Article:

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Manuscript Details

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Title	The effects of different opacifiers on the translucency of experimental dental composite resins
Article type	Full Length Article

Abstract

Objective: The aim of this study was to evaluate the effects of different opacifiers on the translucency of experimental dental composite-resins. Methods: Three metal oxides that are used as opacifiers were tested in this study: titanium oxide (TiO2), aluminium oxide (Al2O3) and zirconium oxide (ZrO2). Experimental composite-resins were fabricated containing 25 wt.% urethane dimethacrylate (UDMA)-based resin matrix and 75% total filler including different concentrations of metal oxides (0, 0.25, 0.5, 0.75 and 1wt.%) blended into silane treated barium-silicate filler. The specimens (15.5 mm diameter and 1 mm thickness) were light-cured and tested in the transmittance mode using a UV/VIS spectrophotometer at wavelengths from 380-700 nm under a standard illuminant D65. The colour differences (ΔE^* ab) between different concentrations of opacifiers were also measured in transmittance mode based on their Lab values. Results: Statistical analysis by ANOVA and Tukey's test showed a significant decrease ($p<0.05$) in light transmittance with the addition of opacifiers to the experimental composite-resins. There was a linear correlation between different concentrations of TiO2 and Al2O3 and total transmittance. Total transmittance was also found to be wavelength dependent. The colour differences for the concentrations of 0-1 wt.% of the opacifiers were above 1 ΔE^* unit, with Al2O3 showing the smallest colour shift. Significance: The type and the amount of the opacifiers used in this study had a significant effect on the translucency of the experimental UDMA-based dental composite resins. The most effective opacifier was TiO2, followed by ZrO2 and Al2O3 in decreasing order, respectively.

Keywords	Composite resin; opacifier; translucency; color; dental materials.

Corresponding Author	Keyvan Moharamzadeh
Corresponding Author's Institution	University of Sheffield
Order of Authors	Karine Haas, Gulelala Azhar, Duncan Wood, Keyvan Moharamzadeh, Richard van Noort

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School Of Clinical Dentistry.

Keyvan Moharamzadeh BSc DDS PhD FHEA FDSRCS
Senior Clinical Lecturer and Honorary Consultant in Restorative Dentistry
Specialist in Prosthodontics Periodontics Endodontics and Restorative Dentistry
Claremont Crescent
Sheffield S10 2TA
Tel: 0114-271 7910
Fax: 0114-226 5484
E-mail: K.Moharamzadeh@Sheffield.ac.uk

05 November 2016

To: Journal Dental Materials

Dear Sir/Madam

We would like to submit the attached manuscript entitled "The effects of different opacifiers on the translucency of experimental dental composite resins" for consideration for possible publication in the Journal DentalMaterials.

We look forward to your favorable consideration of our manuscript

Yours Sincerely

Keyvan Moharamzadeh

1. Introduction

It has been shown that the appearance of a restoration is influenced by many factors including color, translucency and opacity, light reflectance and transmittance, and surface texture[1]. The inherent translucency of tooth structure and different morphology across the surface contribute to the complexity of achieving a natural looking restoration. Furthermore, it is often challenging for the clinician to mask the dark visual effect of the oral cavity on a class III or class IV restoration, or when trying to mask intense discolorations on the tooth structure. In order to overcome these problems, the opaque shades and dentin shades of dental composite resins have been manufactured. These new shades have higher opacity compared to the standard monochromatic dental composite shades [1-5].

According to Ragain and Johnston [6], a translucent material or a tooth undergoes four optical phenomena when light reaches it: (I) specular transmission of the light flux through the tooth; (II) specular reflection at the surface; (III) diffuse light reflection at the surface; and (IV) absorption and scattering of the light flux within the dental tissues.

The color and translucency of the composite resin are influenced by its shade, thickness and background color [7]; matrix composition [8]; filler particle size and content [9], pigment additions [10] and potentially the initiation component and filler coupling agent [11]. It has been also reported that translucency and color of resin composites are affected by depth of cure [12], light transmittance [13], and two wavelength-dependent elements such as absorption coefficient and scattering coefficient [14].

Scattering of light is an effect of refraction and reflection at the interface between the resin matrix and particles or voids [13]. It has been reported that opacifiers in composite resins can act as scattering centers and therefore, affect their translucency.

