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Influence of Carbamide Peroxide on the Flexural Strength of Tooth-colored Restorative Materials: An *In Vitro* Study at Different Environmental Temperatures

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Clinical Relevance

The flexural strength of dental materials can be affected by carbamide peroxide. The environmental temperature can be thought to be a factor influencing the bleaching effects on dental materials.

SUMMARY

The current study investigated the influence of carbamide peroxide on the flexural strength of tooth-colored restorative materials at two environmental temperatures. Seven restorative materials were used, including four resin composites (Filtek Z350, Filtek Z250, Synergy Flow and Filtek P60), a polyacid-modified composite (Dyract AP), a conventional glass-ionomer cement (Ketac Molar Easymix) and a ceramic (Vitablocs Mark II). For each type of material, 80 bar-shaped specimens were fabricated and divided into four groups (n=20): bleaching group at 25°C, control group at 25°C, bleaching group at 37°C and control group at 37°C. The specimens in the bleaching groups were treated with 10% carbamide peroxide gel for eight hours/day, while the control specimens in their respective groups were stored in deionized water. After 14-day treatment, the flexural strength of the specimens was determined using a universal testing machine. All the results were analyzed with ANOVA and the Tukey's post-hoc test. The data were also submitted to Weibull distribution. The

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flexural strength and its Weibull distribution of polyacid-modified composite and glass-ionomer cement were more seriously affected by bleaching agents than the resin composite and ceramic, especially at the higher environmental temperature.

INTRODUCTION

Since its introduction, tooth bleaching has attracted a great deal of attention from both dentists and patients.[1] As the most popular approach of the current bleaching technique, night-guard vital bleaching usually involves the application of a moderate concentration of carbamide peroxide (CP) to teeth via a custom-fabricated tray.[2-3] Since bleaching agents may come into contact with restorations, efforts have been made to investigate the effects of CP or hydrogen peroxide (HP) on dental materials.[4] Previous studies have investigated the possible effects of bleaching agents on restorative materials, including changes in color,[5-7] surface microhardness,[8-10] surface roughness[11-13] and surface morphology.[14,15] However, conflicting results have been reported.[5-6,8-14] It was suggested that the contrasting results may be due to differences in bleaching regimens, bleaching agents and the restorative materials used.[16] However, an additional important factor to consider might be environmental temperature. Different environmental temperatures were employed in previous studies; some were carried out at room temperature (about 25°C),[6,8,14] others at 37°C,[5,10-13] while the balance were carried out in the absence of any statement of environmental temperature.[7,9,11,15] In general, the rate of chemical reaction of the bleaching agents can be accelerated by increasing the temperature. For example, a 10°C rise can double the rate of reaction.[17] Possibly, differences in the environmental temperatures during bleaching can contribute to the conflicting results. Therefore, it is important for the bleaching agent to be evaluated for its effects on restorative materials at different environmental temperatures. Unfortunately, there is no research published relating to environmental temperature when evaluating the effects of bleaching agents on dental materials.

Hannig and others[18] reported that bleaching agents soften all the subsurface layers (0.1 mm-2.0 mm) of tested resin composites and polyacid-modified composites. It is conceivable that softening of the subsurface layers leads to a decrease in the flexural strength of bleached restorative materials. However, little information regarding this effect is available.

The current study investigated the effects of CP on the flexural strength of tooth-colored restorative materials at two different environmental temperatures. Two null hypotheses were proposed: 1) environmental temperature has no influence on the bleaching effects of restorative materials; 2) bleaching has no effect on the flexural strength of restorative materials.

