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Effect of Thin-Layer Graphene Doping on The Color and Surface Hardness of Dental Ceramic

Doping Yöntemi ile İnce Bir Tabaka Grafen Uygulamasının Dental Seramiklerin Rengine ve Sertliğine Etkisi

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Abstract

Objective: The most common cause of clinical failures in all-ceramic dental restorations is crack formation in the veneering ceramic. The aim of this study was to determine whether graphene doping would change the characteristics of hardness and discoloration of the ceramic veneer surface.

Material-Method: Thirty disk-shaped cores (10 mm in diameter and 0.8 mm in thickness) were prepared. Three different ceramic systems, IPS Empress (E) (Ivoclar Vivadent, Schaan, Liechtenstein), IPS e.max Press (EP) (Ivoclar Vivadent, Schaan, Liechtenstein), and Turkom Cera (TC) (Turcom-Ceramic SDN-BHD, Kuala Lumpur, Malaysia) were tested, each with n=10. The Vickers hardness and color difference (ΔE) values were measured before and after doping with graphene. Surface analysis was performed with XRD, XPS, and SEM. The Wilcoxon signed-rank test was performed to compare hardness values. The Kruskal-Wallis test was performed to compare ΔE values among all groups. The Kruskal-Wallis one-way ANOVA was used for the post hoc tests after the Kruskal-Wallis test (α=0.05).

Results: A significant difference was found among the groups and the mean values of ΔE (p=0.002). According to the post hoc test results, this difference was found between TC and E groups (p=0.002). Although graphene doping increased hardness significantly in group E, it was also found to decrease in group TC. The mean ΔE values indicated clinically noticeable (over the limit of 3.7) color change in all groups.

Conclusions: Graphene doping may change the surface hardness of dental ceramics depending on the content of the ceramic. Similarly, depending on the content of the ceramic, it may affect its color to varying degrees. Graphene doping increased surface hardness only in group E but negatively affected the color of ceramic. Its application could be useful in the palatal region.

Keywords: Dental ceramics, Color, Doping, Hardness.

Özet

Amaç: Tam seramik dental restorasyonlarda klinik başarısızlıkların en sık nedeni veneer seramiklerinde çatlak oluşumudur. Bu çalışmanın amacı, grafen dopingin seramik kaplama yüzeyinin sertlik ve renk bozulma özelliklerini değiştirip değiştirmeyeceğini belirlemektir.

Materyal-Metot: Otuz adet disk şeklinde örnek (10 mm çapında ve 0.8 mm kalınlığında) hazırlandı. Üç farklı seramik system kullanıldı, IPS Empress (E) (Ivoclar Vivadent, Schaan, Lihtenştayn), IPS e.max Press (EP) (Ivoclar Vivadent, Schaan, Lihtenştayn) ve Turkom Cera (TC) (Turcom-Seramik SDN-BHD, Kuala Lumpur, Malezya), her birinden (n=10) adet test edildi. Vickers sertliği ve renk farkı (ΔE) değerleri grafen doping öncesi ve sonrasında ölçüldü. Yüzey analizi, XRD, XPS ve SEM ile yapıldı. Sertlik değerlerini karşılaştırmak için Wilcoxon signed-rank testi uygulandı. Tüm gruplar arasında ΔE değerlerini karşılaştırmak için Kruskal-Wallis testi uygulandı. Kruskal-Wallis sonrası post hoc testlerinden Kruskal Wallis tek yönlü ANOVA kullanıldı (α=0,05).

Bulgular: Gruplar arasında ve ΔE'nin ortalama değerleri arasında anlamlı bir fark bulundu (p=0,002). Post-hoc test sonuçlarına göre, bu fark TC ve E grupları arasında bulundu (p=0,002). Her ne kadar grafen doping E grubunda sertliği anlamlı olarak arttırsa da, TC'de de azaldığı bulundu. Ortalama ΔE değerleri, tüm gruplarda klinik olarak belirgin (3,7 sınırın üzerinde) renk değişikliğini göstermiştir.

