Effect of Fe2O3 and CeO2 on Transmittance of Y2O3 Stabilized Tetragonal Zirconia Polycrystals

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Abstract

The present study is to investigate the effect of metal oxides Fe_2O_3 and CeO_2 on the structural and optical characteristics of Y_2O_3 stabilized tetragonal zirconia ceramic (Y-TZP). Different concentrations of Fe_2O_3 and CeO_2 are added into Y-TZP to fabricate the colored Y-TZP. The chromaticity and transmittance were measured by spectrophotometer. The effect of each colorant on color and transmittance of Y-TZP is quantitatively studied. Most of the previous related studies used indirect methods for measurement, but in order to get more accurate results, we chose the direct measurement method. It is found that the addition of Fe_2O_3 and CeO_2 not only changes the color of Y-TZP, but also reduces the light transmittance. The SEM characterization and XRD analysis are then performed to check the microstructure and crystal structure respectively, by which we try to understand the microscopic principle. Our findings can improve the understanding of coloration of Y-TZP by mental oxides, and support a quantitative assessment approach for aesthetic performance of dental restoration material.

1 Introduction

Ceramic, a commonly used prosthetic material in stomatology, has very good biocompatibility, excellent corrosion resistance and wear resistance [1]. It has been currently recognized as the most realistic dental restoration material due to the prominent aesthetic performance [2]. Compared with the traditional metal-ceramic restoration materials, all-ceramic restoration materials have no metal base, allowing light to pass through it that creates a layered feel more similar to natural teeth. This largely improves the aesthetic effect [3, 4] so that the all-ceramic restorations are widely favored by both patients and doctors [5]. With the continuous development of dental computer-aided designing and manufacturing technology (CAD/CAM), Y2O3 stabilized tetragonal zirconia polycrystals (Y-TZP) has been widely used as a ceramic material in clinical practice [6]. Thanks to the distinct advantages like similar mechanical properties to metals and good biocompatibility, Y-TZP has rapidly developed to be an ideal dental restoration material in clinical [7].

Nowadays, the commercial Y-TZP ceramic materials are mostly white with a single color, which is difficult to meet the aesthetic requirements of clinical repair [8]. The surface of Y-TZP often needs to be treated by shading in clinical practice. However, this kind of treatment always reduces the translucency of the material, diminishing the aesthetic effect of the restoration. From the view of material, one strategy is fabricating the Y-TZP itself with the color close to natural teeth. In this sense, the laborious shading procedure can be avoided and the thickness of facing porcelain can be reduced as well.

In recent years, with the development of dental ceramics, different nano Y-TZP powder preparation technology, processing molding and sintering process has improved the aesthetic properties of Y-TZP ceramics. At the same time, more and more scholars begin to pay attention to this problem [9, 10]. As an alternative strategy, coloring Y-TZP ceramics may be a better way to improve its aesthetic effect. Once the Y-TZP material is colored, the thickness of the veneering ceramic could be greatly reduced. The color can be adjusted through a small amount of veneering ceramic, which allows the clinician to reduce the amount of tooth preparation and maximize the preservation of the patient's teeth [11]. In general, there are two coloring methods. One is the external coloring method. The base of Y-TZP ceramic pre-sintered body is first soaked using coloring liquid, such as Lava and Vita system [12]. The colored body is then molded by dense sintering. However, the operation time and coloring method of this technique have great influence on the final color of Y-TZP ceramic [13]. The color allocation and clinical operation are both difficult for technicians, and the clinical ideal color effect cannot be always obtained [13]. The other is the overall internal coloring method, which color the Y-TZP ceramic by directly adding colorant to Y-TZP powder before sintering. The common colorants are metal oxides, such as Er2O3, Fe2O3 and CeO2 [14]. Yoshida et al. has found that the addition of Er2O3 and Fe2O3 can color the zirconia ivory while the addition of CeO2 makes the zirconia yellow [12]. Generally, the overall internal method is better than the external method because it can support more uniform and stable coloring of Y-TZP ceramics.

However, coloring the Y-TZP ceramics to gain a fantastic aesthetic effect of teeth is still changing. After adding the colorant, the color and transmittance of Y-TZP ceramics simultaneously change. Moreover, both the kind and proportion of the colorant influence the coloration [15]. Therefore, this study focuses on selecting two metal oxide colorants (Fe2O3 and CeO2) reported in the literature that have obvious coloring effects on Y-TZP and have less impact on mechanical properties [14]. To explore the changes in the color ratio and transmittance of Y-TZP ceramics after coloring with a single metal oxide through the direct measurement method, in order to obtain a more clinically ideal Y-TZP internal coloring scheme.

2 Experimental

2.1 Specimen preparation

The Y-TZP power (OZ-3Y) was purchased from Guangdong Orient Zirconia Industry Science and Technology Corporation in China. The two mental oxides, i.e., Fe_2O_3 and CeO_2, were purchased from Beijing Chemical Reagent Company in China. The Fe_2O_3 and CeO_2 were first added to Y-TZP to make a mixed powder. The concentrations of Fe_2O_3 and CeO_2 were 0.03 wt.%, 0.06 wt.%, 0.09 wt.%, 0.12 wt.% 0.15 wt.% and 1.0 wt.%, 1.5 wt.%, 2.0 wt.%, 2.5 wt.%, 3.0 wt.%, 3.5 wt.%, 4.0 wt.%, respectively. The mixture was molded under cold isostatic pressure of 200 MPa and sintered at 1500 °C for 2 hours. The sintered body was then fabricated into specimen with a size of 10 mm × 10 mm × 1 mm and the error in thickness was less than 0.02 mm. There were 3 specimens in each case.

2.2 Color measurement

The color was characterized using the 1976-L*a*b* chromaticity system recommended by the International Commission on Illumination (CIE), where L* is the lightness index, a* the green-red and b* the blue-yellow are the chromaticity coordinates, respectively. The values of L*, a*, b* for each sample were measured by the spectrophotometer (CS-5, datacolor, USA). The measured results were based on D65 illuminant and a 2-degree standard observer. Each specimen was measured five times on each side and the average value was taken finally.

2.3 Transmittance measurement

The transmittance was measured by the spectrophotometer mentioned above in the dark room, as demonstrated in Fig. 1. Both the uncolored and colored Y-TZP was measured. All the specimens were measured in the visible light range of 400–700 nm. The two sides of each test piece were measured three times and the average value was taken. The formula for calculating the transmittance *T* is [20]:

$$T = \frac{I}{I_0} \times 100\% \tag{1}$$

where I and I_0 are the intensity of light passing through the specimen, and the intensity of the light source, respectively.

2.4 Structure characterization

The unstained Y-TZP, 0.15 wt% Fe_2O_3 colored Y-TZP and 4 wt% CeO_2 colored Y-TZP fracture samples were sprayed with gold and observed under a field emission scanning electron microscope (Hitachi S-4500, Japan). The crystal structure of the unstained Y-TZP, 0.15 wt% Fe_2O_3 colored Y-TZP and 4 wt% CeO_2 colored Y-TZP were characterized by using X-ray diffractometer (XRD) (D/max-RB, Japanese Science, Japan), use copper target Kα line, wide-angle continuous scanning, 2θ from 10° to 90°, step length 0.02°, 4°/min.

2.5 Statistical analysis

SPSS18.0 statistical analysis software was used to perform One-way ANOVA and LSD test on the obtained integral transmittance of visible light. Any difference could be observed in the transmittance of Y-TZP ceramics with different mass percentages of colorants. The correlation analysis test with test level $\alpha = 0.05$ was also carried out.

3 Results And Discussion

3.1 Effect of colorants on color of Y-TZP

The results of changes in lightness and chromaticity of the colored Y-TZP are shown in Fig. 2&3. In Fig. 2, one can learn that the lightness value gradually decreases as the concentration of Fe_2O_3 increases. When the concentration of Fe_2O_3 exceeds 0.06 wt%, the values of a* and b* increase significantly. It indicates that Fe_2O_3 have greater influence on the b* value than the a* value. In Fig. 3, we also observe that the a* value of colored Y-TZP decreases as the concentration of CeO_2 increases. However, the amplitude is smaller than that of Fe_2O_3 colored case and the coloring effect is weaker. The value of b* increases slightly with the increase of CeO_2 concentration, and the overall wavier range is relatively small. The lightness value fluctuates with the change of CeO_2 concentration, indicating that the change of CeO_2 concentration has little effect of CeO_2 on the lightness of colored Y-TZP.

The colorant Fe_2O_3 used in this study can make Y-TZP shift to red color, and CeO_2 can make the color of Y-TZP get yellow. At the same time, the ultraviolet can be strongly absorbed by Fe^{3+} [17]. When Y-TZP was added with Fe_2O_3, the value of chromaticity increases and the lightness decreases. Simultaneously, the color may change to red direction. The result indicates that the less Fe_2O_3, the higher brightness value. The brightness value of 0.03 wt% Fe_2O_3 colored Y-TZP is 84, while the brightness value of 0.15 wt% Fe_2O_3 colored Y-TZP ceramics is only 75. The lightness value decreases and the hue value gradually increases with the increase of Fe_2O_3 concentration. Fe_2O_3 has an effect on the a^* value of Y-TZP, but has a larger effect on the b^* value. As can be seen, Fe_2O_3 has a strong coloring ability to Y-TZP, and gives Y-TZP a red color, but reduces its lightness value. Cerium has two states: Ce^{3+} and Ce^{l+} in ceramics. Ultraviolet rays are strongly absorbed by cerium ions. Under certain conditions, the ultraviolet absorption band of cerium ions can enter the visible light region and produce light yellow. The results of this experiment show that the coloring ability of CeO_2 to Y-TZP is weak. As the mass percentage of CeO_2 increases, the a^* value of colored Y-TZP changes little, between − 4 and − 5. The b^* value can get higher with the increase of CeO_2, but the amplitude is small. It shows that CeO_2 plays a certain role in coloring Y-TZP. In summary, in this experiment, different concentrations of Fe_2O_3 and CeO_2 were added to Y-TZP to prepare colored Y-TZP ceramic materials with different colors. The color space of L^*, a^* and b^* is 75.27 ~ 87.00, -5.14 ~ 3.55 and 10.80 ~ 23.87 respectively. Compared with the Vita-3D Master color palette, the brightness range is higher than the color palette. After adding the decorative porcelain, it can achieve a more ideal lightness space, which is suitable for matching with the decorative porcelain to meet the clinical color matching requirements.

3.2 Effect of colorants on transmittance of Y-TZP

The results of the relationship between the transmittance of Y-TZP and the wavelength are presented in Fig. 4&5. As the wavelength increases, the transmittance also increases. The transmittance curve of uncolored Y-TZP is almost a straight line, and the visible light transmittance is significantly higher than that of colored Y-TZP. As the concentration of Fe_2O_3 and CeO_2 increases, the transmittance of Y-TZP decreases as well.

As shown in Table 1, there was a statistically significant difference between the uncolored Y-TZP, 0.03 wt% Fe_2O_3 colored Y-TZP, and 0.15 wt% Fe_2O_3 colored Y-TZP groups($P < 0.05$). In terms of visible light transmittance, uncolored Y-TZP > 0.03 wt% Fe_2O_3 colored Y-TZP > 0.15 wt% Fe_2O_3 colored Y-TZP. The above results indicate that the change in the content of Fe_2O_3 colorant has a significant effect on the visible light transmittance of Y-TZP ceramics.

Table 1
Effect of Fe_2O_3 on transmittance

Wavelength	Uncolored Y-TZP(%)	0.03 wt%Fe_2O_3 colored Y-TZP(%)	0.15 wt%Fe_2O_3 colored Y-TZP(%)	F	*P*
400–700 nm	20.56 ± 2.39	14.5 ± 1.44	11.84 ± 0.39	46.265	<0.05

As shown in Table 2, there was a statistically significant difference between the uncolored Y-TZP, 1 wt% CeO_2 colored Y-TZP, and 4 wt% CeO_2 colored Y-TZP groups($P < 0.05$). In terms of visible light transmittance, uncolored Y-TZP > 1 wt% CeO_2 colored Y-TZP > 4 wt% CeO_2 colored Y-TZP. The results above indicate that the change in the content of CeO_2 colorant has a significant effect on the visible light transmittance of Y-TZP ceramics.

