Influence of Wine Components on the Reflection Properties of the Visible Light of Dental Composites

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In our study we aimed to identify the individual influence of the main components of wine on the studied composites. The samples were immersed in water, wine, alcohol, tartaric acid solution, at 37^{0}C. We measured the reflectance of the surfaces of the composite materials with UNICAM 4 UV-VIS spectrophotometer after 24 h, 7 days and 28 days. The t test results for delta %$(x_{nm})_{ts}$ of the dental composite material at the same moment in time and different solutions (within the group test) identified a single group which revealed no statistical significant difference. The studied materials at the same moment in time (between group tests) will respond statistically different to the immersing solution. We expect that the changes in the composition of composite materials, including activation systems, inorganic fillers, resin matrix, silane will change the response of the composite materials to the immersion solutions.

Keywords: dental composite materials, wine components, reflectance changes

The dental materials are selected by the dentist based on the different physical, mechanical properties, on the chemical reactivity and on the biocompatibility. Parts of the physical properties are the optical properties. The best optical properties regarding dental restoration materials have the composites [1].

Most of the studies that focus on the optical properties of the dental materials measure the CIEL*a*b* parameter and afterwards the colour change with ΔE [2-4]. Other studies examine translucency, the contrast ratio [5-7] and the opalescence. [5, 7, 8] Only a few studies analyse the spectral distributions [8, 9].

It is known that the composites change their optical properties when being introduced in different staining solutions [10-13]. Minimum modifications in colour occur at the immersion of the composites in water and major ones occur at the immersion in red wine [13-15]. Red wine is a complex natural product containing besides water approx. 10-13% ethanol, tartaric acid (majority) and natural pigments (especially malvidin 3-glucoside). The influence of the components of wine on the optical modifications of the composites is little known. In our study we aimed to identify the individual influence of the main components of wine on the studied composites.

Because the resistance to the classes of dental composites has varied in our prior studies we brought in this study a composite of the latest generation, a nanocomposite respectively a microhybrid composite [12-14]. We tested the qualities of the light refection of the materials studied before and after the immersion in the solutions of the main components of wine. The objective of this experiment has been to analyse the influence of the cofactors from wine on the reflectance of the surfaces of the composite materials. The null hypothesis is that we will not be able to analyse the influence of the factors of wine on the reflection properties of the composites.

Experimental part

Two different material composites were taken into study Valux Plus shade A2, Filtek Ultimate A2 Body Shade. The table below shows the composition of the dental materials (table1).

The samples were realized with a mould having a diameter of 30mm and a thickness of 2mm. These dimensions are necessary so as to be able to be read with UNICAM UV-Vis [2]. The composite was inserted and compressed with the spatula in mould afterwards a polyester film was applied and pressed with a 1mm thick glass stab.

The polymerization of the materials was performed in 9 points, with a photo-polymerization lamp LED Elipar Freelight 2, 3M ESPE, guide ∅ 8 mm, 1000mW/cm^2, 20 s.

Table 1
CHEMICAL COMPOSITION OF DENTAL COMPOSITES

Composite name	Organic phase	Inorganic phase	Manufacturer
Valux Plus	Bis-GMA, TEGDMA	zirconia/silica	3M ESPE
Filtek Ultimate	Bis-GMA, UDMA, TEGDMA PEGDMA, Bis-EMA	SiO_2, ZrO_2, SiO_2/ZrO_2 aggregates	3M ESPE

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In conformity with the recommendations of the producer of the polymerization lamp an exposure of 10 seconds is sufficient for a material layer of 2 mm thickness of the shade A2. The total surface of polymerization is of 706.50mm^2 (for π =3.14). The surface of the polymerization lamp guide is of 50.24 mm^2 (π=3.14). The surface of a circle with a large diameter can be completely covered with circles of a smaller diameter only if they overlap. Because of the need of overlapping the exposure areas we chose to increase the exposure time to 20 s and to use a pendulum movement to cover a surface bigger than that of the guide, similar to the technique used in the realization of fillings of large dimensions. The energy required for polymerization of material is 70.65J. The amount of energy used for polymerization was 90.432J. The polymerization of the material with a technique similar to the one used in the dental office increases the clinical significance of the results.

Because the thickness of the composite layer is not larger than 2.5 mm it was sufficient to realize the polymerization of the samples on a single surface [16]. The surface was finished in several steps with abrasive paper with increasing granulation up to 1600 grits. The final thickness of the samples was of 2mm (±5%). The checking of the thickness has been performed with a digital micrometer. Then all prepared specimens were stored in distilled water at 37°C for 24 hours for rehydration and completion of the polymerization [13, 12, 17].