Sonuç: Grafen doping, seramiklerin içeriğine bağlı olarak dental seramiklerin yüzey sertliğini değiştirebilir. Benzer şekilde, seramik içeriğine bağlı olarak, rengini değişen derecelerde etkileyebilir. Grafen doping, yalnızca E grubunda yüzey sertliğini arttırdı, ancak seramiğin rengini olumsuz yönde etkiledi. Palatal bölgede kullanımı faydalı olabilir.

Anahtar kelimeler: Dental seramik, Renk, Doping, Sertlik.

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Introduction

Although IPS e.max Press (lithium disilicate glass ceramic system) has the same composition as IPS Empress II, its physical properties and appearance have been improved (1). Turkom-Cera was developed as a high purity alumina (99.98%) with better fracture resistance than the other available ceramic systems (2). Dental ceramic restorations are usually formed in layers (3), with aesthetic but weak veneer ceramics layered on dense and strong ceramic substructures. The most common cause of clinical failure is crack formation in the veneer ceramics (4). Improvements at the surface level are often needed to prolong the lives of materials. According to Griffith's theory of brittle fracture (5), ceramic breakage is caused by microcracks within the ceramic, rather than by atomic bond breakage. Microcracks in ceramics proliferate continuously under stress and are connected to each other. This makes the actual failure forces in ceramics lower than their theoretical values (6). If crack propagation conditions can be controlled, the mechanical properties of the ceramics can be improved to some extent (7). Factors that exceed the brittleness of bioactive ceramics and increase their mechanical performance include an increased resistance to crack propagation in the ceramic and a reduction of crack defects in the ceramic, thereby alleviating stress concentration at the crack tip (8, 9). Over the last few years, interest in low-dimensional nanomaterials (LDNM) including carbon nanotubes, graphene and boron nitride nanotubes has increased because of their favorable biocompatibility, large surface-specific areas, and high mechanical properties (10). Graphene is considered the strongest material known due to the flexibility of bonds between carbon atoms (11).

The combination of two or more materials may result in composites having different physical and chemical properties and higher mechanical properties. Because of their advanced capabilities, composites are widely used in dentistry (12). However, studies of graphene/ceramic composites are scarce, and the authors are unaware of studies on the application of doping to dental ceramics. Graphene can promote the proliferation and activity of osteoblasts (13). In addition, in vivo studies have reported that the addition of graphene has no adverse effect on the organic microenvironment of mice (14, 15).

However, there are some problems that need to be solved for graphene-ceramic composites. First, graphene with an oxidation temperature of 600°C is likely to be structurally damaged during the sintering of ceramics (16). The second is the collection of graphene, which may affect the performance of graphene. The strong van der Waals force between the layers can easily lead to this, making it difficult to obtain a homogeneous distribution in the matrix (17).

Teeth and dental restoratives are semi-translucent materials that allow partial light transmission, the color of which can be measured with spectrophotometers (18-21). O'Brien et al. (22) interpreted clinical differences by classifying acceptable ΔE values. A value of $\Delta E \geq 3.7$ can be detected by 100% of observers and identifies a clinically detectable color difference. In restorations in which the ΔE value is greater than 3.7, the color difference has been interpreted as clinically incompatible. The aim of this study was to investigate the effects of graphene doping on the surface hardness and color change of three different ceramic systems produced in a standard color (A1). The null hypothesis was that doping would not affect the color or the surface hardness of the ceramic.