Metal oxides such as titanium oxide (TiO_2), aluminium oxide (Al_2O_3) and zirconium oxide (ZrO_2) are known opacifying agents which are added in minute amounts to the resin mixture [13-15].

However, studies regarding the effects of pigments and opacifiers at different concentrations in composite resins are rare. An ideal opacifier is the one that is able to mask the unwanted discoloration or background darkness efficiently in minute concentration.

The aim of this study was to evaluate the effects of different opacifiers on the translucency of the experimental dental composites.

2. Materials and Methods

2.1. Specimen Composition:

All the materials used in this study for fabrication of the experimental composites, except for the opacifiers (metal oxides), were supplied by Dentsply (Konstanz, Germany).

Resin matrix was prepared by mixing the following ingredients: UDMA (99.22%), camphorquinone (CQ) (0.3%), dimethylaminobenzoic acid ethyl ester (DMABE) (0.3%), 3,5-di-tert-butyl-4-hydroxytoluene (BHT) (0.12%) and 2-hydroxy-4-methoxybenzophenone (HMBP) (0.06%).

The experimental composite resins were produced by mixing 25wt.% of resin matrix with 75 wt.% of filler.

The filler used was silane treated barium silicate glass filler (particle size 1.5µm). Three metal oxides were used as opacifiers: titanium oxide (TiO_2), aluminum oxide (Al_2O_3) and zirconium oxide (ZrO_2) - particle size of all <5µm, according to manufacturer (Sigma-Aldrich, Dorset, UK).

2.2. Specimen Groups

13 groups (Table 1) of experimental composite resins were made containing different concentrations of the opacifiers: 0.25, 0.5, 0.75 and 1 wt.%. The metal oxides were blended in the filler mixture, giving the same total filler content of 75 wt.% for all four groups. A control group with no opacifier was also prepared.

As the silica filler varied in minute amounts for the four groups to give the same total content of 75 wt% of filler, an additional group was tested in a pilot study containing no opacifier and 1 wt% reduction of glass filler and compared with the control group to evaluate whether varying only these minute concentrations of silica filler would significantly affect the translucency. No significant differences in optical properties were seen and therefore, only one control group was used for the purpose of this study (75 wt% of filler).

2.3. Specimen Fabrication

The ingredients were measured for the desired weight using an analytical balance (Mettler AJ100, Greifensee, Switzerland) and then were mixed by hand in small flexible plastic containers. Once mixed to a homogeneous paste, the experimental resin was ready to be placed into the moulds.

A polycarbonate sheet of 1.5 mm thickness, containing six holes of 15.5 mm diameter, was made to act as mould for the specimens. Each group of unpolymerized resin composite specimens was packed into the six moulds over a glass plate using a condenser, making sure no bubbles were created. Another glass plate was placed over the polycarbonate sheet and firm pressure was applied for twenty seconds. The specimens were then light-cured from both sides in three different locations for a total of 90 seconds. The light source unit (QHL 75, Dentsply) had an intensity setting of 450mW/cm^2.

Of the six polymerized specimens, three were chosen based on homogeneity and lack of porosities. The other three were discarded. A total of thirty-nine specimens were selected for the study (N=39).

The specimens were ground using a silicon carbide grinding paper (Buehler-Met® II, Buehler UK, Coventry) P400 to the thickness of 1.3 mm, and subsequently polished with a P1200 to the thickness of 1mm (±0.05 mm) for a smooth finish. This was carried out on a grinder-polisher machine (Buehler Metaserv, Buehler UK) rotating at 200 rpm speed. A micrometer was used to check thickness of the specimens in five different locations (one at the centre and four at the corners). A bright light source was used to check for porosities. Specimens that showed inappropriate thickness and/or porosities were discarded and replaced.

Each specimen was then rinsed with water, dried and stored with the other two specimens of the same group in a dry environment in a self-sealing small poly bag.

2.4. Measurement of Optical Properties:

Optical properties data were collected using a computer-controlled spectrophotometer (Lambda 2, PerkinElmer, Massachusetts, USA) with integrating sphere accessors. Transmittance (total, diffuse and total direct) was measured in the wavelength range of 380-700nm under standard illuminant D65 at 1nm intervals. Color coordinates, L* (lightness), a* (red-green chromaticity index), and b* (yellow-blue chromaticity index) were determined from the total transmittance data using Pecol color software (PerkinElmer, USA).