METHODS AND MATERIALS

An at-home bleaching system and seven kinds of tooth-colored restorative materials were used in this *in vitro* study (Table 1). The restorative materials used include four resin composites (a nano-hybrid, a microhybrid, a flowable and a packable resin composite), a polyacid-modified composite (compomer), a conventional glass-ionomer cement (CGIC) and an industrially-sintered

Table 1: *Materials Tested*

Material	Code	Batch #	Type	Main Composition[a]	Manufacturer
Filtek Z350	Z350	6018A3	Nano-hybrid resin composite	Combination of aggregated zirconia/ silica cluster filler, Bis-GMA, UDMA, TEGDMA and Bis-EMA	3M ESPE, St Paul, MN, USA
Filtek P60	P60	8100A3	Packable resin composite	Zirconia/silica filler, Bis-GMA, UDMA and Bis-EMA resins	3M ESPE, St Paul, MN, USA
Filtek Z250	Z250	1370A3	Micro-hybrid resin composite	Zirconia/silica filler, Bis-GMA, UDMA and Bis-EMA resins with small amounts of TEGDMA	3M ESPE, St Paul, MN, USA
Synergy Flow	FLOW	0090777	Flowable resin composite	Bis-GMA, Bis-EMA, TEGDMA, strontium glass, silanized amorphous silica, hyrophobed	Coltène/ Whaledent AG, Altstätten, Switzerland
Dyract AP	DY	0706000707	Polyacid-modified composite	UDMA, TCB resin, Alkanoyl-poly-methacrylate, strontium-fluoro-silicate glass, strontium fluoride, photo initiators, butyl hydroxy toluene, iron oxide pigments	Dentsply DeTrey GmbH, Konstanz, Germany
Ketac Molar Easymix	KM	305242	Conventional glass-ionomer	Polycarboxylic acid, aluminum-calcium-lanthanum fluorosilicate glass	3M ESPE AG, Seefeld, Germany
Vitabloc Mark II for Cerec	MK2	6902	Machinable feldspathic porcelain	Aluminum oxide and silica	Vita Zahnfabrik, Bad Säckingen, Germany

[a] *Manufacturer's data*

ceramic material used for CAD/CAM restorations. All represent the most commonly used categories of tooth-colored restorative materials.

For each kind of restorative material, 80 bar-shaped specimens (12 mm length × 2 mm width × 2 mm height, A3 shade) were fabricated. The ceramic specimens were made from Vitablocs Mark Ⅱ for Cerec (Vita Zahnfabrik, Bad Säckingen, Germany) using a water-cooled diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). The specimens from the remaining six materials were fabricated with silicone molds according to their respective manufacturers' instructions. All the specimens were then stored in deionized water at 37°C for 24 hours. After storage in deionized water, the specimens were ground and polished on the four longest surfaces by the same operator using medium, fine and superfine discs (Sof-Lex, 3M ESPE, St Paul, MN, USA) rotating in one direction. The width and height of the polished specimens were each measured three times with a digital micrometer (Mitutoyo, Tokyo, Japan) and the mean values were recorded to calculate the flexural strength. Subsequently, all the specimens were stored in 37°C deionized water for seven days.[19] Prior to the bleaching treatment, the specimens were covered with sculpturing wax (Shanghai Medical Instruments Co, Ltd, Shanghai, China), excluding one randomly selected longitudinal surface (bleaching surfaces).

The specimens from every restorative material were equally divided into four groups (n=20): bleaching group at 25°C (group 25B), control group at 25°C (group 25C), bleaching group at 37°C (group 37B) and control group at 37°C (group 37C).

Bleaching Procedure

Opalescence PF 10% (Ultradent, South Jordan, UT, USA), a commercially available at-home bleaching system containing 10% CP, was employed in the current study. The specimens in groups 25B and 37B were treated with bleaching gel eight hours a day for 14 days in a wet chamber at 25°C and 37°C, respectively. The bleaching gel was applied directly onto the bleaching surfaces of the 25B and 37B specimens at a 0.5 mm thickness. At the end of each bleaching procedure, the bleached samples were cleaned with a soft toothbrush under running deionized water. They were then placed in deionized water until the next application at 25°C and 37°C, accordingly. With regards to groups 25C and 37C, the specimens were stored in deionized water at 25°C and 37°C, respectively, for the duration of the experiment. The deionized water used for storage was renewed every day.

Flexural Strength Measurements

Subsequent to the 14-day treatment procedure, the covering wax was carefully removed from each specimen. The flexural strength tests for all the specimens were done with a universal testing machine (Instron Model 8841, Canton, MA, USA) at a crosshead speed of 1.0 mm/minute until the specimens fractured. During testing, the specimen was placed on the supports with its bleaching surface facing the crosshead of the testing machine to simulate biting stress on the surface.