Material and Methods

Ten disks (10 mm in diameter, 0.8 mm in thickness) each of IPS Empress and IPS e.max-Press were prepared with a framework thickness of 0.8 mm and a veneer thickness of 0.5 mm. IPS Empress and IPS e.max Press disks were prepared by using the IPS Empress special wax (Elastiwax; Ivoclar Vivadent AG) in accordance with the manufacturer's recommendations. Dental wax specimens were placed in phosphate-bonded investment material (IPS Empress, Esthetic Speed Investment; Ivoclar Vivadent AG). The pressing procedure for IPS Empress (Shade Vita A1) was about 20 minutes at 1075°C. MO1 (Medium Opacity) ingots (Vita A1 color) for the IPS e.max-Press specimens were pressed at 960°C. Ten disks of Turkom-Cera alumina gel (Turkom-Ceramic SDN-BHD) were used for Turkom-Cera frameworks and sintered at 1150°C for 5 minutes after 6 hours of drying. Crystallization was completed by applying granulated glass (no. 2 Turkom-Cera Crystal Hardener; Turkom-Ceramic SDN-BHD) for 45 to 60 minutes at 1150°C. All specimens were measured by using a digital caliper. The recommended veneering ceramics (in A1 color) were applied in thicknesses of 0.5 mm over the frameworks: IPS Empress Aesthetic Veneer for E; IPS e.maxCeram for EP; and VITA VM 7 for T. The IE and IEP specimens were fired in the EP600 vacuum porcelain furnace, and the TC specimens were fired in the P300 vacuum porcelain furnace at the manufacturers' recommended firing temperatures for the recommended times. After the Graphene Nanoplatelet powder (99.5%, 6 nm, NANOGRAFI CO. LTD) were added on veneer ceramics, specimens were fired in the P300 vacuum porcelain furnace at the manufacturers' recommended firing temperatures.

After the graphene doping on the veneer ceramic surfaces color measurements were performed with a spectrophotometer (Spectro ShadeTM MICRO; MHT Optic Research AG). Measurements were made under the D65 standard lighting conditions corresponding to daylight, and the device was calibrated before each measurement. Measurements were performed on a standard white background (L=92.9, a=1.2, b=-2.4), and the mean CIEL*a*b* value was obtained by measuring each specimen three times. The ΔE values of the ceramic specimens were calculated using the following formulae: ΔE=[(ΔL)2+(Δa)2+(Δb)2] ½ (ΔE=color difference), where ΔL=L2*-L1* (ΔL=brightness values); Δa=a2*- a1* (Δa=determined the difference in the red-green scale); and Δb=b2*-b1* (Δb=determined the difference in the green-yellow scale). The L2, a2, and b2 values represented the CIEL*a*b* values measured after coating of the ceramic specimens, and the L1, a1, and b1 values represented the initially measured CIEL*a*b* values (23).

The color differences (ΔE) and Vickers hardness values (VHN) of the specimens prepared using 3 different ceramic systems (n=7) were measured before and after graphene doping. Surface analyses were performed with XRD, XPS, and SEM. The obtained data were analyzed using statistical software (PASW Statistics v18.0; SPSS Inc.). The normal distribution of the quantitative variables was examined with the Shapiro Wilk test. Kruskal-Wallis tests were performed to compare ΔE values among all groups, and the Kruskal-Wallis one-way ANOVA was used for the post hoc tests after the Kruskal-Wallis test (α=0.05). The Wilcoxon Signed Ranks Test was used to compare hardness values before and after doping.

The surface chemistry of a specimen from group TC, graphene doped, was investigated by X-ray photoelectron spectroscopy (XPS) (Figure 1). The analyses were performed with a Specs Flex-Mod. Monochromatized AlKα X-rays with an excitation energy of 1486.7 eV were used. Signals were detected by using a 2D CCD detector with a 150-mm hemispherical energy analyzer.

The surfaces of a specimens from the TC group, graphene doped, were examined by XRD (Figure 2). X-ray diffraction measurement was performed by a PANalytical Empyrean X-ray diffractometer with CuKα (λ=1.5406 Å). Measurements were performed between 20 and 90 degrees. Scanning electron microscope (SEM) images (Figure 3) were made from a single specimen of each group after the specimens had been stored in distilled water for 24 hours at 37 °C. The initial Vickers hardness values (VH) (Shimadzu Dynamic HMV-G 21) of all the ceramic specimens were then measured under a 0.98-N load with a 10-second dwell time prior to coating. Five different measurements were made from each specimen, and the arithmetic mean was obtained (24). The hardness of each specimen was remeasured after the coatings were applied (Figure 4).