For Total Transmittance and Diffuse Transmission, measurements were taken for every wavelength from 380 nm to 700 nm, resulting in 321 readings. For Total Transmittance measurement, a specimen was placed in the transmission port (entry port) of the spectrophotometer and a white reference material was placed in the reflectance port.

For Diffuse Transmission, a light trap needs to exist in the reflectance port. The light trap absorbs the direct transmission, and therefore only scattered light is measured. A light trap can be either a black background or an open port. In this study, an open port was chosen as a light trap.

For direct transmittance, the values of total transmittance were subtracted from diffuse transmittance, to measure light passing through the samples without scattering.

Color measurements were taken using CIE Lab values in total transmittance mode. Color difference (ΔE*) was measured using the following equation:

$$\Delta E^*ab = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{0.5}$$

2.5. Statistical analysis

Statistical analysis of the data was carried out by one-way ANOVA followed by Tukey's analysis, as well as Regression Analysis using the Minitab statistical analysis software.

3. Results

Mean total, diffuse and direct transmittance for different concentrations of titanium oxide (TiO2), aluminium oxide (Al2O3) and zirconium oxide (ZrO2) are presented in Figures 1, 2 and 3 respectively The charts show that the addition of TiO_2 had the most significant reduction in transmittance of the experimental resin composites, whilst ZrO_2 and Al_2O_3 were in the second and third rank, respectively.

Statistical analysis by one-way ANOVA followed by Tukey's test showed that total transmittance of the experimental resin composites were significantly decreased by the addition of the opacifiers used in this study.

Regression analysis showed that there was a linear correlation between concentrations of TiO_2 and total translucency (r^2=92.9%) and between concentrations of ZrO_2 and total translucency (r^2=92.8%).

Regression analysis also showed that Al_2O_3 had a less linear correlation (r^2=0.87) between different concentrations and total translucency of experimental resin composites.

CIE Lab results for TiO_2 showed that L* (Lightness) values varied from 84.50 (0%) to 36.90 (1%), which represents a shift towards the black end (darker) of the L* scale. However, the a* values showed less variation from 1.36 (0%) to 1.47 (1%), indicating a small shift to the red end of the a* scale. The b* values showed a considerable shift from 15.95 (0%) to 29.13 (1%), which is towards the yellow end of the b* coordinate.

CIE Lab results for Al_2O_3 showed that L* values varied from 84.50 (0%) to 75.14 (1%), which is less reduction in lightness than for TiO_2. The a* values did not vary considerably from 1.36 (0%) to 1.47 (1%), indicating a small shift to the red end of the a* scale, which was the same result as for TiO_2. The b* values showed a shift from 15.95 (0%) to 10.55 (1%), which is towards the blue end of the b* coordinate.

CIE Lab results for ZrO_2 showed that L* values varied from 84.50 (0%) to 60.70 (1%), which was also a reduction in lightness. The a* values showed a change from 1.36 (0%) to 1.54 (1%), indicating a small shift to the red end of the a* scale. The b* values showed a shift from 15.95 (0%) to 21.19 (1%), which is towards the yellow end of the b* coordinate.

The color difference between the composites with different concentrations of the opacifiers are shown in Table 2.

4. Discussion

The increasing demand for aesthetic procedures encourages the manufacturers to develop dental composites with shades that can highly mimic the natural tooth and also have the ability to hide tooth discolorations. These shades include dentine, enamel, opaque and bleach shades which contain various opacifiers and pigments. However little is known about their effect on the optical properties of the composite resins.

The use of experimental resin composites in this study allowed the control of the amount of certain ingredients and the elimination of variables, such as different additives found in different commercial dental composites. It was possible to examine different concentrations of only one component, such a specific opacifier within a range that would influence the aesthetics but would have minimum effect on the total filler content that is important for optimal mechanical properties.

The types of opacifiers chosen for this study were based on their properties as demonstrated by other studies [16-18] including good availability, affordable price, and biocompatibility. The opacifiers were metal oxides and their particle sizes were chosen to be the closest available to the glass filler particle size. Titanium oxide has a high refractive index, is a hard material and exists in various forms: anastase, rutile, brookite (very rare) and amorphous. Zirconum oxide also has a high refractive index and good mechanical properties. It is usually grown by reactive electron beam evaporation of zirconium in an oxygen background to compensate for possible dissociation during melting. Aluminum oxide is created when two aluminum atoms and three oxygen atoms combine together. Aluminum is a metal and oxygen is a gas. The compound is crystalline.

