	1.82	HS
TCTe vs. TETe	0.71	HS
TFTe vs. TETe	2.52	HS
TCCo vs. TFCo	1.701	HS
TCCo vs. TECo	1.33	HS
TFCo vs. TECo	3.03	HS

Table (7) LSD comparing between composite resins used in  $\Delta E_2$  values.

Subgroups	LSD values	Sig.
TCBTe vs. TFBTe	3.18	HS
TCBTe vs. TEBTe	0.87	HS
TFBTe vs. TEBTe	4.05	HS
TCBCo vs. TFBCo	3.73	HS
TCBCo vs. TEBCo	0.65	HS
TFBCo vs. TEBCo	4.38	HS

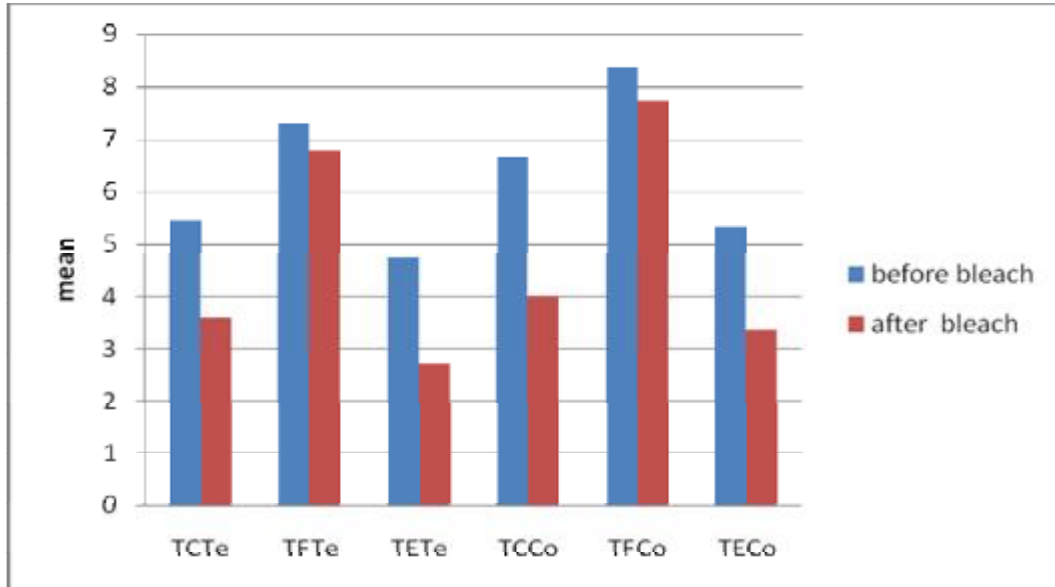


Figure (1) Bar chart shows the mean color change for all groups before and after bleaching.