The flexural strength (σ) values were calculated based on the following equation[20]:

$$\sigma = \frac{3FL}{2BH^2} \quad (1)$$

where F is the failure load (in N), L is the distance between the supports (in mm, 10 mm in this study) and B and H are the width and height of the specimen, respectively (all in mm).

Statistical Analysis

Statistical analysis was performed with the SPSS statistical software package SPSS 13.0 for Windows (SPSS, Chicago, IL, USA). Normal distribution was checked by the Kolmogorov-Smirnov test. The level of significance was established as α=0.05. One-way analysis of variance (ANOVA) with the Tukey's post-hoc test was used to analyze the results of flexural strength.

To determine the clinical significance of the fracture analysis, data obtained from the flexural strength were also submitted to Weibull distribution.

According to the Weibull distribution,[21-22] the probability of failure (P_f) of a brittle material can be calculated with the following equation:

$$P_f = 1 - \exp\left(-\left(\frac{\sigma}{\sigma_o}\right)^m\right) \quad (2)$$

where m is the Weibull modulus related to the dispersion of the failure data, σ_0 is the characteristic strength representing the stress level in which 63.21% of the specimens will fail and σ is the strength at a given P_f.

The probability of failure can be estimated from the equation below:

$$P_f = 1 - \frac{i}{n+1} \quad (3)$$

where i is the ranking of the strength data of ascending order and n is the number of specimens.

Using equations 2 and 3, the Weibull modulus and the characteristic strength of the tested materials can be calculated through linear regression on a plot of the double logarithm of 1/(1- P_f) against the logarithm of the strength values. The slope of this line is the Weibull modulus, while the intercept at the Y-axis is $m\ln(\sigma_0)$.[23]

Table 2: *Mean Values (MPa) and Standard Deviation (in parentheses) of Flexural Strength and* p*-values of Multiple Comparisons*

Material	**Group**				***p*-values**			
	25C	**25B**	**37C**	**37B**	**25C vs 25B**	**37C vs 37B**	**25C vs 37C**	**25B vs 37B**
Z350	154.25 (18.84)	149.28 (15.26)	155.12 (17.69)	156.34 (17.22)	0.801	0.996	0.999	0.573
P60	162.46 (18.94)	167.13 (20.09)	159.14 (20.52)	159.82 (16.46)	0.865	0.999	0.946	0.621
Z250	173.94 (16.44)	169.19 (14.28)	170.97 (15.50)	173.11 (16.30)	0.773	0.973	0.932	0.858
FLOW	134.29 (16.60)	135.49 (15.97)	136.14 (18.18)	132.25 (16.61)	0.996	0.885	0.986	0.929
DY	103.41 (17.72)	90.55 (16.10)	98.44 (16.99)	76.16 (17.82)	0.092	0.001*	0.798	0.047*
KM	35.17 (8.01)	31.00 (8.75)	32.54 (7.99)	27.60 (9.70)	0.427	0.278	0.770	0.601
MK2	133.72 (8.67)	137.18 (9.43)	137.24 (9.90)	131.55 (8.31)	0.628	0.149	0.508	0.215

**Statistically significantly different.*

The 95% confidence intervals (CI) of m and σ_0 were then calculated. Two experimental groups were considered to be statistically similar for Weibull parameter m or σ_0 when their CI overlapped. The cumulative failure distribution of flexural strength data was generated to estimate failure probability at different levels of stress.

RESULTS

The flexural strength results of all the materials are shown in Table 2. For DY, the group 37B showed significantly lower flexural strength compared to the group 37C (bleaching effect). Moreover, the flexural strength of the group 37B specimens was significantly lower than that of the group 25B specimens (temperature effect). No statistically significant differences were found in any other multiple comparisons for the remaining six types of tested materials.