The sample preparation stage of this study aimed for minimum amount of porosity in the specimen discs. The pilot study involved the use of a vacuum machine to eliminate any air bubbles in the specimens, however this method was not successful as porosities were clearly visible in the specimen after four hours storage in the vacuum. The preparation method using glass slabs and manual pressure was then tested, and proved to be successful in producing minimum amount of porosities. The specimens were checked against a bright light source for presence of porosities and discarded accordingly. One may argue that this is a subjective method of accessing the specimens since it relies on the vision system of the observer, which may be different from another observer [18, 19].

The method to test the translucency of the composites by the transmittance mode is a simple method. As the purpose of this study was to evaluate the effect of different opacifiers and concentrations on the translucency of resin composites, a simple method was preferred. Other authors have previously measured translucency of composites and porcelains using transmittance mode [7, 20, 21].

The first part of the results of this study showed a linear relationship between the concentrations of TiO_2 and total translucency. With small additions of TiO_2, significant reduction in translucency was observed which was consistent with a previous study [22]. Adding small amounts of ZrO_2 into the resin composites also reduced the translucency with a linear relationship between the concentrations, however it was not to same extent as seen with TiO_2. The results for Al_2O_3 addition also showed a reduction in translucency with increasing amounts, nevertheless it did not show a linear correlation. These findings have not been previously reported in the literature. Since particle sizes were the same according to the manufacturer, reasons for these observations can be due to the difference in refractory indices of the materials. It is known that great mismatches of refractory index between the filler and the matrix can increase the opacity of the composites due to multiple reflection and refraction at the matrix phase interface [23]. This phenomenon causes a decrease in light transmittance, whereas a close match results in higher transmittance and therefore, more translucency [24]. The refractive indices of $TiO_{2,}$ Al_2O_3 and ZrO_2 are 2.49, 1.77 and 2.22, respectively. The barium silicate glass filler used in this study have a refractive index of 1.53 and the UDMA resin matrix has a refractive index of 1.48. As the refractive index of TiO_2 is the highest among all, it has the greatest mismatch with the resin matrix, which explains why this material causes higher increase in the opacity of the composites compared to the other two opacifiers with the same concentrations. The second most effective opacifier agent shown in this study is ZrO_2, followed by Al_2O_3, producing the smallest effect. These results are consistent with their differences of refractive index as mentioned above and the mismatch between them and the resin matrix. The diffuse transmittance followed the same curve pattern as for total transmittance. When analysing the total direct transmittance, however, it is noticed that Al_2O_3 shows less direct transmission than $ZrO_{2,}$ at the concentration of 0.25%. Some factors may have influenced these results: porosities within the resin composite causing more scattered light; or variations in filler fraction and filler thickness [25]; an error in mixing uniformly the resin composites; or any other procedural factors. Besides, the difference of numbers is not great when one looks at the scale of the direct transmittance values.

Data for total transmittance for different wavelengths showed the wavelength dependency of the measurements. These results were consistent with previous studies [13,20]. A decrease in light transmittance at lower wavelengths may be explained by higher scattering of light in the material. Furthermore, the pattern of the curve as wavelength increases shows a dip between 485-500, which may relate to the absorption peak of the photosensitizer (camphorquinone) in this range causing an increase in absorption and therefore, a rapid change in light transmission as shown in the study by dos Santos [20].

When analysing the graphs of total transmittance per wavelength for all opacifiers, the curves of 0% and 1% concentrations of TiO_2 showed a variation that was not proportional for the whole of the spectrum (380-700nm). This was not the case for the other two opacifiers. Thus, TiO_2 at a higher concentration may produce less variation in light transmittance across the spectrum.

The CIE Lab results showed that the addition of all opacifiers caused a decrease in lightness of the experimental resin composites, with TiO_2 showing darker values, followed by ZrO_2, and Al_2O_3 showing the lightest values. For the a* values, all opacifiers produced a small shift towards the red end of the scale, with ZrO_2 producing the biggest shift. For the b* values, it was found that TiO_2 and ZrO_2 caused a big shift to the yellow end of the b* coordinate, whereas Al_2O_3 produced a small shift towards the blue end of the b* coordinate.