Table 2
Effect of CeO_2 on transmittance

Wavelength	Uncolored Y-TZP(%)	1 wt% CeO_2 colored Y-TZP(%)	4 wt% CeO_2 colored Y-TZP(%)	F	*P*
400–700 nm	20.56 ± 2.39	13.09 ± 0.32	11.83 ± 0.22	335.21	<0.05

It can be seen from Table 3 and Fig. 6 that the single-factor analysis of variance for the mean visible light transmittance between the four groups showed statistical differences between the groups($P < 0.05$). The results of the multiple comparison of 0.15 wt% Fe_2O_3 colored Y-TZP group, 0.03 wt% Fe_2O_3, 1 wt% CeO_2 colored Y-TZP group and 4 wt% CeO_2 colored Y-TZP group show that there are statistical differences between each other.

Table 3
Effect of Fe_2O_3 and CeO_2 on transmittance

Wavelength	1 wt% CeO_2 colored Y-TZP(%)	4 wt% CeO_2 colored Y-TZP(%)	0.03 wt%Fe_2O_3colored Y-TZP(%)	0.15 wt%Fe_2O_3colored Y-TZP(%)	F	*P*
400–700 nm	13.09 ± 0.32	11.83 ± 0.22	14.5 ± 1.44	11.84 ± 0.39	41	<0.05

Transmittance is used to characterize the quality of Y-TZP translucency. In addition to color, translucency is an important optical characteristic. Natural teeth have good translucency, especially in the anterior teeth aesthetic area, which can make the color of teeth more beautiful. Therefore, it is important that the restoration has good translucency. The translucency of Y-TZP is affected not only by its own chemical characteristics, crystal arrangement, particle size, etc. [18], but also by other factors such as colorant, measurement method, ceramic thickness, and manufacturing process [19]. Common methods for measuring translucency are direct and indirect methods. The direct method is simple to operate and can be conveniently used for the measurement and comparison of tooth translucency. But the direct method cannot be used for clinical testing. However, in the invitro test, the direct method can directly obtain the transmittance of the material. The results are not expressed by the emissivity of the material. The results obtained are more reliable than the indirect method. In this experiment, we chose the direct measurement method and expected to analyze the change of Y-TZP transmittance more accurately.

In this experiment, it can be seen from Fig. 4&5 that the visible light transmittance of uncolored Y-TZP and colored Y-TZP is wavelength-dependent. In the visible light region of 400–700 nm, as the wavelength increases, the transmission increases as well. This phenomenon is similar to the conclusions of previous studies [20–23]. The results obtained in this experiment indicate that the visible light transmittance of Y-TZP increases with increasing wavelength, and is similar to other dental ceramics. As the thickness of the ceramic layer increases, its transmittance will decrease [16]. Therefore, in this experiment, the thickness of the Y-TZP test piece was selected to be 1 mm. Studies have shown that the thickness of a porcelain layer of 1 mm has a transmittance of about 40% [24, 25], so this thickness is also the conventional thickness of ceramic materials for transmission.

The colored Y-TZP can absorb visible light of a certain wavelength. Thus,its visible light transmittance is lower than that of the uncolored Y-TZP. The more the content of colorant, the more absorption of light, the smaller the transmittance of the material. In this experiment, there was a statistical difference in visible light transmittance between the colored Y-TZP and uncolored Y-TZP test groups ($P < 0.05$), indicating that Fe_2O_3 and CeO_2 have a certain effect on the translucency of Y-TZP. There was a statistically significant difference in visible light transmittance between test groups with different colorant contents ($P < 0.05$), indicating that the visible light transmittance of colored Y-TZP is related to the content of colorant, and changes in the content of colorant can cause changes in transmittance. The color of transparent materials is mainly determined by the spectral composition of light transmission, and the color of opaque materials is determined by the reflection spectrum [26–28]. The Y-TZP produced in this experiment has a certain translucency. The color of Y-TZP seen by the human eye is composed of two parts. One is that the light reflected by the Y-TZP surface enters the human eye, the other is that the light transmitted through the Y-TZP reaches its background, and the light reflected by the background passes through the test piece again, and is reflected, transmitted, and absorbed. This part of the light enters the human eye and is mixed with the front part of the light to form the color of Y-TZP, so the visible light transmittance of Y-TZP is related to its color. In this experiment, the visible light transmittance values between the colored Y-TZP groups are statistically different, but the difference is relatively small, Spyropoulou et al. [29] measured the Y-TZP of Nobel Procera Company using indirect methods, which are divided into three types: light, medium and dark. The CR values are 0.880 ± 0.007 for light color, 0.877 ± 0.011 for medium color, and 0.885 ± 0.009 for dark color. The experimental result is that the medium-color Y-TZP has the best light transmission, which may be due to the error caused by the indirect method. However the transmittance values of the three groups have little difference, which is similar to the experimental results. Thus, it is difficult to distinguish the translucency of colored Y-TZP with our eyes.

In summary, this experiment has studied the visible light transmittance of colored Y-TZP, but there are still some problems in the current research that need to be further explored. On the one hand, the thickness of Y-TZP is different under different clinical conditions. Does the change of transmittance of colored Y-TZP affect its aesthetic effect? On the other hand, the impact of the decrease in transmittance on the aesthetic effect of the patient’s oral cavity still needs further clinical trials.

3.3 Interpretation in terms of microstructure and crystal structure

The SEM micrographs of uncolored Y-TZP, 0.15 wt% Fe_2O_3 colored Y-TZP, and 4 wt% CeO_2 colored Y-TZP fully-sintered specimens are given in Fig. 7. The structure of 0.15 wt% Fe_2O_3 coloring Y-TZP and 4 wt% CeO_2 coloring Y-TZP is dense and the grain size is uniform, which guarantees its structural stability. The grain size is about 0.5 ~ 0.7 μm. The fracture modes of colored Y-TZP are transgranular fracture and intergranular fracture. The influencing factors of ceramic translucency include the nature of materials such as impurities, pores, and grain boundaries [18]. The Y-TZP powder used in this experiment has a small particle size and can shorten the diffusion path of pores when sintered. This can make the Y-TZP structure uniform and have a certain translucency. Studies have shown that the translucency of Y-TZP is related to the crystal grain size [30]. When the wavelength and grain diameter of incident light are similar, Y-TZP has the strongest light absorption. Therefore, the translucency of Y-TZP ceramics can only be improved by moving the diameter of the crystal grains away from the wavelength region of visible light [16]. In this test, the SEM photo of the colored Y-TZP shows that the sintering has basically reached densification, the grain size is uniform, the grain size does not exceed 0.5 μm, the grain size is relatively small, and the number of grain boundaries is large. Excluding providing more channels, the Y-TZP material's light loss is reduced, which is beneficial to obtain high translucency.

The XRD was used to analyze the crystal structure of uncolored Y-TZP, 0.15 wt% Fe_2O_3 colored Y-TZP and 4 wt% CeO_2 colored Y-TZP final sintered body (Fig. 8). Compared with the control group uncolored Y-TZP, the peak positions of colored Y-TZP are basically the same as tetragonal $ZrO_{2,}$ which indicates that the main phase structure of the colored Y-TZP is tetragonal zirconia. The peaks of Fe_2O_3 and CeO_2 did not appear in the XRD. The peak positions of 0.15 wt% Fe_2O_3 colored Y-TZP and 4 wt% CeO_2 colored Y-TZP at 2θ angles of 34.8° and 59.6° are higher than those of uncolored Y-TZP.

There are three crystal forms of ZrO2 including monoclinic phase, tetragonal phase and cubic phase. At room temperature, ZrO2 appears as a monoclinic phase and then turns into a tetragonal phase when heated to about 1100℃. As the temperature rises to 2370℃, it will transform into a cubic phase structure [31, 32]. In the transformation of different structures, the volume of ZrO_2 will change greatly and then cause the crack, which limits its application in high temperature fields [33]. After the stabilizer is added, the morphology and structure are more stable, which expands the application field of ZrO_2 [34, 35]. The

XRD results show that the structure of Y-TZP crystal is still stable. It means that the oxides did not change the crystal structure of Y-TZP, which proves that Y-TZP can be used as an ideal dental restoration material. The results show that Y-TZP and the colorant are well combined because there are no obvious peaks of colorant in the XRD pattern.

This experiment mainly discusses the effect of adding colorants on the optical properties of Y-TZP, but further physical properties are still needed for the changes in ceramic strength.

4 Conclusions

As the amount of Fe_2O_3 increases, the lightness value of Y-TZP drops from 84 to about 75. With the decrease of lightness, the hue values of a* and b* increase at the same time, and the increase of b* value is larger than that of a* value. With the increase of CeO_2 content, the b* of Y-TZP coloring increases, but the range is smaller, and the coloring effect is weaker. The value of a* decreases slightly with the increase of CeO_2 content, and the overall fluctuation range is not large. The change of CeO_2 content has little effect on the brightness of colored Y-TZP. Comparing the color adjustment range of Y-TZP by CeO_2 and Fe_2O_3 colorants, it is found that these two colorants can adjust the color brightness of Y-TZP similar to the commonly used Vita-3D Master color palette in clinical practice. As the content of the two colorants increases, the visible light transmittance gradually decreases. The SEM photo shows the structure of 0.15 wt% Fe_2O_3 coloring Y-TZP and 4 wt% CeO_2 coloring Y-TZP is dense and the grain size is uniform, which guarantees its structural stability, and Y-TZP and the colorant are well combined because there are no obvious peaks of colorant in the XRD pattern. The diffraction peaks of 0.15 wt% Fe_2O_3 colored Y-TZP and 4 wt% CeO_2 colored Y-TZP at 2θangles of 34.8°and 59.6°are higher than those of uncolored Y-TZP.

Declarations

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Figures

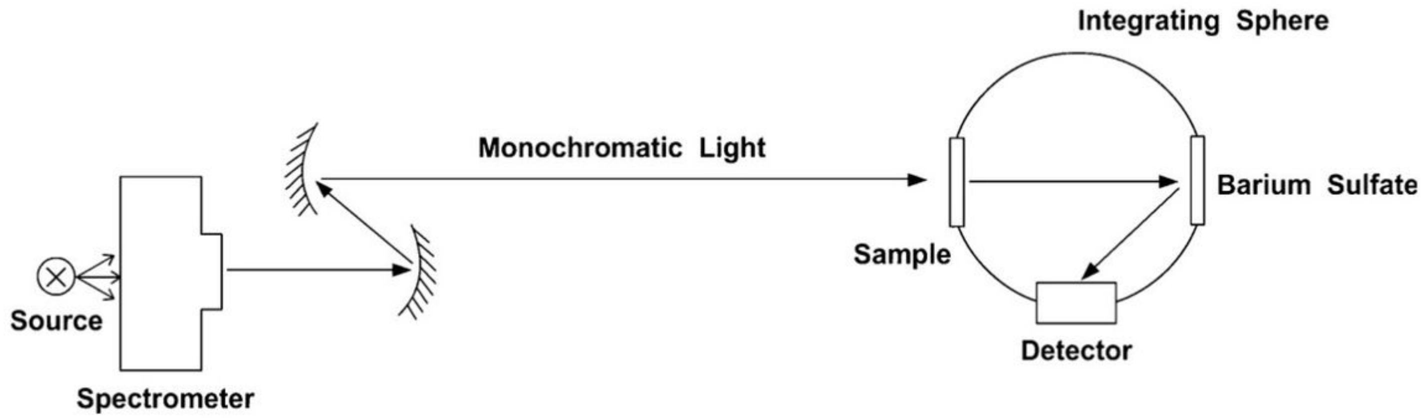
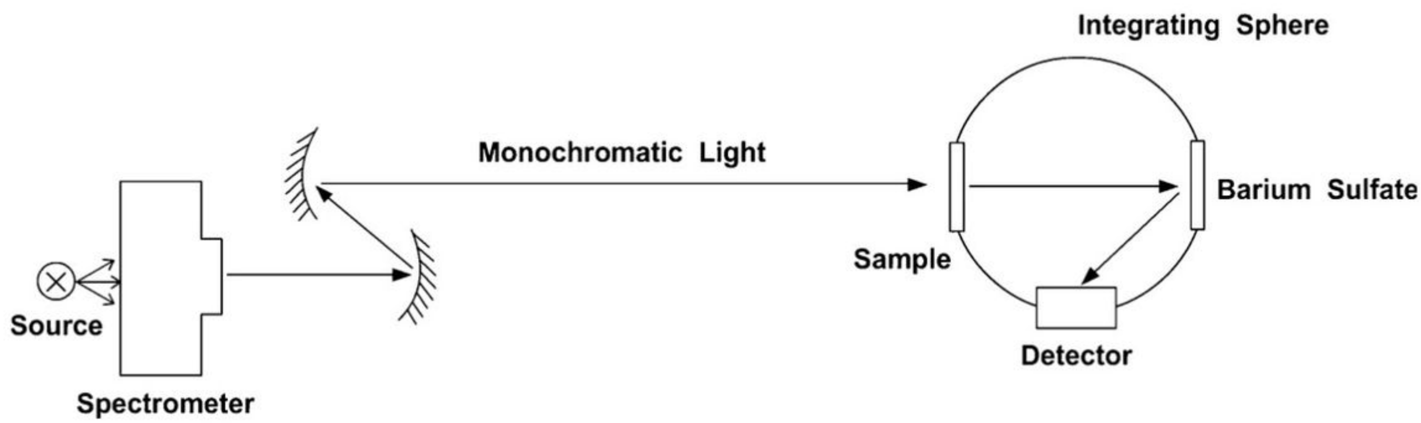