The Weibull parameter σ_0, m and their respective 95% CI for all the groups are listed in Table 3. The high cor-

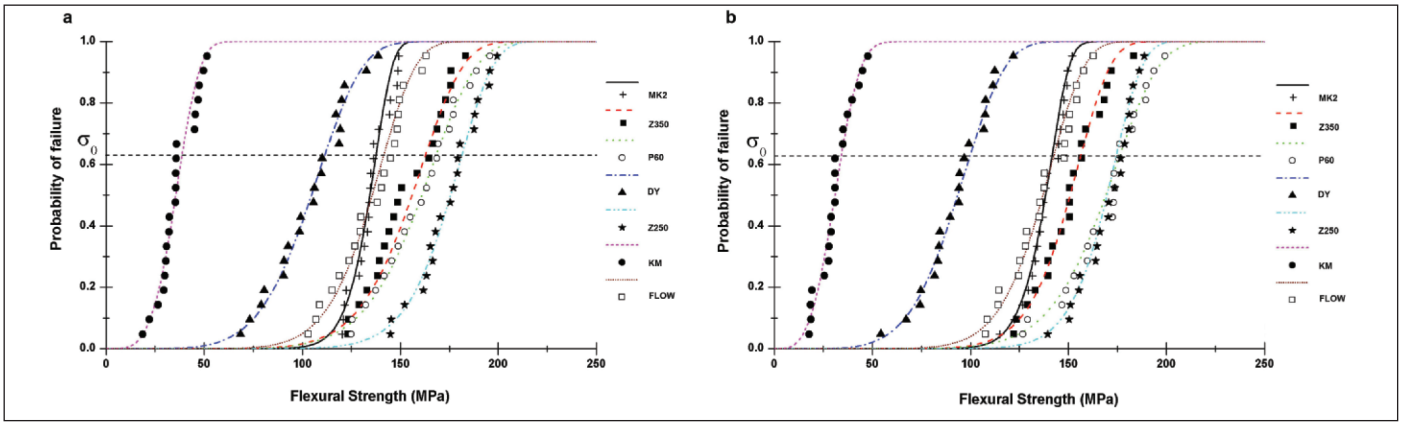

Figure 1: *Weibull cumulative failure curves of the samples treated at 25°C: (a) 25C groups; (b) 25B groups.*

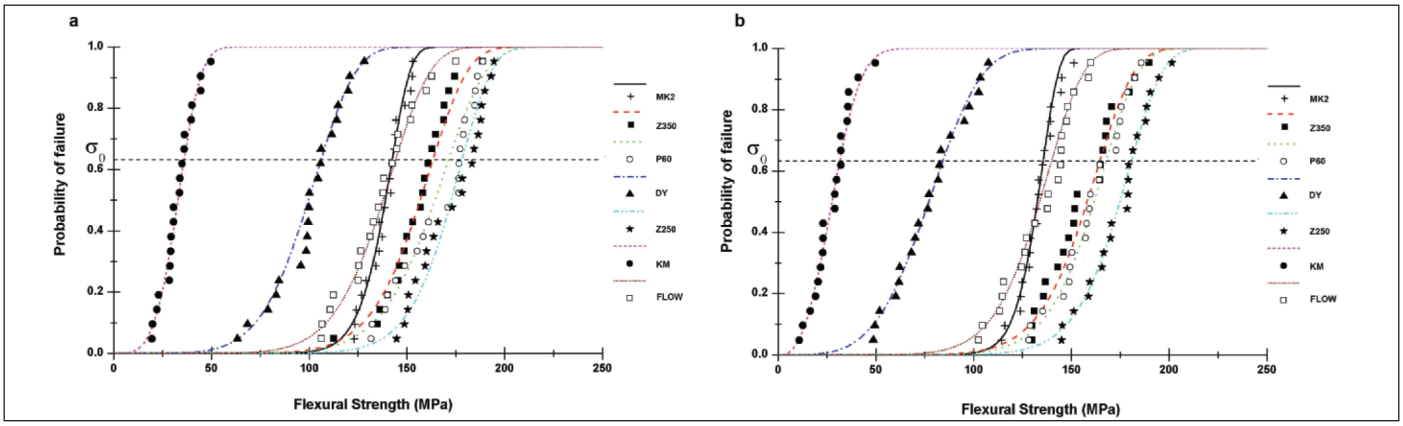

Figure 2: *Weibull cumulative failure curves of the samples treated at 37°C: (a) 37C groups; (b) 37B groups.*