The color difference (ΔE*) results showed that the addition of all three opacifiers to the resin composites produced color differences above one, which is considered perceptible to the human eye [26]. Color difference was higher for TiO_2, followed by ZrO_2, and Al_2O_3 in decreasing order, respectively. It was found that color differences for TiO_2 were also perceptible to the human eye in the study by Yu [22] where measurements for color difference were made in the reflectance mode, different from the present study that used transmittance mode. The reason for discrepancies in the color change may relate to the selective absorption and scattering of light by the opacifier particles [14].

The opacifier with the smallest color change was Al_2O_3. This may relate to the fact that Al_2O_3 was the least effective in changing the translucency of the resin composites. Therefore, an increase in the opacity of the composites in this study also caused an increase in the color difference. Another study also found that transmittance color is influenced by the translucency of the material [23]. However, color of a material cannot be measured using only one optical property such as light transmittance [13] and other measurements may be needed to measure color changes efficiently.

The opacifiers are only intended to alter the translucency of the composites and the color should be controlled by the addition of pigments only. This would make the composite formulations predictable in terms of the resultant color and translucency of the material, and it would potentially improve the process of shade matching.

The limitations of this study included the use of only three opacifiers with similar particle sizes although different forms of agglomeration of the particles may have occurred. Particle sizes were the closest available to the particle size of the silica filler. Smaller particles of the opacifiers have been previously studied [22]. Another limitation of this study was the fact that the evaluation of the samples relied on visual inspection of the observer.

Most studies evaluating the translucency and color of composite resins have used commercially available composites, which contain different opacifiers. There are no studies published in the literature comparing different types of opacifiers with different concentrations and evaluating their effects on the translucency of composite resins in such a strictly controlled experimental set up used in this study.

Further studies to investigate the effects of other pigments and colorants used in dental composites on their optical properties are recommended.

5. Conclusions

Within the limitations of the present study, TiO_2, ZrO_2, and Al_2O_3 decreased the translucency of the experimental composite resins. There was a linear correlation between the amount of the opacifiers in concentrations between 0 – 1% and the translucency of the experimental composite resins.

The type and amount of opacifier had a significant effect on the translucency of experimental resin composites. The addition of the opacifiers also significantly influenced the perceptible color of the composites by approximately 1 ΔE^* unit. The ranking of the opacifiers in terms of the highest effect on the opacity and color change was TiO_2, ZrO_2 and Al_2O_3 in decreasing order, respectively.

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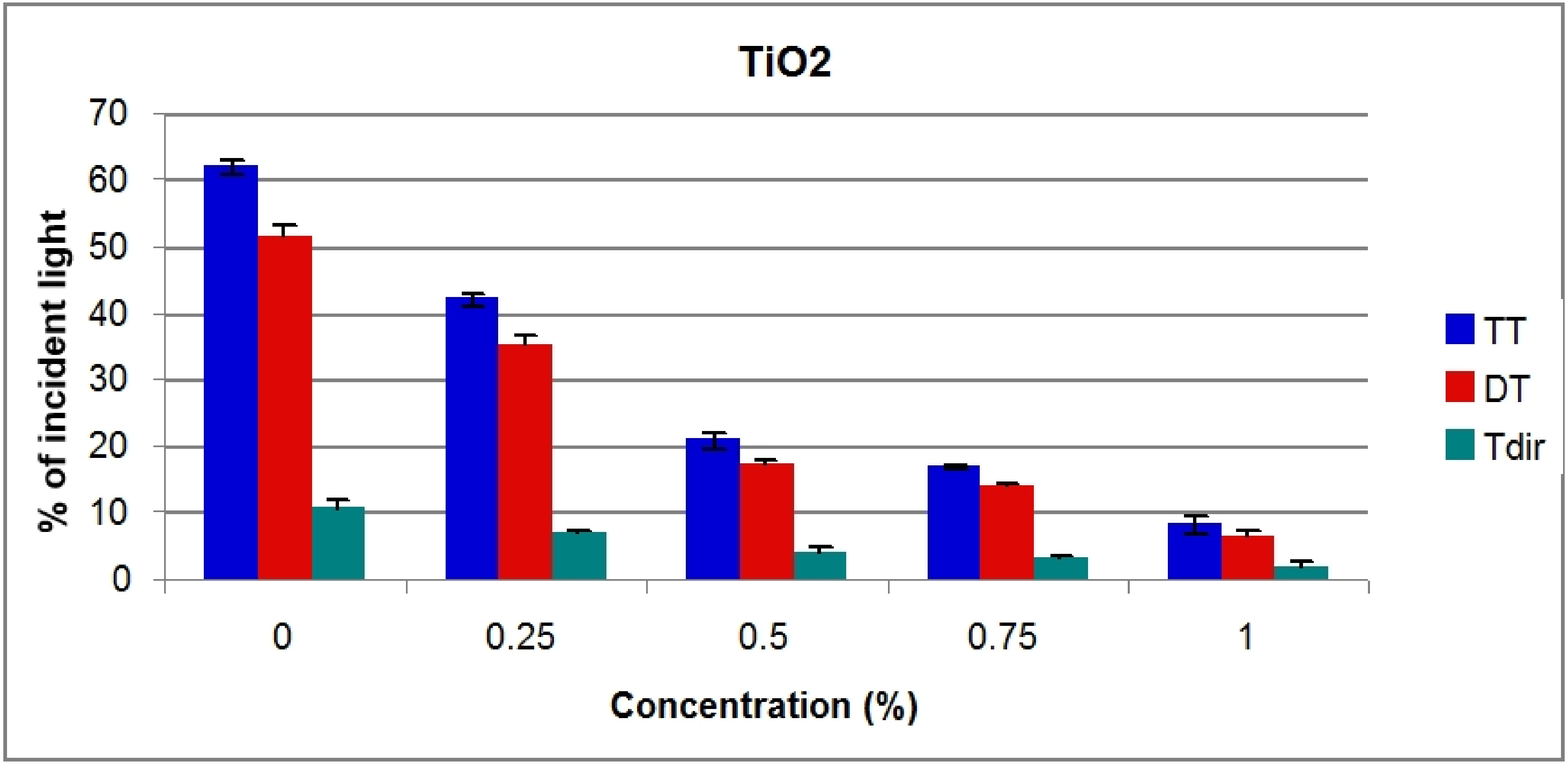
Figure captions