Figure 1

Schematic diagram of spectrophotometer



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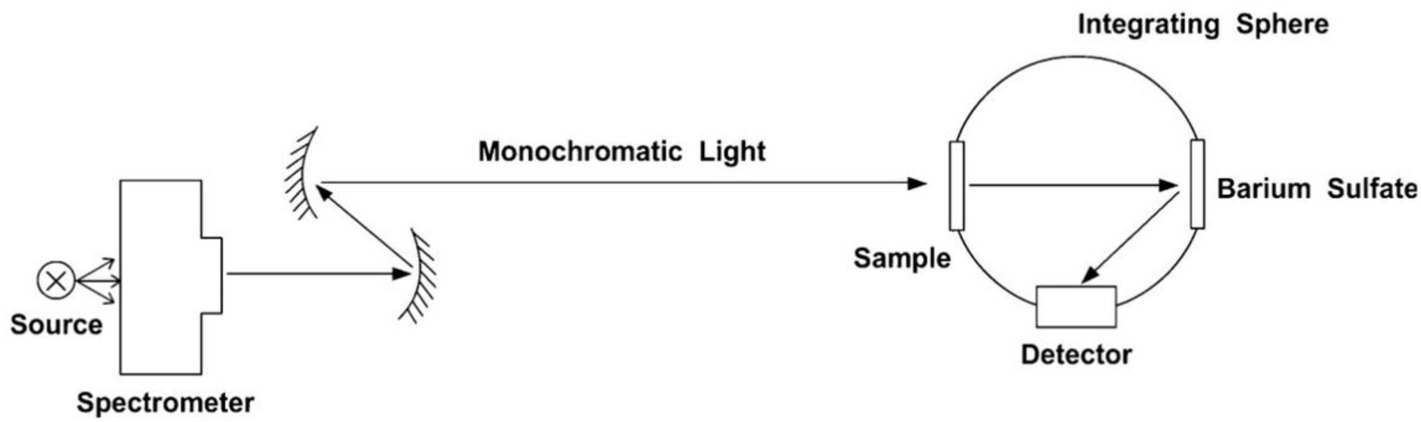

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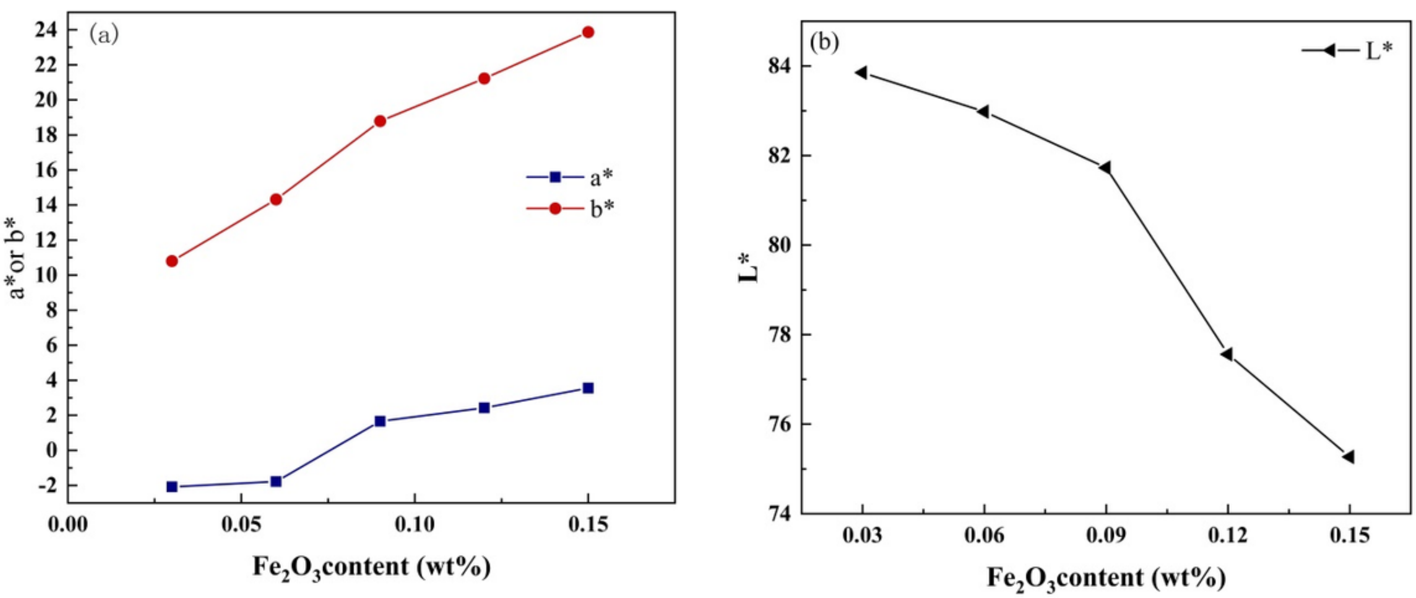
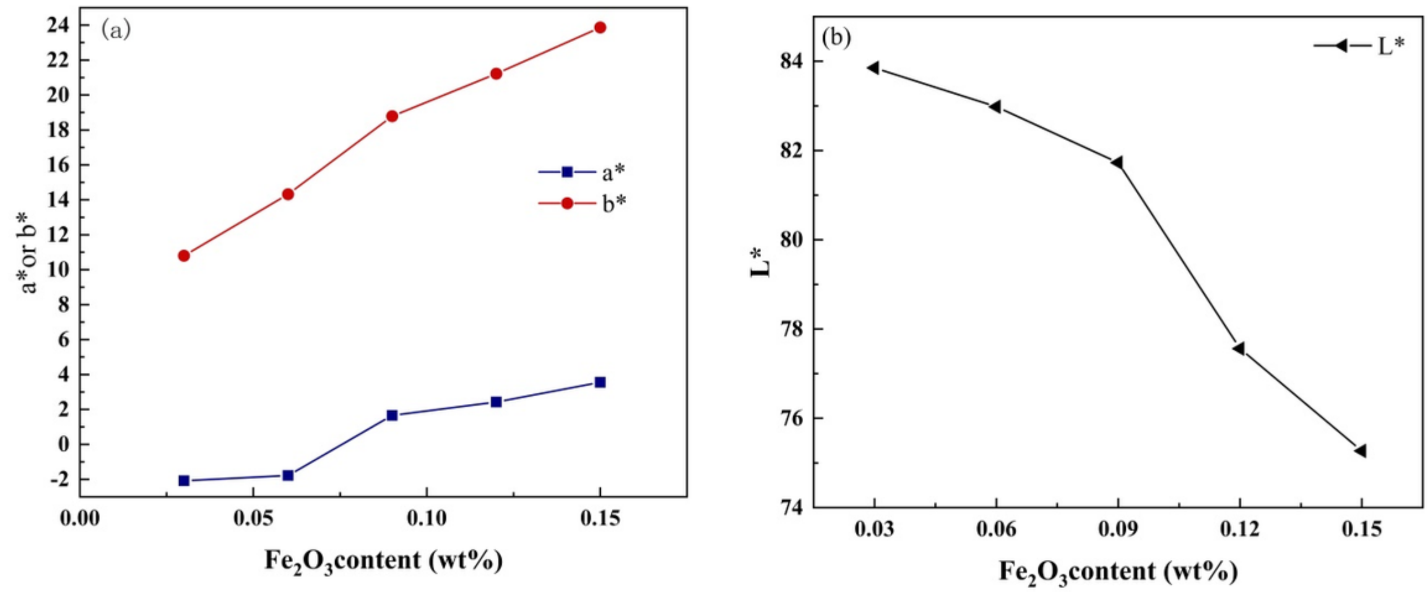


Figure 2

Effect of Fe2O3 concentration on (a) a*b* and (b) L* value of Y-TZP. 52x15mm (1200 x 1200 DPI)



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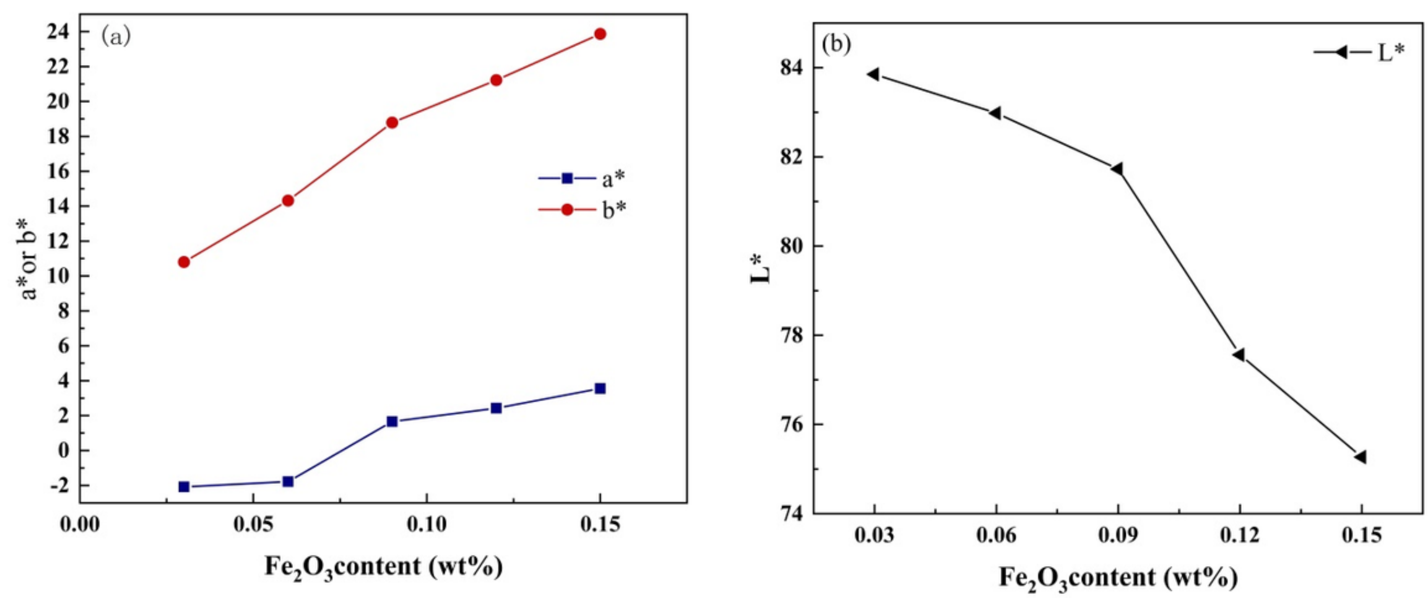