Table 3: *Weibull Analysis of Flexural Strength of All Materials*

Material	Group	Weibull Modulus (m)	95% CI of m	Characteristic Strength (σ_0) (MPa)	95% CI of σ_0	Correlation Coefficient
Z350	25C	8.40	7.68-9.11	162.96	154.18-172.24	0.97
	25B	10.20	8.68-11.73	156.40	149.43-163.69	0.92
	37C	9.07	8.30-9.85	163.24	155.08-171.83	0.97
	37B	9.27	7.71-10.84	164.44	156.39-172.90	0.91
P60	25C	8.79	7.93-9.64	171.28	162.45-180.58	0.95
	25B	8.67	8.14-9.20	176.29	167.08-186.00	0.98
	37C	8.07	7.11-9.03	168.49	159.06-178.48	0.96
	37B	9.85	8.22-11.46	167.69	159.96-175.80	0.90
Z250	25C	11.15	10.81-11.49	181.51	174.10-189.24	0.98
	25B	12.19	10.79-13.59	174.28	167.76-181.06	0.94
	37C	11.57	10.90-12.24	178.19	171.17-185.50	0.94
	37B	11.20	10.19-12.21	180.60	173.26-188.26	0.97
FLOW	25C	8.57	8.26-8.89	141.65	134.17-149.55	0.98
	25B	9.00	8.46-9.54	142.66	135.48-150.22	0.99
	37C	7.97	7.26-8.69	144.16	135.99-152.82	0.96
	37B	8.31	7.73-8.89	139.75	132.14-147.79	0.98
DY	25C	5.73	4.93-6.52	111.59	102.89-121.02	0.93
	25B	5.55 ⌉	5.33-5.77	99.97	91.94-108.71	0.98
	37C	5.91 ⌉ *	5.53-6.30	105.96	97.94-114.63 ⌉	0.97
	37B	4.14 ⌋ * ⌋	3.73-4.84	84.15	75.21-94.15 ⌋ *	0.96
KM	25C	4.21	3.56-4.86	38.74	34.69-43.26	0.92
	25B	3.58	3.14-4.01	34.48	30.28-39.26	0.94
	37C	3.90	3.53-4.44	36.04	31.99-40.60	0.94
	37B	2.79	2.60-2.99	31.18	26.39-36.83	0.97
MK2	25C	15.89 ⌉	13.74-18.04	137.64	133.67-141.73	0.93
	25B	15.32 ⌉ *	14.41-16.23	141.64	137.41-146.01	0.98
	37C	14.79 ⌋ * ⌋	13.20-16.38	142.36	137.95-146.91	0.95
	37B	16.85	16.17-17.53	135.45	131.76-139.24	0.99

**Statistically significantly different.*

relation coefficients (all ≥0.90) indicated close data adherence to the Weibull distribution. In the current study, the m values ranged from 2.79 to 16.85. For DY and KM, the group 37B showed significantly lower m values than the group 37C. Under the bleaching treatment at 37°C, the DY specimens exhibited a statistically significant decrease in characteristic strength (σ_0) compared to the control specimens at 37°C (bleaching effect). Furthermore, bleaching at 37°C produced statistically significant influences on the m values of DY and KM compared to the corresponding specimens bleached at 25°C (temperature effect). Figures 1 and 2 show the cumulative probability of failure as a function of stress developed in the specimens treated at different environmental temperatures. For the DY specimens, bleaching at 37°C affected the flexural strength, as shown by the significant shift of the cumulative failure curve to the left compared with the control specimens at 37°C.

Apart from the effects mentioned above, bleaching at either 25°C or 37°C had no significant influence on the flexural properties of any other tooth-colored restorative materials tested. Moreover, storage in deionized water at either 25°C or 37°C did not produce any statistically significant influence on the flexural properties of the seven esthetic restorative materials.