Figure 1 Translucency of experimental composite resins containing different concentrations of TiO_2

Figure 2 Translucency of experimental composite resins containing different concentrations of Al_2O_3

Figure 3 Translucency of experimental composite resins containing different concentrations of ZrO_2


TiO2
% of incident light
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60
50
40
30
20
10
0
0
0.25
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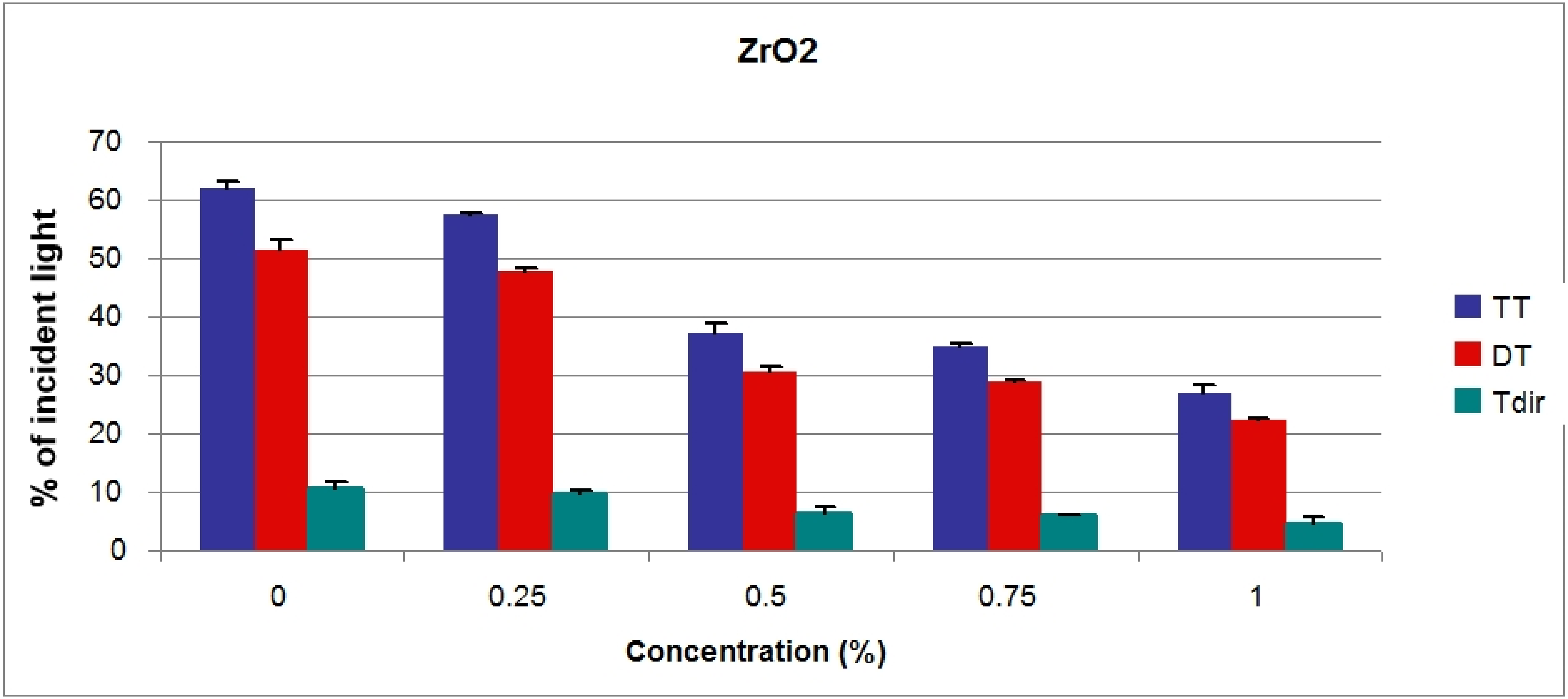
1
Concentration (%)
TT
DT
Tdir