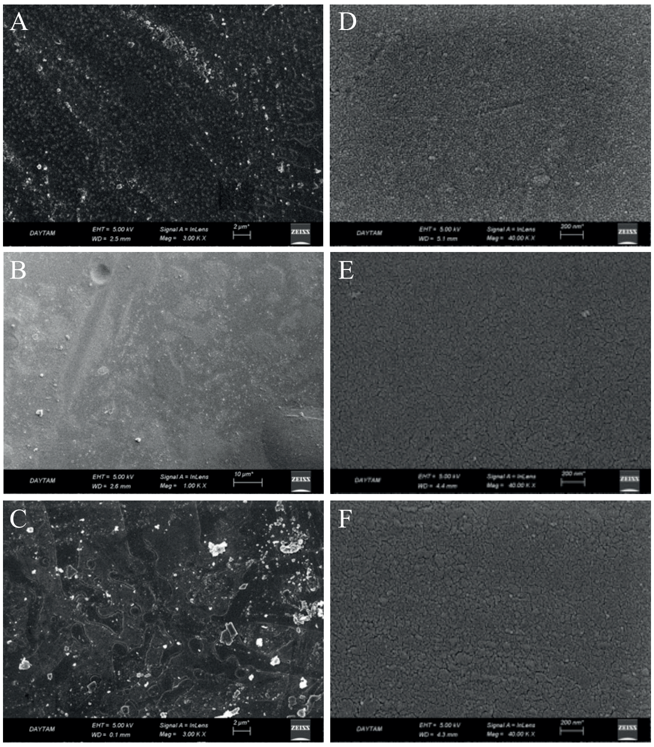

Figure 3. SEM Images of with and without graphene doping
A: Coated IPS e.max Press, B: Coated Turkom Cera, C: Coated IPS Empress, D: Uncoated IPS e.max Press, E: Uncoated Turkom Cera, F: Uncoated IPS Empress

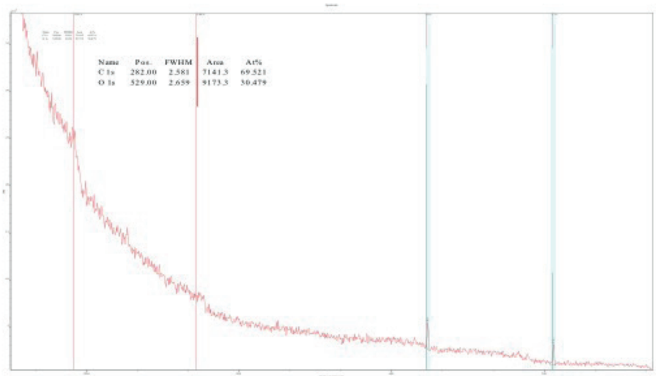

Figure 1. IXPS analysis: The appearance of elements C and O indicates successful doping