Based on the results described above, the two null hypotheses—that the bleaching treatment has no effect on the flexural strength of the dental materials and the environmental temperature has no influence on the bleaching effects on the dental materials—were rejected.

DISCUSSION

Flexural strength, defined as the failure stress of a material as measured in bending, is generally considered to be a meaningful mechanical property for brittle materials that are much weaker in tension than in compression.[24] As stipulated in ISO 4049,[25] the dimension of the beam specimens for the flexural strength test should be 25 mm × 2 mm × 2 mm. However, the recommended length is not clinically realistic. Furthermore, the specimens that match the ISO dimension are difficult to prepare without flaws. Therefore, the dimension of the specimens in the current study was 12 mm × 2 mm × 2 mm, which is analogous to the method described by Yap and Teoh.[26]

Although CGIC was categorized as a filling material, it exhibited low flexural strength in the current study. A possible reason may be that the specimens were tested in air. It has been pointed out that CGIC would

Table 4: p-*values of Multiple Comparisons of Microhardness at Different Subsurface Levels*

Material		**0.1 mm**	**0.2 mm**	**0.3 mm**	**0.5 mm**	**1.0 mm**
Z350	25C vs 25B	0.651	0.972	0.927	0.665	0.798
	37C vs 37B	0.979	0.353	0.987	0.990	0.885
	25C vs 37C	0.912	0.550	0.998	0.942	0.590
	25B vs 37B	0.999	0.864	0.867	0.815	0.982
P60	25C vs 25B	0.944	0.981	0.996	0.977	0.844
	37C vs 37B	0.894	0.765	0.993	0.937	0.999
	25C vs 37C	0.910	0.900	0.954	0.996	0.931
	25B vs 37B	0.848	0.910	0.742	0.799	0.986
Z250	25C vs 25B	0.996	0.840	0.996	0.997	0.917
	37C vs 37B	1.000	1.000	0.983	0.493	0.989
	25C vs 37C	0.974	0.995	0.991	1.000	1.000
	25B vs 37B	0.993	0.949	0.970	0.571	0.986
FLOW	25C vs 25B	0.796	0.778	0.971	1.000	0.769
	37C vs 37B	0.458	0.999	0.996	0.670	0.986
	25C vs 37C	0.996	0.871	0.994	1.000	0.975
	25B vs 37B	0.985	1.000	1.000	0.695	0.810
DY	25C vs 25B	0.029*	0.986	0.797	0.996	0.903
	37C vs 37B	<0.001*	0.003*	0.029*	0.044*	0.581
	25C vs 37C	0.988	0.964	0.788	0.563	0.067
	25B vs 37B	<0.001*	<0.001*	0.031*	0.619	0.219
KM	25C vs 25B	1.000	0.991	0.997	0.992	0.869
	37C vs 37B	<0.001*	0.034*	0.967	0.951	0.707
	25C vs 37C	0.970	0.999	0.981	1.000	0.853
	25B vs 37B	<0.001*	0.022*	0.916	0.856	0.728
MK2	25C vs 25B	1.000	0.986	0.801	0.807	0.691
	37C vs 37B	0.878	0.862	0.995	1.000	0.745
	25C vs 37C	0.999	0.817	0.995	0.880	0.952
	25B vs 37B	0.850	0.999	0.975	0.996	0.952

*Statistically significantly different.

dehydrate on exposure to air and form microcracks on the surface. Thus, the low flexural strength may be partially due to the current testing conditions.[27] After bleaching at 37°C, the compomer showed a significant decrease in flexural strength. This result correlated well with the surface and subsurface microhardness loss of bleached compomer.[18-19] The severe softening of surface and subsurface layers of compomer could have contributed to the reduction in flexural strength.

On the other hand, it has been suggested that the strength distributions of quasi-brittle materials are more properly described by Weibull statistics, rather than the mean strength values determined based on a Gaussian strength distribution.[28] Therefore, the Weibull distribution was employed to analyze the flexural strength data. The Weibull distribution provides a way of assessing dependability of the material and disclosing the probability of failure at any selected level of stress. As a measure of the variability of flexural strength in a brittle material and its dependence on flaw size distribution, a higher m, even in association with slightly lower mean fracture strength, is often preferable to a lower m associated with a higher mean fracture strength. From a clinical viewpoint, knowing σ_0, which is defined as the stress level in which 63.21% of the specimens will fail, is also important.