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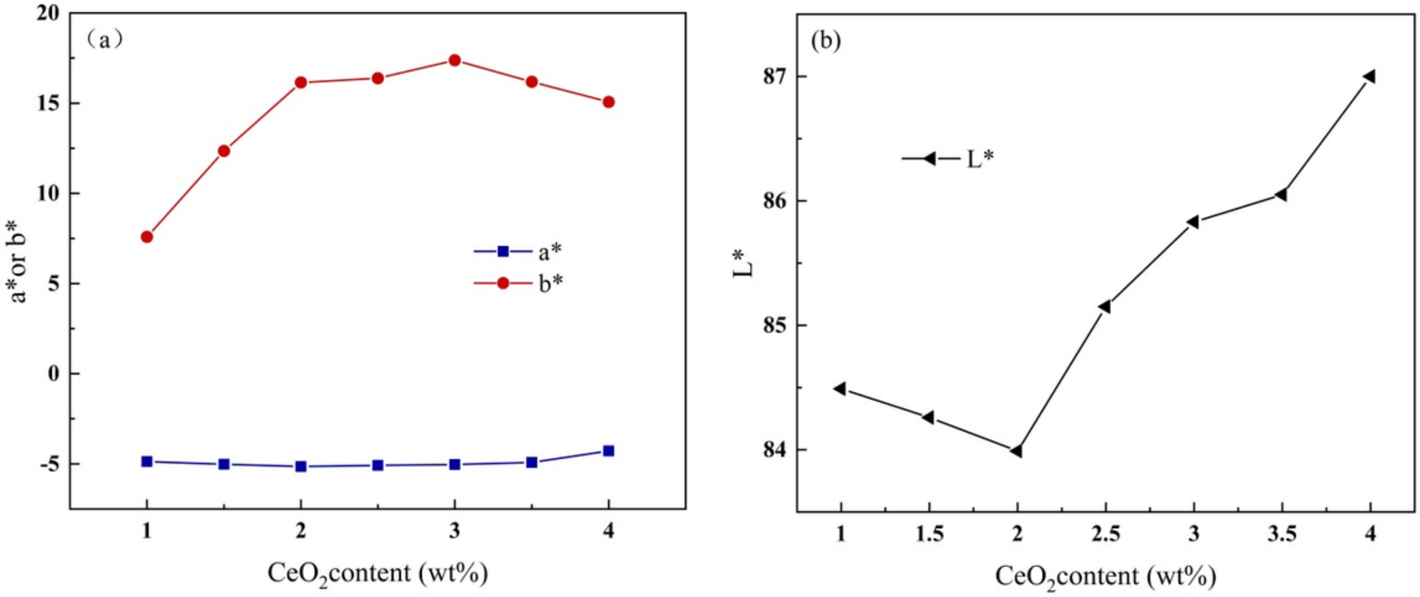
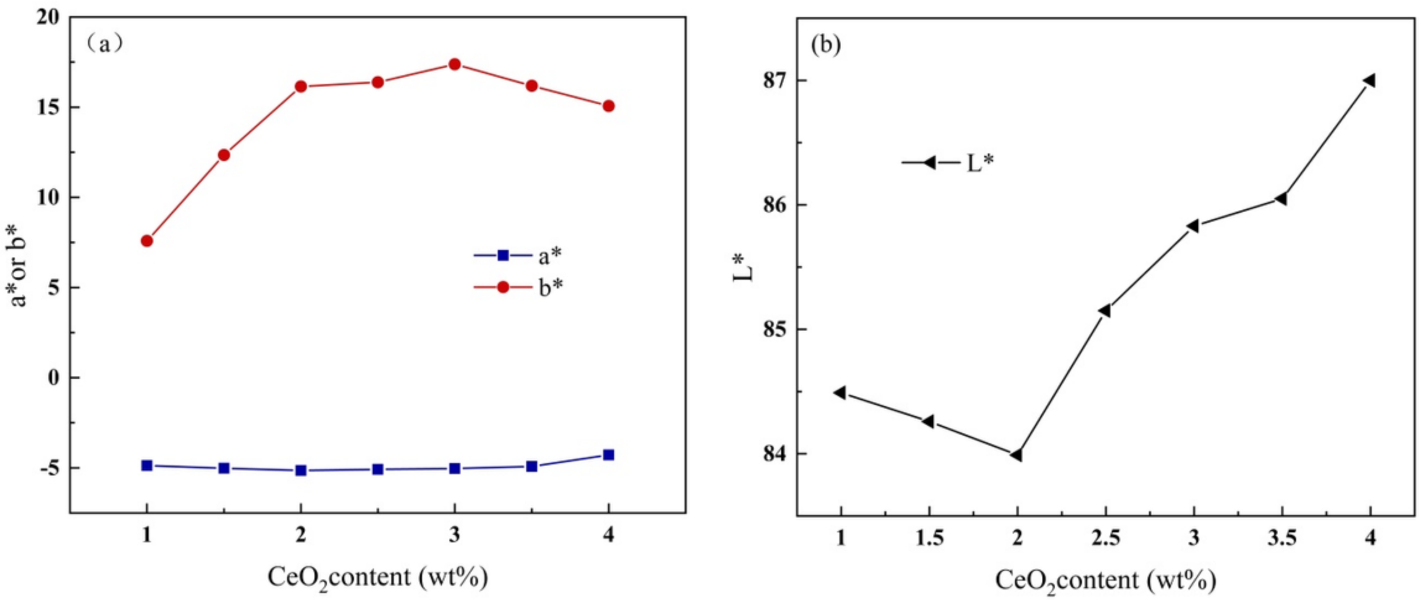


Figure 3

Effect of CeO2 concentration on (a) a*b* and (b) L* value of Y-TZP. 52x15mm (1200 x 1200 DPI)



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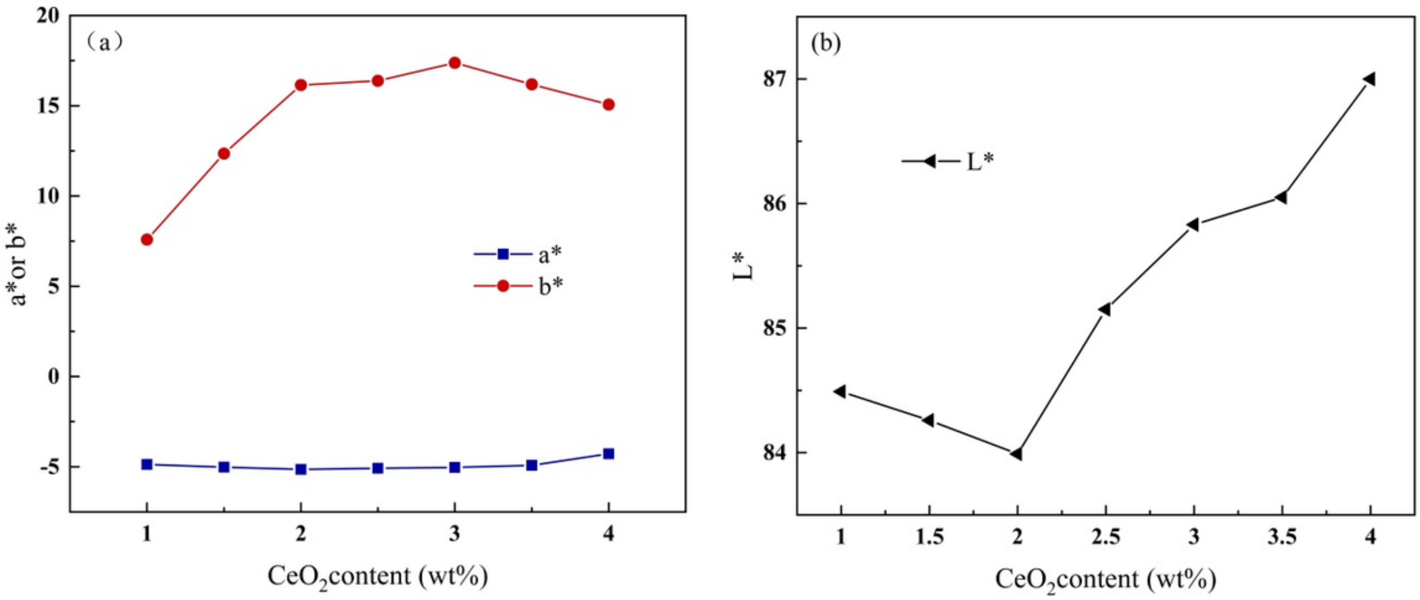