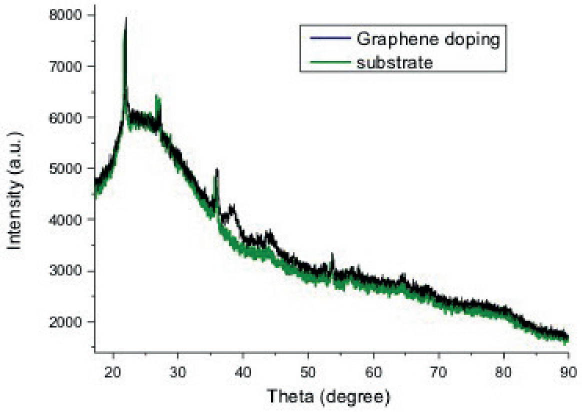

Figure 2. XRD (X-ray diffractometer) analysis. Peak at 36.73 represents graphene

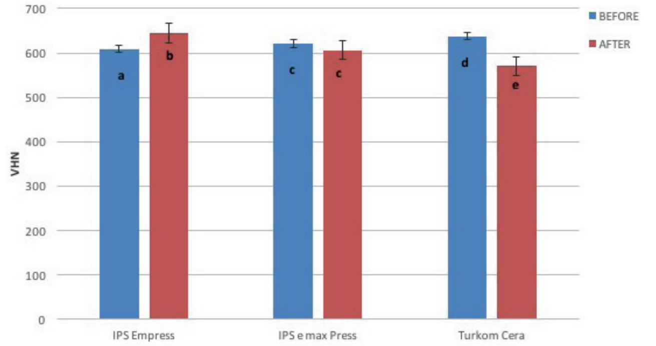

Figure 4. Vickers hardness values before and after graphene doping

Results

The mean ΔL, Δa, Δb, ΔE was obtained from all groups, and the results of the statistical comparison are shown in Figure 5 (different letters indicate significant differences). Significant differences were found among the groups as a result of the Kruskal-Wallis test, which was performed to compare ΔE values between the groups (p=0.002). According to post hoc test results, this difference was found between the TC and E groups (p=0.002). The color darkened as a result of graphene doping. Regarding the mean ΔE values, color change was higher than the clinically noticeable limit (22) of ΔE=3.7

in all groups (Table 1). In summary, the graphene doping contributed to the hardness of group E.

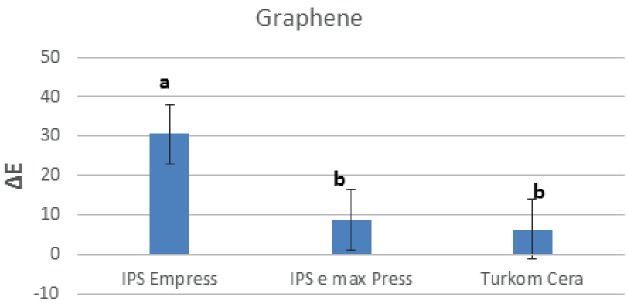

Figure 5. Color change before and after doping

Table 1. Statistical analysis values for color differences

		Ceramic Materials		
		IPS Empress	**IPS e.max Press**	**Turkom Cera**
ΔE	Mean	30.46	8.62	6.40
	Standard Deviation	0.74	2.23	0.73
	Median	30.30	7.70	6.30
	Minimum	29.50	6.90	5.50
	Maximum	31.30	12.50	7.50

Table 2. Statistical analysis values for hardness

			HARDNESS1	HARSNESS2
Ceramic Materials	**IPS Empress**	Mean	608.60	644.40
		Standard Deviation	0.89	26.88
		Median	608.00	635.00
		Minimum	608.00	617.00
		Maximum	610.00	673.00
	IPS e.max Press	Mean	620.60	606.00
		Standard Deviation	9.66	20.78
		Median	619.00	591.00
		Minimum	608.00	591.00
		Maximum	635.00	633.00
	Turkom Cera	Mean	638.00	570.80
		Standard Deviation	16.32	46.39
		Median	645.00	597.00
		Minimum	618.00	510.00
			652.00	611.00

The results of the Wilcoxon signed-rank test to compare the hardness values before and after doping between the groups indicated a significant difference in the other two ceramics except the IPS e.max Press group (p=0.136) (Table 2). Although graphene doping increased hardness significantly in group E (p=0.043), hardness decreased in the TC group (p=0.042).

The presence of the C and O element in the XPS analysis demonstrated that the coating had been made. The binding energy of the advantageous C element was used to correct the spectrum. An almost stoichiometric graphene film was obtained as seen from the inset Figure 1. According to XRD analysis, sharp peaks came from the substrate. In contrast, the graph shown on the black line shows 2 broad peaks. Graphene peaks appear at 36.73. The results of SEM analysis before and after doping show that was done successfully.

Discussion

Although LDNMs are potential nanofillers for strengthening bioactive ceramics, maintaining their structural stability during the fabrication of ceramic composites has been difficult due to long processes (25). Considering that failure in metal-free ceramic systems is generally at the level of the veneering ceramics, veneering ceramics should be given importance in newly developed systems (26).

Fracture toughness tests determine the sensitivity of a material to crack propagation (27). Due to difficulties in the production of specimens with specific shapes, indentation tests have been developed (28) where a diamond pyramid is indented into the surface. The load, Poisson ratio, modulus of elasticity, and dimensions of the indentation and cracks allow the calculation of fracture toughness with an empirical equation. This test method is suitable only for highly homogeneous materials without large secondary phases (29, 30).