In the current study, resin composites demonstrated a similar Weibull modulus, ranging from 7.97-11.97, which is in agreement with a previous study.[23] After bleaching at 37°C for 14 days, the compomer and CGIC exhibited degraded flexural properties. This indicates that bleaching at 37°C could affect the reliability of the clinical performance of the compomer and CGIC. Characteristically, brittle materials present a population of flaws of different sizes, geometries and orientations. Fracture occurs when the load exceeds a critical value for propagation of the largest and most favorably oriented flaw.[29] Therefore, for the phenomenon observed, two factors could be responsible: some preexisting flaws have been modified or a new flaw population was introduced when the compomer and CGIC specimens were bleached with 10% CP at 37°C. These flaws might act as stress concentrators, lowering the fracture stability of the materials over time.[30] Furthermore, according to the cumulative probability of failure depicted in Figures 1 and 2 at the stress level responsible for the failure of 63.21% of the KM specimens, the probability of failure of the four groups in the other six materials was zero. At the characteristic strength of the flowable resin composite, the probability of failure of the other three resin composites was in a range between 10% and 20%. This result may be due to differences in the filler content of Z350 (59.5% by volume), P60 (61% by volume), Z250 (60% by volume) and Flow (32% by volume).[31]

The results of the current study indicate that CP had a greater effect on the flexural properties of tooth-colored restorative materials at the higher temperature. The following factors may explain this finding: 1) the bleaching agent may induce oxidative cleavage of polymer-chains and impact the resin-filler-interface of dental materials. Based on previous findings, peroxide diffusion can be accelerated by heating. The release of hydroxyl-radicals from peroxide is accelerated by a rise in temperature based on the following equation: $H_2O_2 + 211\ \text{kJ/mol} \rightarrow 2HO\bullet$. This is in accordance with an increase in speed of decomposition of a factor of 2.2 for each temperature rise of 10°C.[32] When the temperature rises, it is conceivable that there is an increase in the release of free radicals from the bleaching gels. The results from the current study were in agreement with this hypothesis. The loss in flexural strength of the compomer bleached at 37°C was about two times more than the specimens bleached at 25°C. At the higher environmental temperature, the bleaching agents could be more reactive and may penetrate into deeper layers of the dental material, producing a more pronounced effect on the mechanical properties of the dental material; 2) a rise in temperature brings about a greater dilation of the restorative materials.[33] This may make CGIC and compomer that is more prone to the effects of the bleaching agents. Further studies are needed to confirm these hypotheses. However, it should be considered that the effects of the bleaching agents could be reduced by dilution and buffering of saliva in the oral cavity.

Based on the findings of the current study, both dentists and material scientists should be aware that, when evaluating the effects of bleaching agents on dental materials, the environmental temperature needs to be taken into account both in the design of a test protocol and the interpretation of the results.

This *in vitro* study is considered to be the first attempt to investigate the effects of CP on the flexural strength of tooth-colored restorative materials at different environmental temperatures. However, there are some limitations that need to be noted. Although the specimens were bleached and stored at 25°C or 37°C, the flexural strength tests were not conducted in a water bath at the respective temperatures due to technical constraints. Furthermore, to maximally simulate a real clinical situation, future studies are needed to investigate the mechanical properties of tooth-colored restorative materials under cyclic loading.

CONCLUSIONS

Under the conditions of this *in vitro* study, the following conclusions can be drawn:

1) The effects of 10% CP on the flexural strength and its Weibull distribution of tooth-colored restorative materials were material dependent.
2) Bleaching at 37°C affected flexural strength and its Weibull distribution of compomer and CGIC. Bleaching at 37°C showed more pronounced effects on compomer and CGIC compared with bleaching at 25°C.
3) The flexural properties of ceramic and resin composite remained stable, regardless of whether or not it has been bleached.
4) No significant differences were found in the flexural properties of unbleached control specimens stored at 25°C and 37°C.

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