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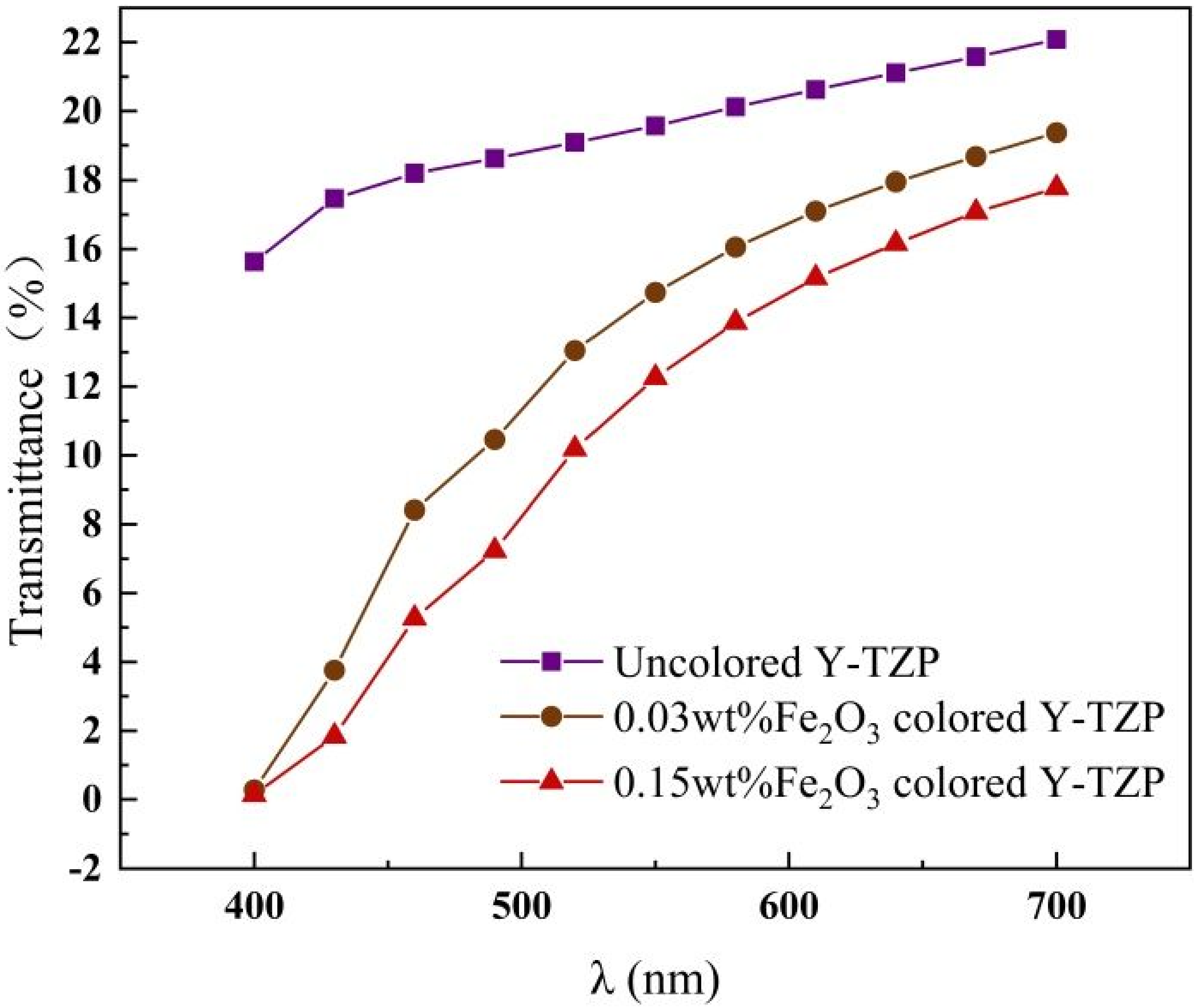
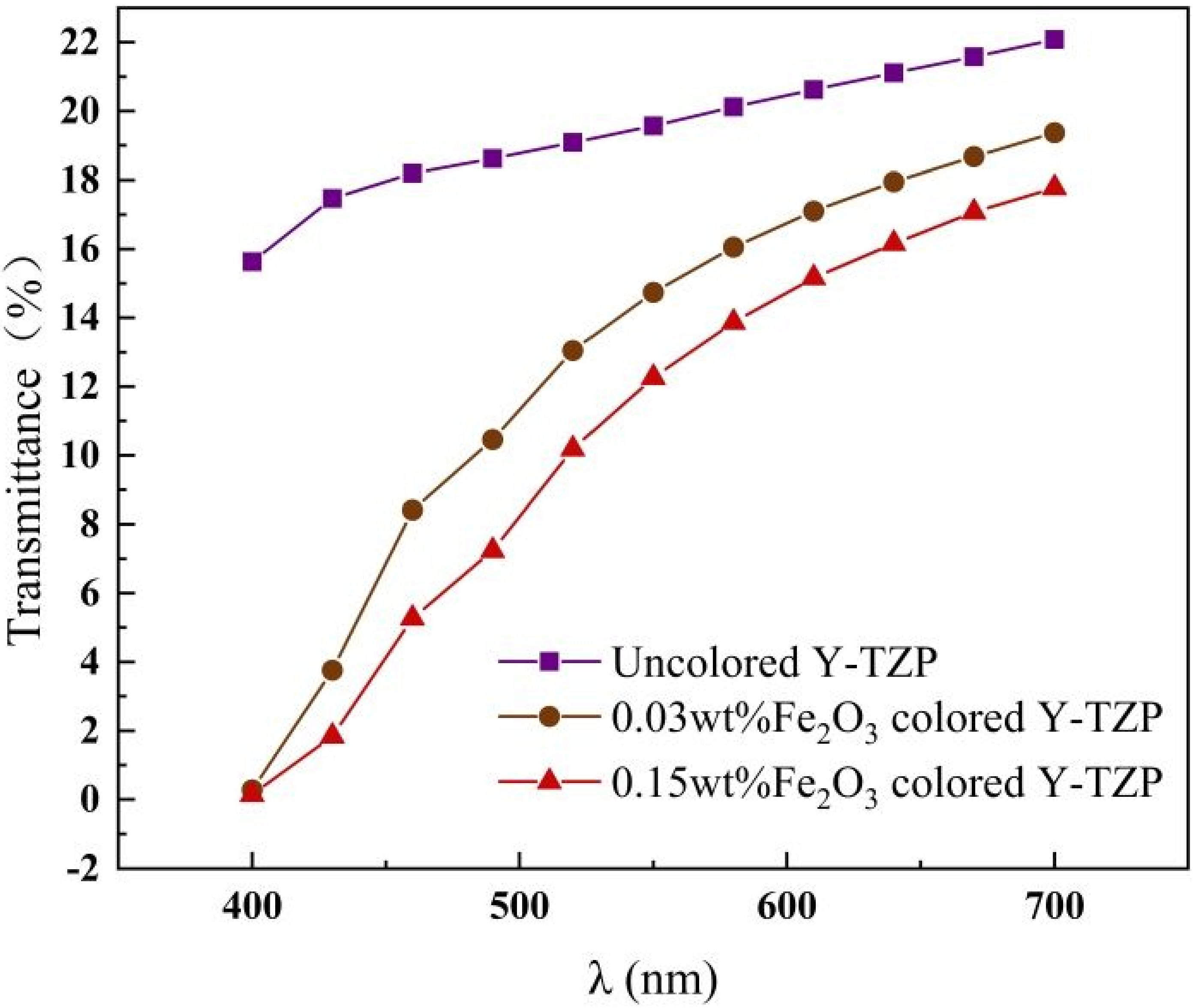


Figure 4

Transmittance of uncolored and Fe2O3 colored Y-TZP on different wavelength



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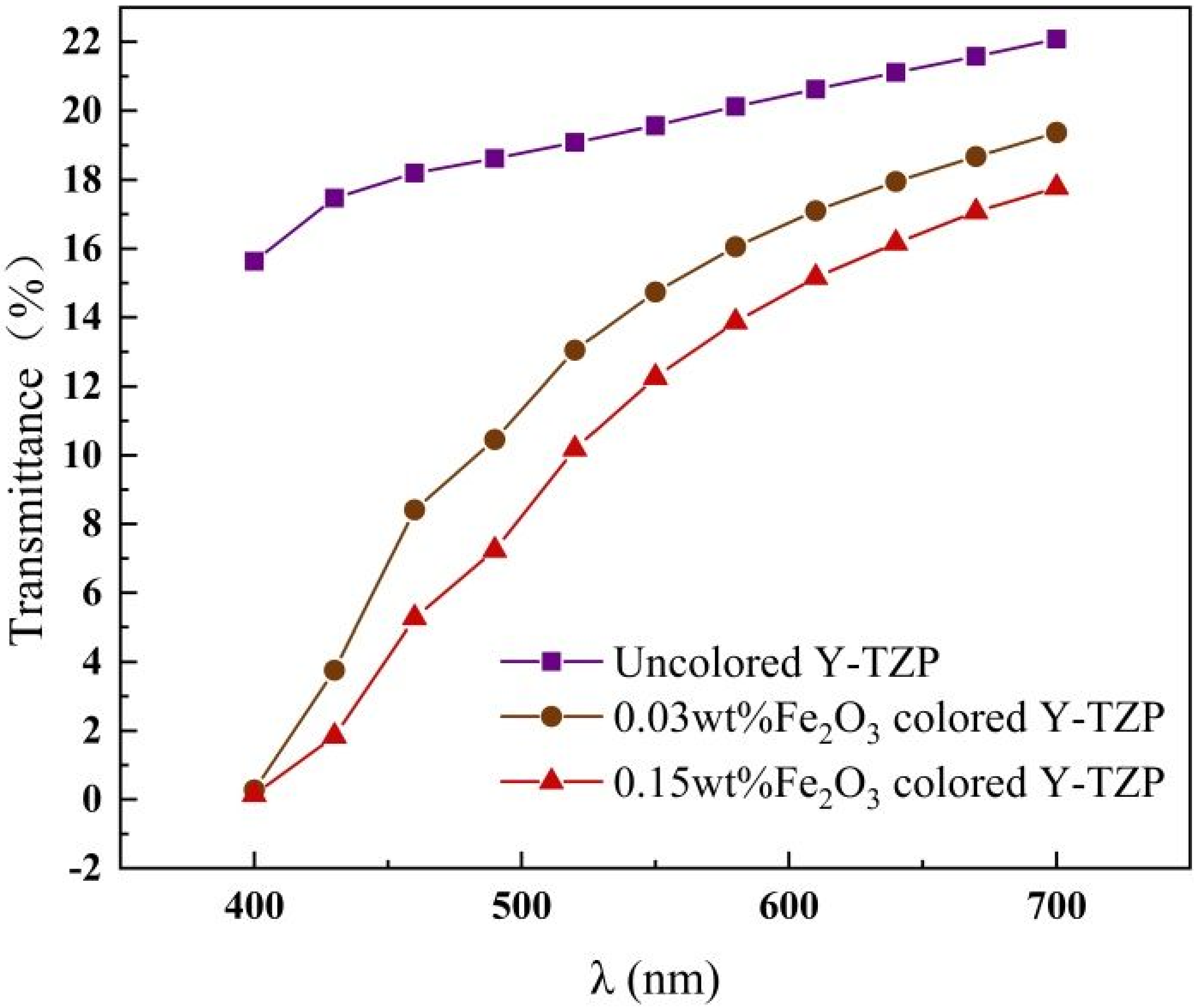

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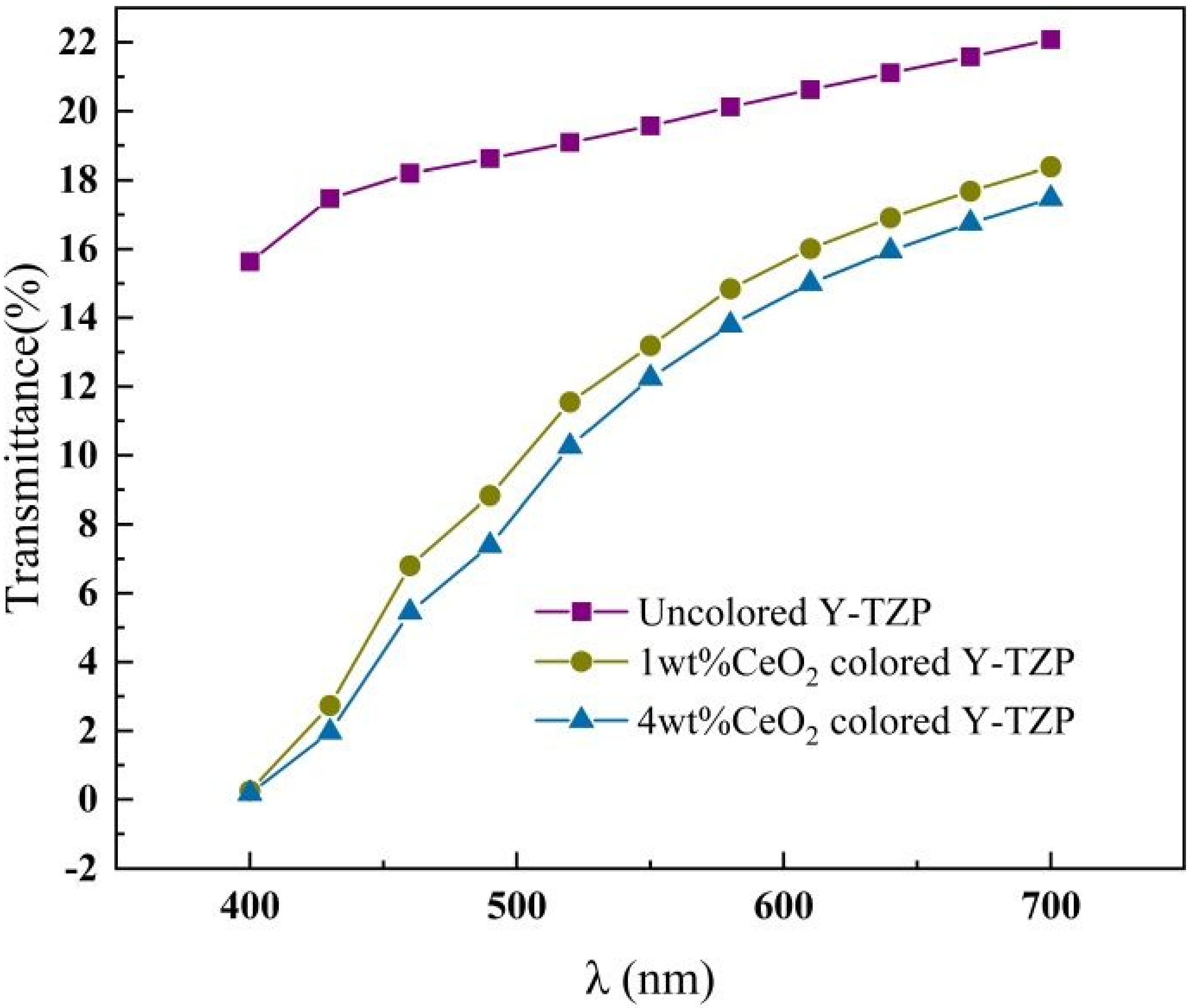