Taşkonak et al. (31) reported that the source of the fracture begins in the veneering ceramic and proceeds immediately after the crack initiating fracture reaches the stress of failure. Although the substructure ceramic is harder than the veneering ceramic, the crack does not deviate or become trapped in the plane when the crack occurs in the coating ceramic. They stated that infrastructure ceramic does not contribute to crack propagation.

In a study (32), 0.9% by weight graphene and 2.7% by weight boron nitrate nano-tubes were added to ceramics; compressive strength and fracture toughness were increased by 207% and 33%, respectively. The combination of 1-dimensional boron nitrate nanotubes and 2-dimensional graphene in this study provides important concepts for investigating the application potential of graphene-reinforced ceramic composites. Coating fibers with hexagonal boron nitride (h-BN) by CVD has been reported to increase the bond strength between fibers and ceramic matrix composites (33).

Although the mechanical properties of bioactive ceramics are typically determined according to their crystal structure, some have evaluated the crack propagation behavior and fracture mechanism of ceramics. They reported that improving the mechanical properties of the ceramic is possible to a certain extent by controlling the conditions of crack propagation (34). Furthermore, in order to increase the fracture resistance of bioactive ceramics and to improve their mechanical performances, the resistance of ceramics to crack propagation and thus the fracture energy should be increased. Crack defects on the surface of and within the ceramic should be reduced, leading to reduction of stress concentration at the crack end (35). The addition of graphene and boron nitrate nanotubes to ceramics increases their fracture strength and fracture toughness (36).

In the last few years, low-dimensional nanomaterials (LDNMs), including carbon nanotubes, graphene, and boron nitride nanotubes, have attracted attention because of their biocompatibility and excellent mechanical properties. Because of these characteristics, LDNMs have become potential nanofillers for strengthening bioactive ceramics (37). However, until now, maintaining the structural stability of LDNMs during the manufacturing of ceramic composites has been challenging because of the length of processing. Studies of dental ceramics using the graphene doping method are lacking. In the present study, we used a doping approach to improve the surface hardness of the veneering porcelain on representative ceramic frameworks. The doping approach is a noninvasive method of surface modification for improving the surface hardness of silicate-based veneering ceramics. Hardness is a valuable property affecting the polishability, occlusal wear resistance, and laboratory processing of the ceramic material (38). If the hardness of the veneering ceramic can be increased by strengthening procedures, its fracture resistance can be increased, thus reducing material failures (39). In the present study, we attempted to increase the superficial hardness by coating a thin layer on the veneering ceramic, because materials with high hardness exhibit high abrasion resistance and are difficult to polish. Graphene, with a crystal structure as a result of the coating, may increase the hardness of porcelain more than graphene with an amorphous structure. Therefore, in the present study, the temperature was not greatly increased while the doping was applied. An increased coating temperature may affect the crystal structure of graphene, a topic for future study.

Limitations of this study include the limited number of test methods and veneer ceramic materials used to determine the mechanical properties. Moreover, the development of new strategies to ensure the structural stability of LDNMs should be pursued. Maintaining the structural stability of graphene during the production of ceramic composites was difficult because of the length of the process (35, 40, 41). In order for graphene doping application not to negatively affect the color of the ceramic, the influence of oxidation temperature on microstructure and electromagnetic interference shielding performance should be investigated. (42) As a result, the use of LDNMs in ceramics is at an early stage, and more research into this promising research area is needed.

Conclusion

According to the results of this study, the application of graphene doping may change the surface hardness of dental ceramics depending on the content of the ceramic. Similarly, depending on the content of the ceramic, it may affect its color to varying degrees. The graphene doping on the ceramic surface may increase surface hardness in group E but may be less clinically useful as the color change is excessive. Nevertheless, it may be useful in the palatal region. Since the oral environment is a combination of complex situations that cannot be imitated by in vitro laboratory test methods, advanced in vivo methods should be developed to achieve closer clinical results.

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