In order to identify the effect of the cofactors on the surface reflectance of the composites we used the water as reference. For a maximum of colour modifications we used red wine (Cabernet Sauvignon 2011, Recas Winery). We prepared ethanol solutions with a concentration equal to that of the studied wine (13% as stated on label) and a solution of tartaric acid in distilled water with a *p*H equal to that of the wine (3.45).

The samples were immersed in water, wine, alcohol solution, tartaric acid, at 37 C (table 2). The samples were subsequently removed from the solutions, rinsed with water and dried, and then measured after 24 h, 7 days, 28 days with UNICAM 4 UV-Vis spectrometer. The measurement was performed on a black background.

The statistical analysis has been performed by means of SPSS Statistics 21 (IBM Corp.) The statistically processed data was of scalar type representing % reflection for a spectral area of 10 nm. Starting from the measured values for each wave/probe length we calculated a medium value $\%(x_{nm})_{ts}$, where x_{nm} represents the interval of wave length for which the calculation is done, t defines the time interval at which the measurement is performed (24h, 7 days, 28 days), s represents the type of solution (water, acid, alcohol, wine). Using the measured values from the start of the experiment as basic values we calculated the variation % reflection by means of the formula delta $\%(x_{nm})_{ts} = \%(x_{nm})_{ts} - \%(x_{nm})_b$ in which x_{nm}, t,s represent the ones defined above while b defines the medium base value for that wave length.

Table 2
ALCOHOL CONCENTRATION AND *p*H OF THE IMMERSION SOLUTIONS

Solutions	Concentration of ethylic alcohol	pH
Water	0%	6.57
Ethylic alcohol solution	13%	5.86
Tartaric acid solution	0%	3.45
Wine	13%	3.45

We consider that the effects of tartaric acid and ethyl alcohol on the composite materials studied occur because of the distilled water and the substances dissolved in it. Subtracting the measured effect of distilled water (witness probe) from the effect of the studied solutions one can find out which is the effect caused by the tartaric acid ($var\%(x_{nm})_{tacid}$) respectively alcohol ($var\%(x_{nm})_{talcool}$). Continuing in the same direction we calculated the colorant effect of the substances from wine excluding the effect of distilled water, of the tartaric acid and that of alcohol ($var\%(x_{nm})_{tvin}$).

The statistical tests were performed both on delta $\%(x_{nm})_{ts}$ and on $var\%(x_{nm})_{ts}$. The graphical representations analyzed have used delta $\%(x_{nm})_{ts}$ and $var\%(x_{nm})_{ts}$ both separately and together. Because we deal with the comparison of some variations of the same wave length we performed the statistical testing with the paired t-test. We compared the samples at the same moment of measurement (24h, 7 days, 28 days) as well as for the same solution at different moments in time.

Results and discussions

For performing the first set of paired t-test we grouped the samples according to their material and moment of measurement. There resulted following pairs for both studied materials: water24h – acid24h, water24h - alcohol24h, water24h - wine24h, acid24h - alcohol24h, acid24h - wine24h, alcohol24h - wine24h, water7d - acid7d, water7d- alcohol7d, water7d - wine7d, acid7d - alcohol7d, acid7d - wine7d, alcohol7d - wine7d, water28d - acid28d, water28d – alcohol28d, water28d - wine28d, acid28d - alcohol28d, acid28d- wine28d and alcohol28d – wine28d. The t test results for delta $\%(x_{nm})_{ts}$ the Valux (3M ESPE) material at the same moment in time and different solutions revealed the existence of differences statistically significant for all combinations (t between -12.1 and 6.67, Sig. (2-tailed) between 0.000 and 0.007). The t test results for delta $\%(x_{nm})_{ts}$ the Filtek material (3M ESPE) the same moment in time and different solutions identified a single group with no statistically significant differences, the group water24h - alcohol24h (t -0.303, sig. (2-tailed) 0.764). For all other pairs the differences were statistically significant (t between-23.38 and 22.97, Sig. (2 tailed) between 0.000 and 0.045)

We compared the variation of reflectance at different moments in time for the samples immersed in the same solution. There resulted following pairs for both materials studied: water24h - water7d, water24h – water28d, water7d - water28d, acid24h - acid7d, acid24h - acid28d, acid7d - acid28d, alcohol24h - alcohol7d, alcohol24h - alcohol28d, alcohol7d - alcohol28d, wine24h - wine7d, wine24h - wine28d, wine7d - wine28d. We observed significant differences between the samples at different moments in time no matter of the immersion solution or of the composite material used. For Valux t had values between -16.01 and -6.19, Sig. (2-tailed) 0.000. For Filtek t had values between -18.18 and 23.00, Sig.(2-tailed) 0.000.