Figure 5

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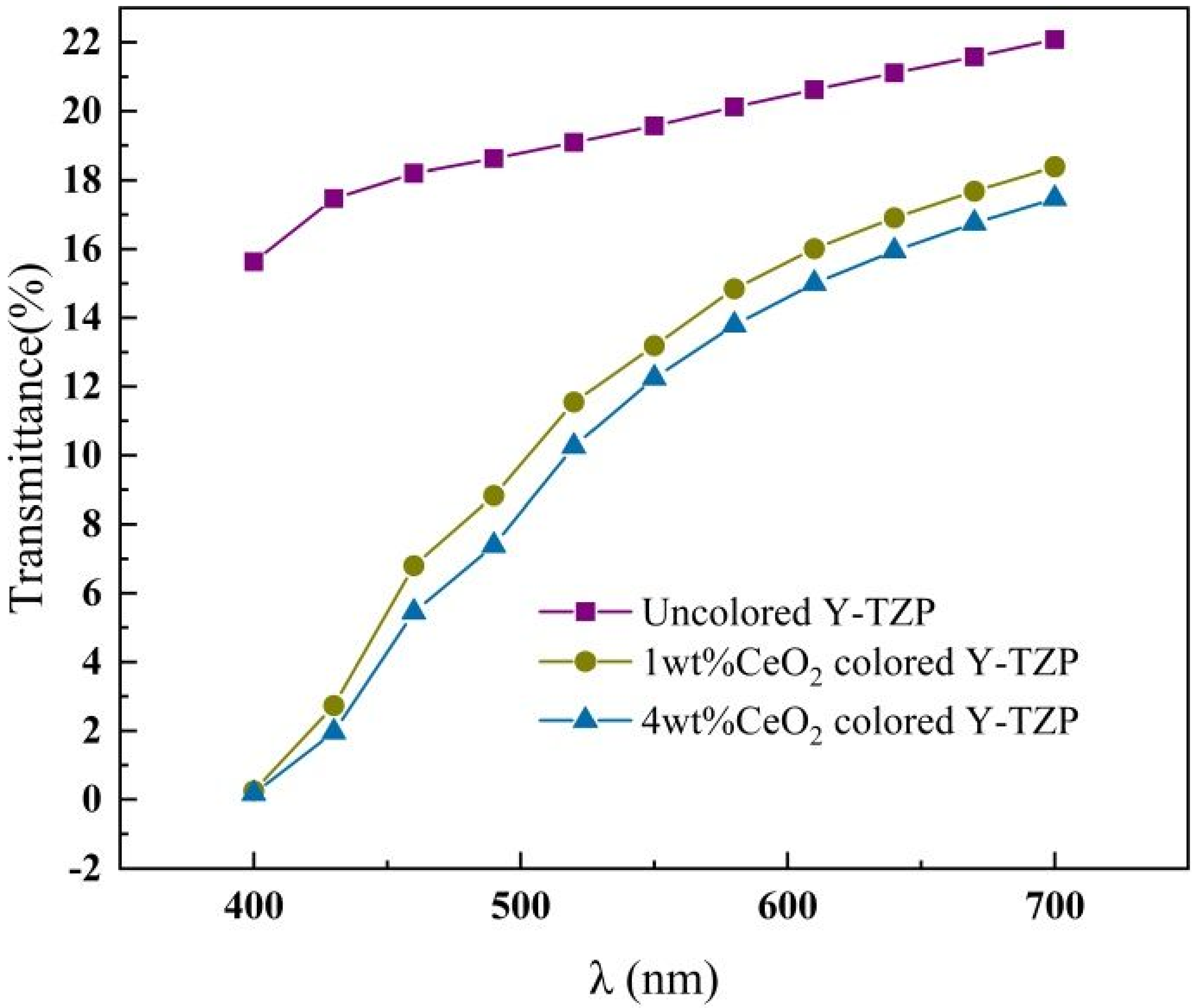

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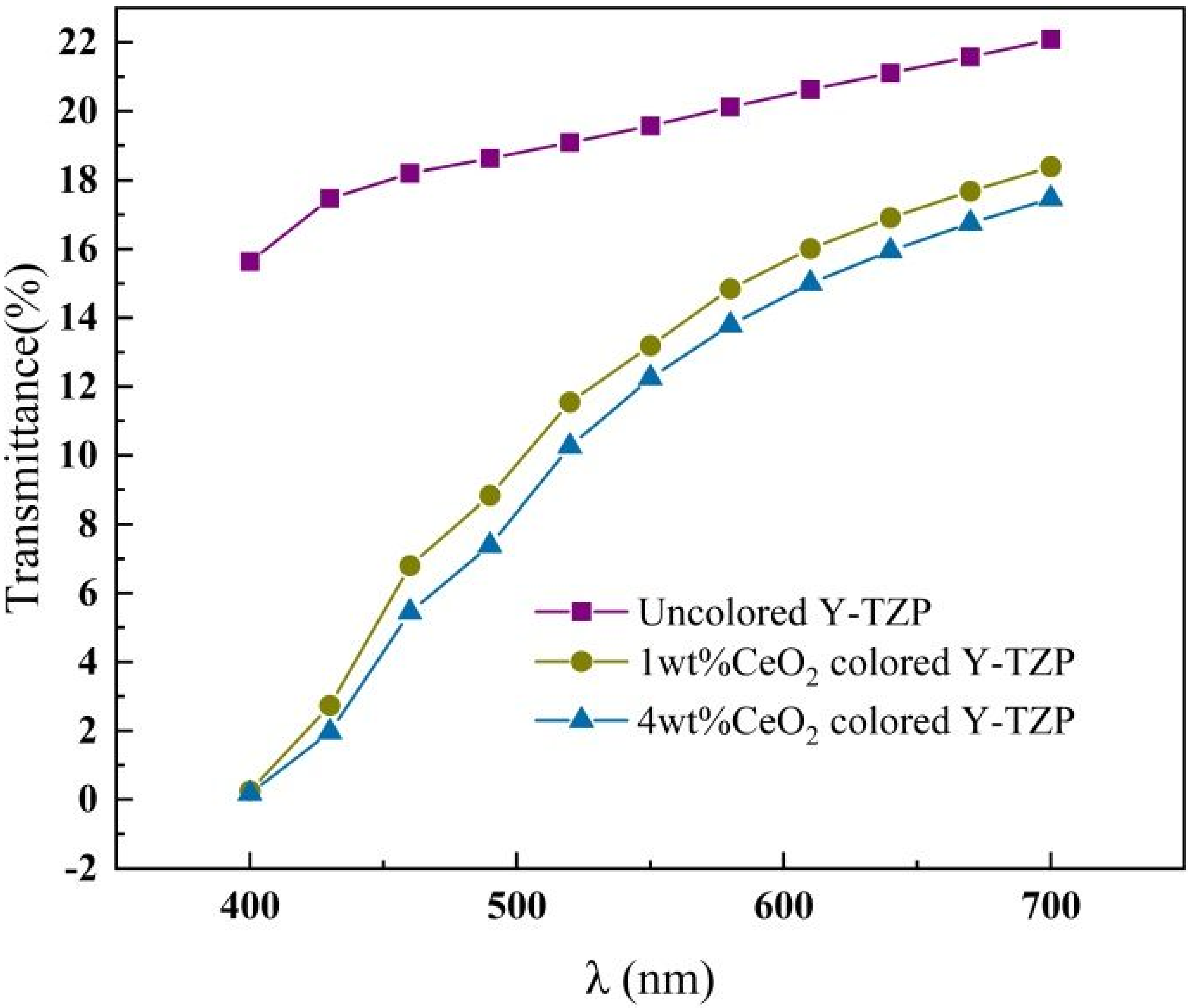

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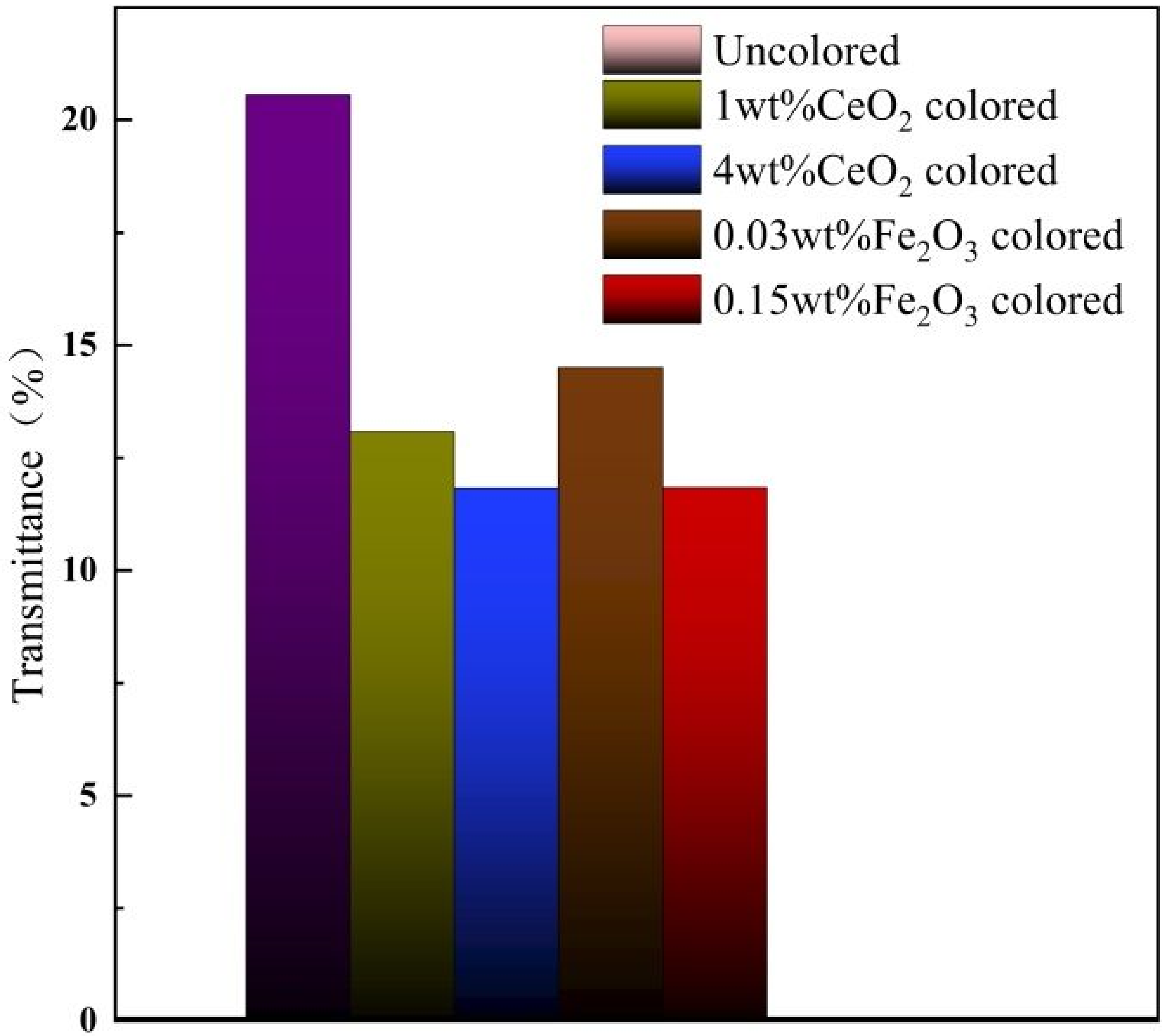
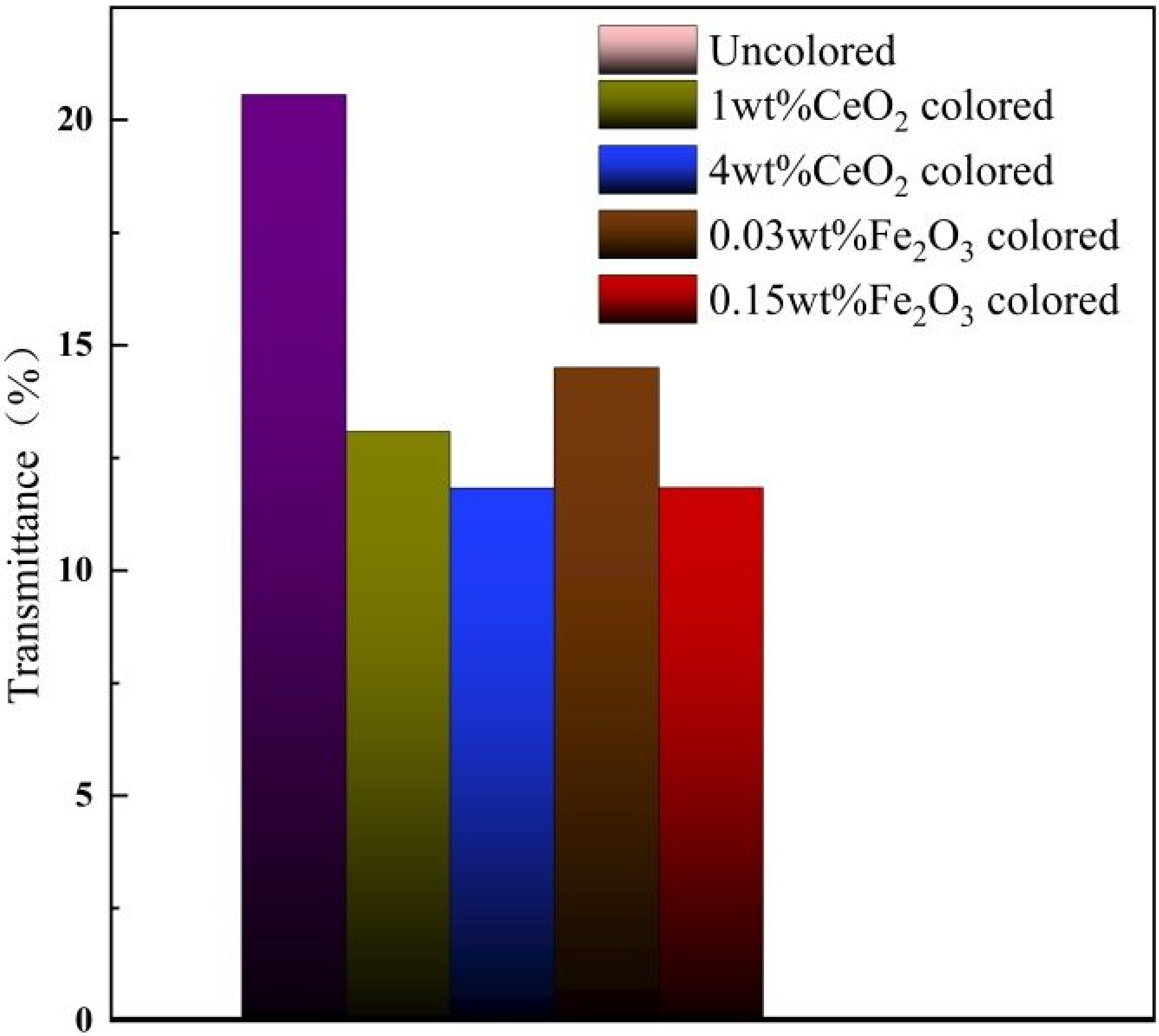