ZrO2
70
60
50
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30
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0
% of incident light
0
0.25
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Concentration (%)
TT
DT
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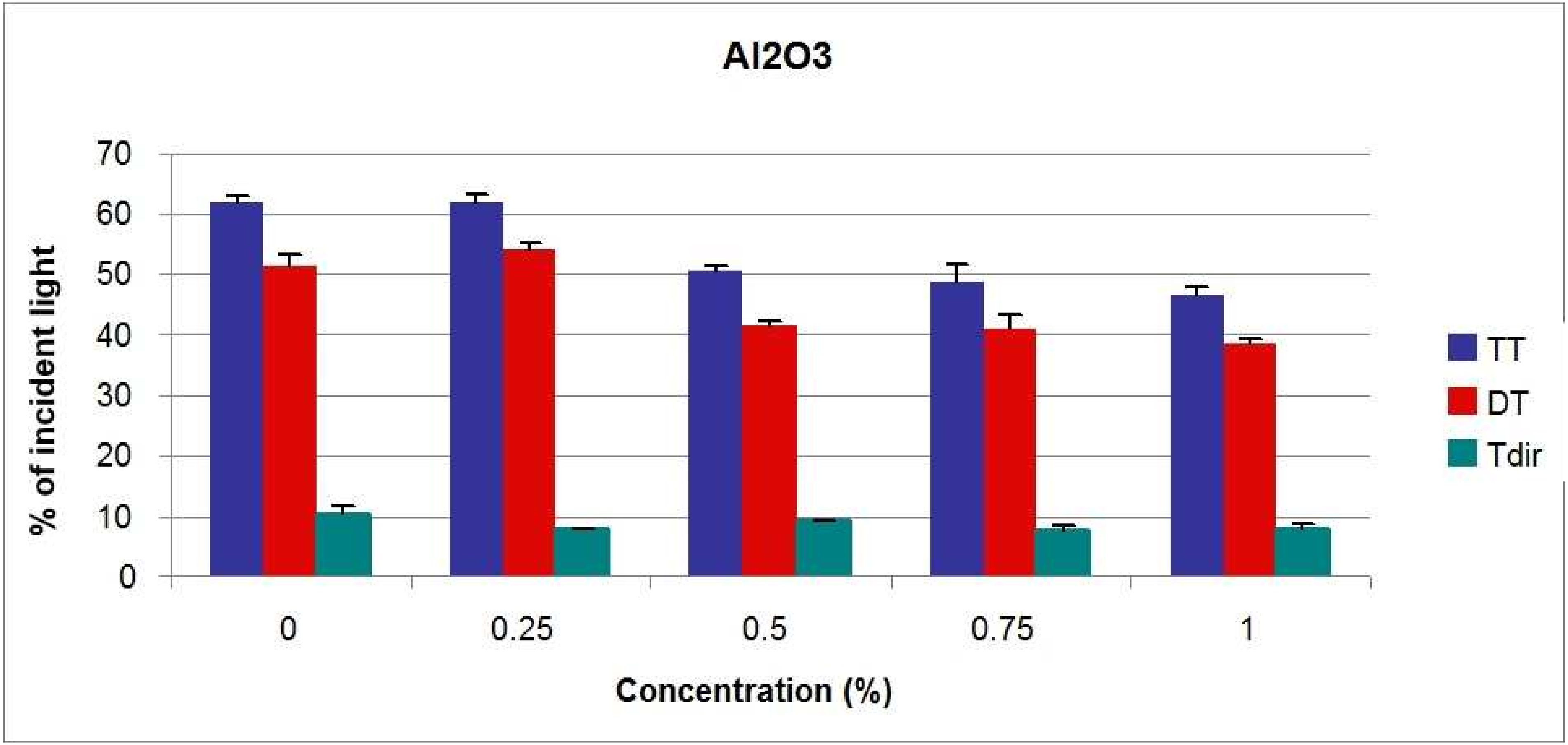
Al2O3
70
60
50
40
30
20
10
0
% of incident light
0
0.25
0.5
0.75
1
Concentration (%)
TT
DT
Tdir