We compared the influence of the acid/alcohol/pigment on the two materials at different time intervals by using $var\%(x_{nm})_{ts}$. The statistical results which are all significantly different indicate that the studied materials at the same moment in time will respond differently to the immersion solution. The realization of statistical tests on the $var\%(x_{nm})_{ts}$ is not justified for the same material because this variation was studied in the statistical analysis of delta $\%(x_{nm})_{ts}$

The graphical analysis indicates that the values of the reflectance tend to slightly decrease in time for the Valux material no matter of the solution of immersion (fig. 1).

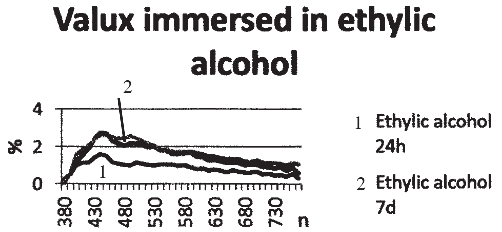

Fig. 1. Pattern typically for dental composite material Valux. Reflectance is reducing in time. (greater values means less reflectance)

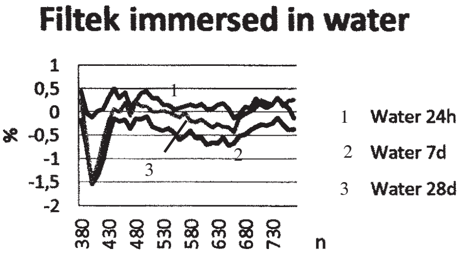

Fig. 2. Reflectance changes induced by immersion of Filtek samples in water at different moments in time

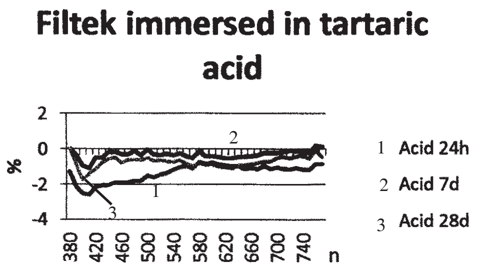

Fig. 3. Reflectance changes induced by immersion of Filtek samples in tartaric acid solution at different moments in time

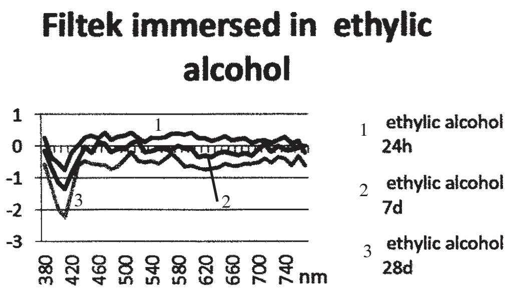

Fig. 4. Reflectance changes induced by immersion of Filtek samples in ethylic alcohol solution at different moments in time

The evolution in time of the reflectance of the Filtek material showed, within the limits of our study, that the samples immersed in distilled water initially presented (24h) a reduction of reflectance. Then, 7 days later an increase of it was shown and then after the 28th day it returned to 0 (fig. 2). In the case of the samples immersed in tartaric acid we observed an initial increase of reflectance but also a decrease of reflectance after 7 days and then a slight increase again after 28 days (fig. 3). The alcohol solution increases the reflectance of the samples and the wine reduces their reflectance in time. (fig. 4 and 5).

The graphical representation of var%$(x_{nm})_{ts}$ according to the solution of immersion, material and time interval highlights the different behaviour of the material when immersed in different solutions (fig. 6). We pointed out above, that the modifications at the Valux material of the total variation (water+solution, delta %$(x_{nm})_{ts}$) are increasing from one measurement to the other no matter of the solution of immersion. The analysis of the graphics

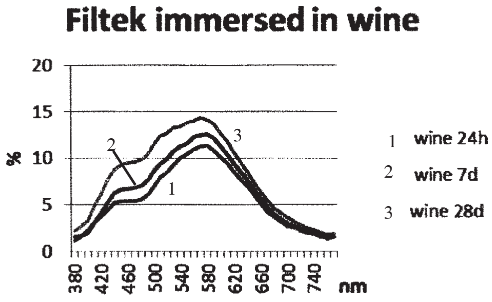

Fig. 5. Reflectance changes induced by immersion of Filtek samples in wine at different moments in time

var%$(x_{nm})_{ts}$ for Valux and Filtek tartaric acid solution and ethylic alcohol solution indicate that the variation of reflectance specific to the diluted substance (var%$(x_{nm})_{ts}$ alcohol and acid) is decreasing compared to that of the 7th day (this means that the reflection after 28 days is higher than the reflection after 7 days). We consider that the decreasing effect of the reflection in time for the Valux material immersed in alcohol solution or acid is caused basically by the water of dilution. In the case of the Filtek material, the modifications occurred at the immersion in tartaric acid or ethanol seems to be caused by the tartaric acid and ethanol and not by the water of dilution.