Figure 6

Transmittance of Fe2O3 and CeO2 colored Y-TZP



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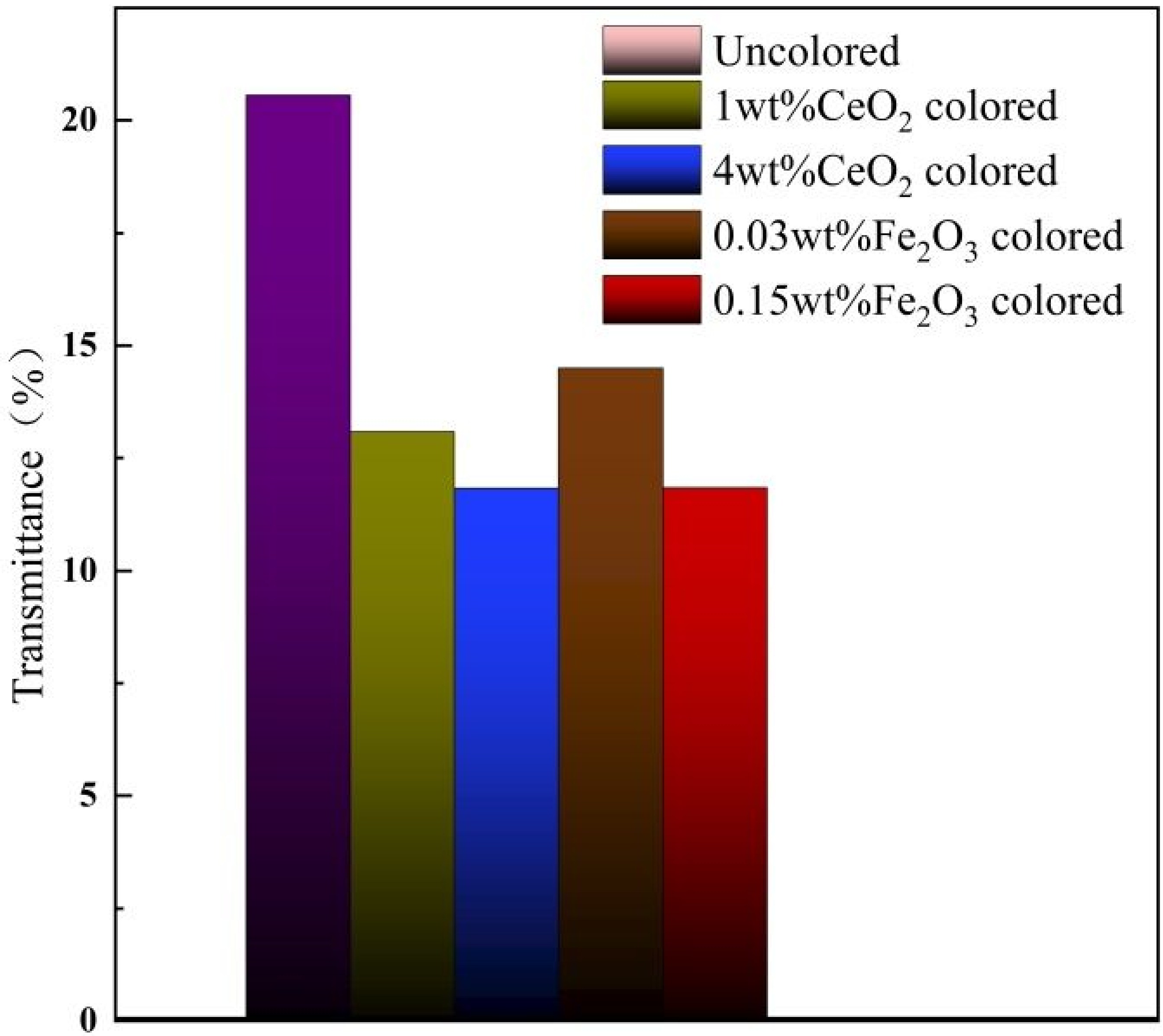

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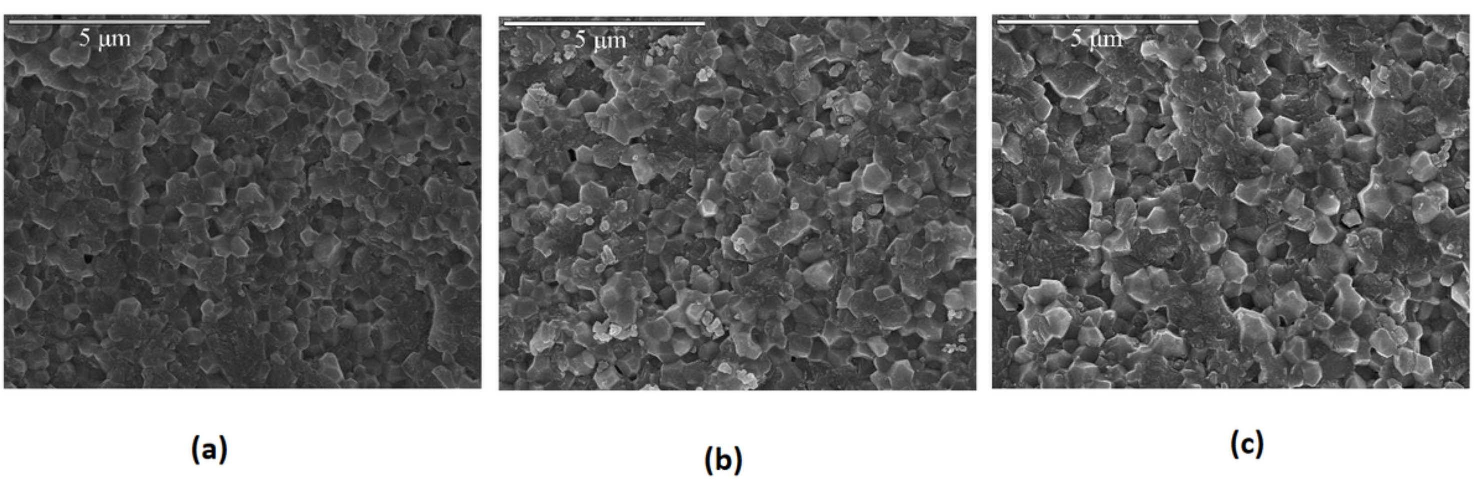
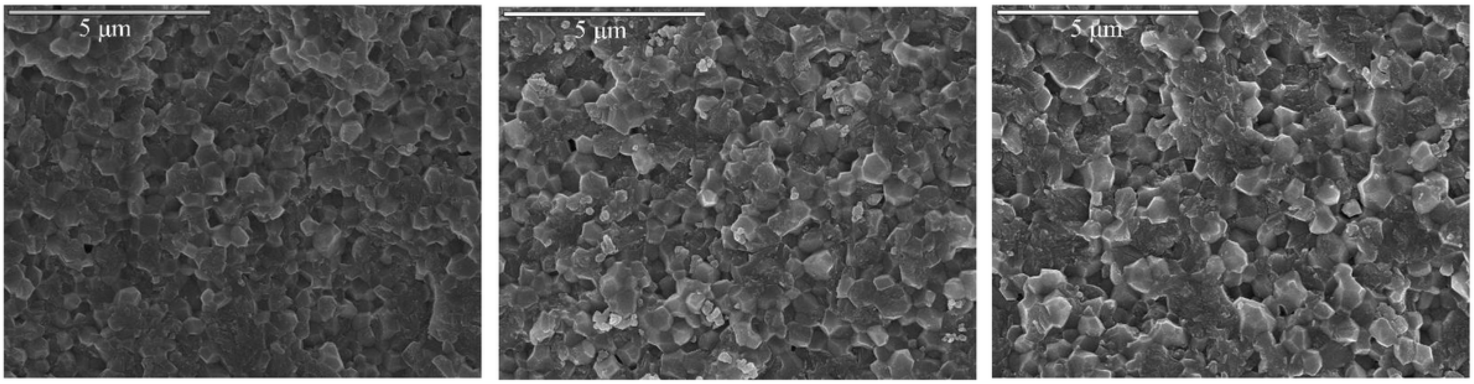


Figure 7

SEM micrographs of uncolored Y-TZP, 0.15wt%Fe2O3 colored Y-TZP and 4wt%CeO2 colored Y-TZP specimens (×10000) 177x121mm (300 x 300 DPI)



(a) (b) (c)

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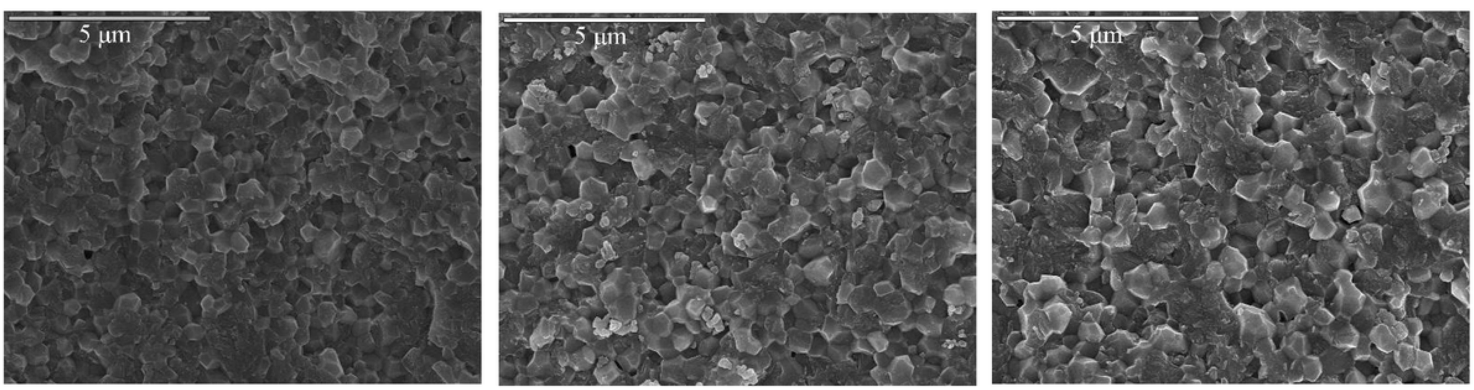