Table 1: Composition of the filler and opacifiers in different experimental composite resins

	Silica Filler wt.%	TiO_2 wt.%	Al_2O_3 wt.%	ZrO_2 wt.%
Composition 1	74.75	0.25	0	0
Composition 2	74.50	0.5	0	0
Composition 3	74.25	0.75	0	0
Composition 4	74	1	0	0
Composition 5	74.75	0	0.25	0
Composition 6	74.50	0	0.5	0
Composition 7	74.25	0	0.75	0
Composition 8	74	0	1	0
Composition 9	74.75	0	0	0.25
Composition 10	74.50	0	0	0.5
Composition 11	74.25	0	0	0.75
Composition 12	74	0	0	1
Composition 13	75	0	0	0

Table 2 Color difference between composite resins with different concentrations of opacifiers

	TiO_2	Al_2O_3	ZrO_2
ΔE* between 0% and 0.25%	15.02	5.91	5.91
ΔE* between 0.25% and 0.5%	15.69	2.47	3.19
ΔE* between 0.5% and 0.75%	9.91	2.53	12.02
ΔE* between 0.75% and 1%	29.44	4.78	10.91

Abstract

Objective: The aim of this study was to evaluate the effects of different opacifiers on the translucency of experimental dental composite-resins.

Methods: Three metal oxides that are used as opacifiers were tested in this study: titanium oxide (TiO_2), aluminium oxide (Al_2O_3) and zirconium oxide (ZrO_2). Experimental composite-resins were fabricated containing 25 wt.% urethane dimethacrylate (UDMA)-based resin matrix and 75% total filler including different concentrations of metal oxides (0, 0.25, 0.5, 0.75 and 1wt.%) blended into silane treated barium-silicate filler. The specimens (15.5 mm diameter and 1 mm thickness) were light-cured and tested in the transmittance mode using a UV/VIS spectrophotometer at wavelengths from 380-700 nm under a standard illuminant D65. The colour differences (ΔE^* ab) between different concentrations of opacifiers were also measured in transmittance mode based on their Lab values.

Results: Statistical analysis by ANOVA and Tukey's test showed a significant decrease ($p<0.05$) in light transmittance with the addition of opacifiers to the experimental composite-resins. There was a linear correlation between different concentrations of TiO_2 and Al_2O_3 and total transmittance. Total transmittance was also found to be wavelength dependent. The colour differences for the concentrations of 0-1 wt.% of the opacifiers were above 1 ΔE^* unit, with Al_2O_3 showing the smallest colour shift.

Significance: The type and the amount of the opacifiers used in this study had a significant effect on the translucency of the experimental UDMA-based dental composite resins. The most effective opacifier was TiO_2, followed by ZrO_2 and Al_2O_3 in decreasing order, respectively.

Keywords: Composite resin; opacifier; translucency; color; dental material.

The effects of different opacifiers on the translucency of experimental dental composite resins

Karine Haas, Gulelala Azhar, Duncan J Wood, Keyvan Moharamzadeh*, Richard van Noort

School of Clinical Dentistry, University of Sheffield,

Claremont Crescent, Sheffield, S10 2TA, United Kingdom

Corresponding author;

Tel: +44 114 2717910, Fax: +44 114 2665326

Email: k.moharamzadeh@sheffield.ac.uk