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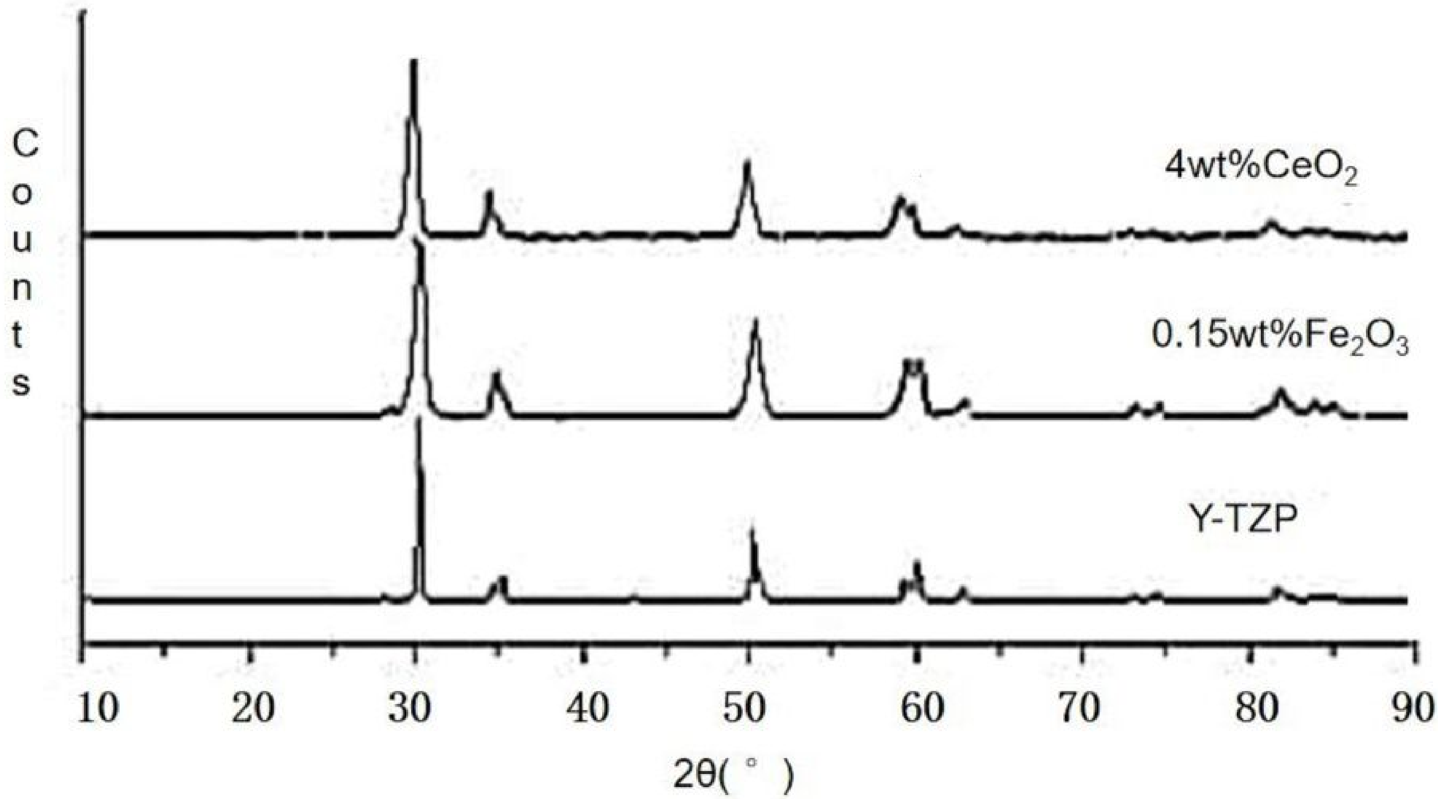
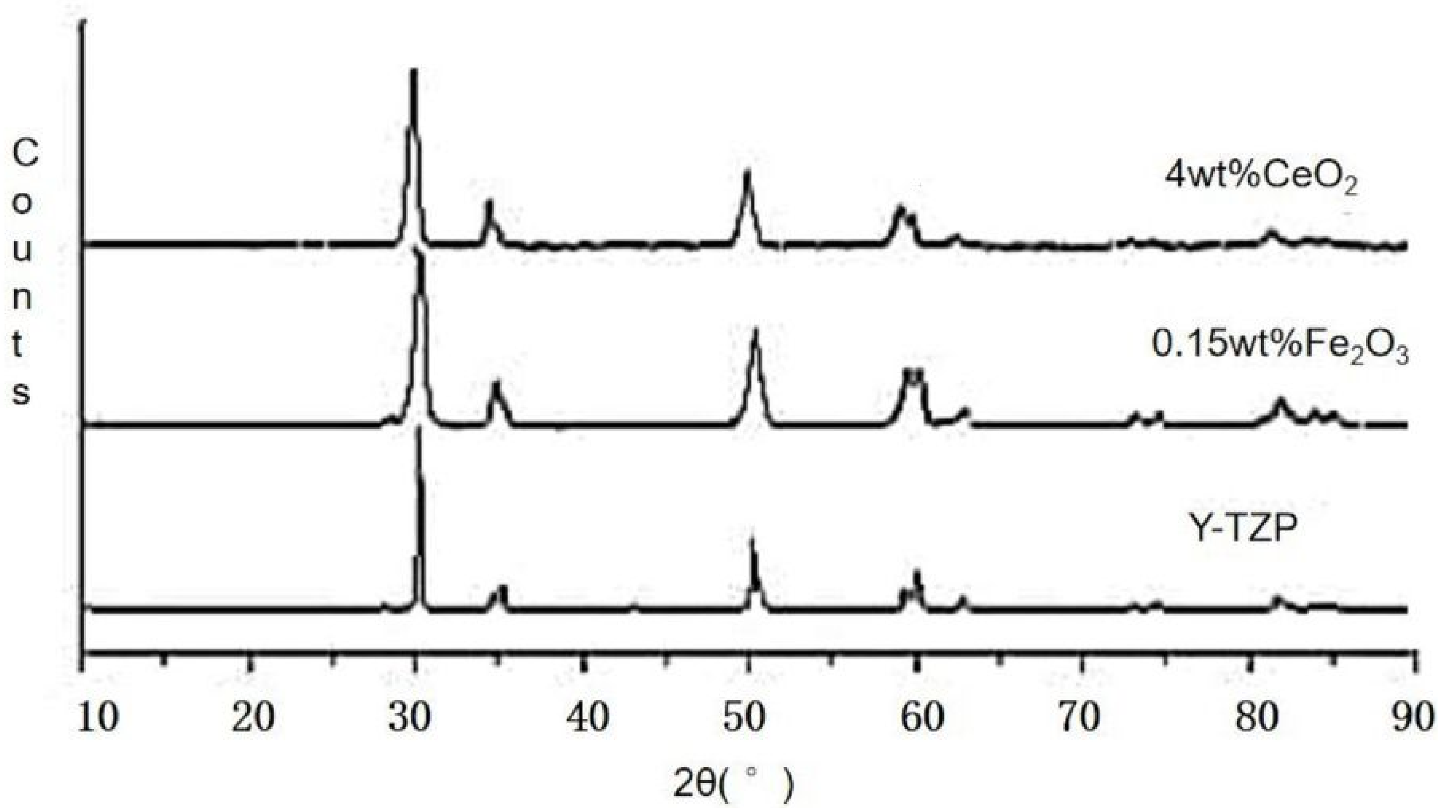


Figure 8

XRD pattern of the specimens 68x56mm (1200 x 1200 DPI)



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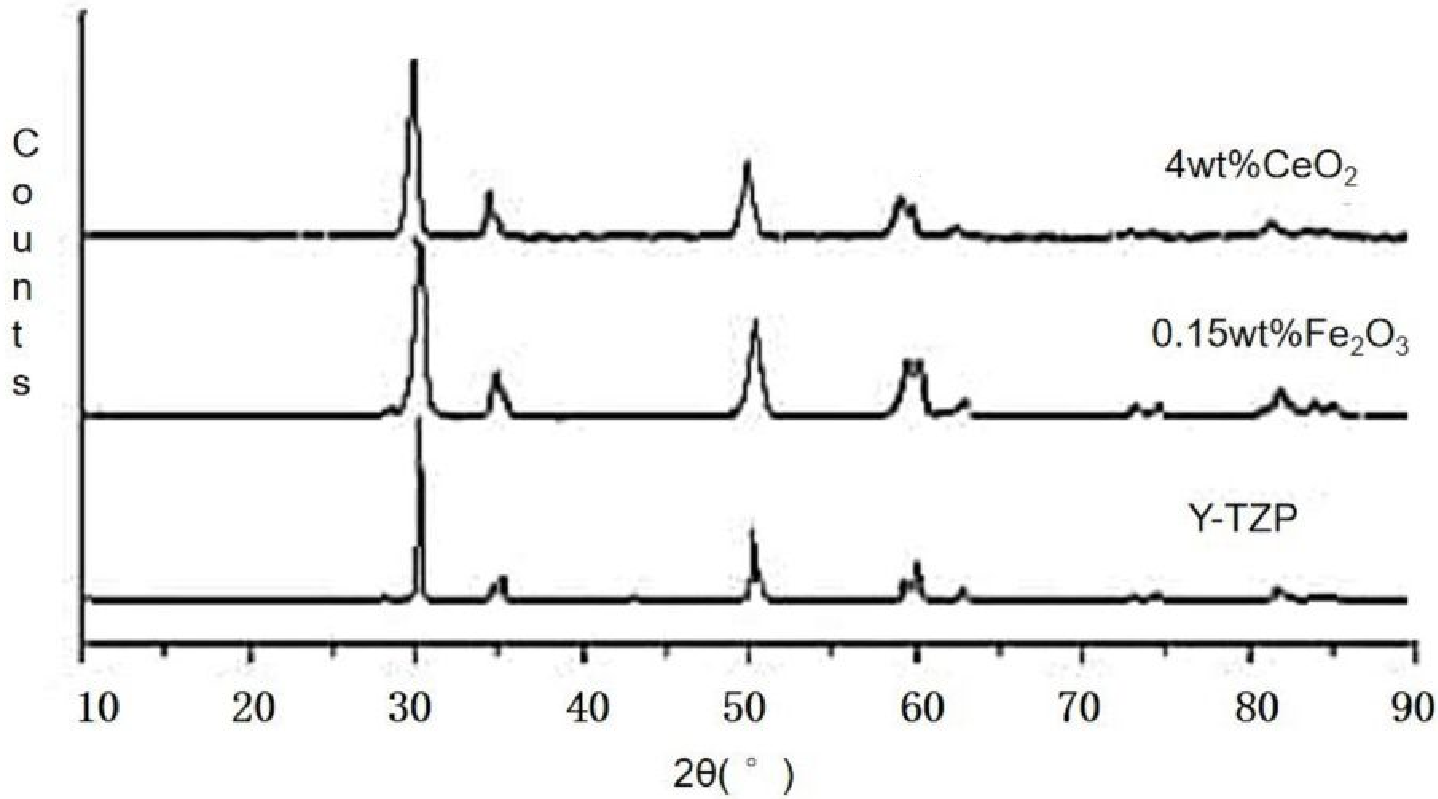

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