A graphical representation for the Filtek material indicating the variation of reflectance of the witness group (water) and the variation caused by the alcohol (fig. 7) shows a different behaviour of reflectance according to the wave length for the two solutions.

Studying the graph for the variation of reflectance specific to the pigments from wine (fig. 8) one can observe that the effect of the pigments on the Filtek material is more intense than on the Valux material for the same moment in time. The effect of the pigments on the Valux material seems to be increasing in time. In case of the Filtek material we have obtained great modifications in colour starting with 24 h after the immersion the variations being small for the next 7 days after which there is a general increase on the whole luminous spectrum.

Studying the above graph (fig. 9), we can observe at the Filtek material after 28 days that in reality the effect of the pigments from wine (the blue line, superior) is slightly reduced by the influence of water, acid and alcohol, stronger towards the extreme blue end of the visual spectrum.

Regarding the Valux material (fig. 10) after 28 days one can observe that the effect of the colorant (blue line) is strengthened by the effect of water and slightly reduced by the effect of tartaric acid and alcohol.

In this study we emphasized % the reflectivity of light at the surface of the composite materials. Our aim was to identify the modifications of the visual spectrum of the material surfaces and not to perform a spectroscopic study regarding the molecular composition of the material. Therefore we did not insist on the chemical signification of some peaks measured during the experiment [18-20]. In this case there were performed measurements on a black background to eliminate the light transmitted and reflected from the background back side of the material as much as possible. The reflection at the surface, the reflection from within the material, the reflection at the surface opposed to the one measured as well as the fluorescence and opalescence of the material remain in discussion for the spectral composition measured.

Some studies discuss about the Kubelka-Munk theory for the evaluation of the colour properties of the materials [21-23]. Because the aim of our study is to identify the

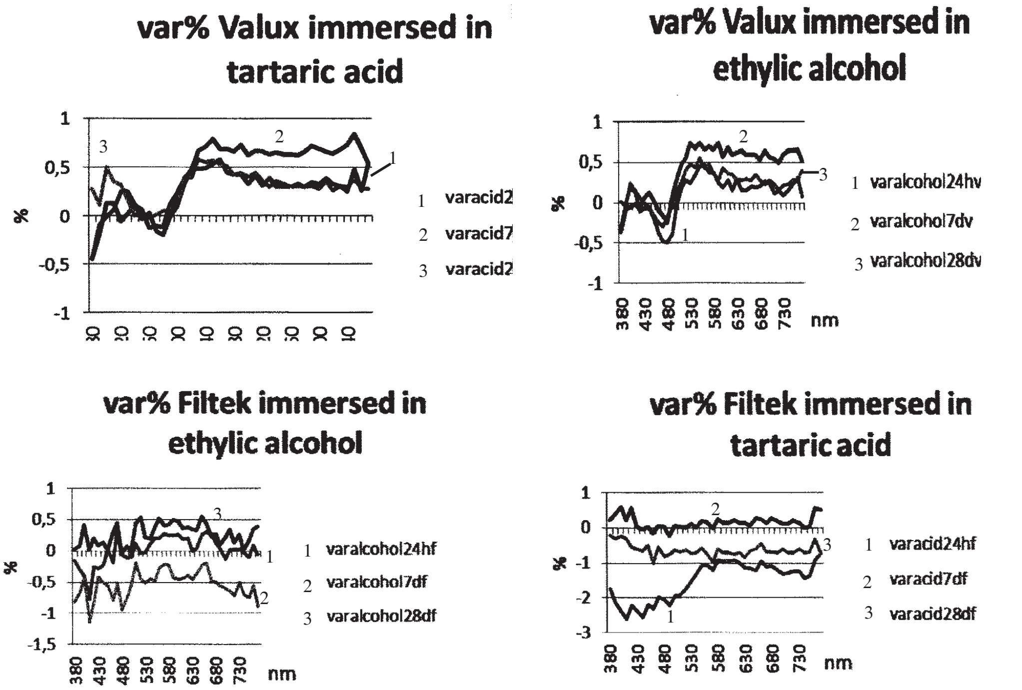

Fig. 6. Comparative graphics for specific effect of tartaric acid and ethylic alcohol on studied composite materials

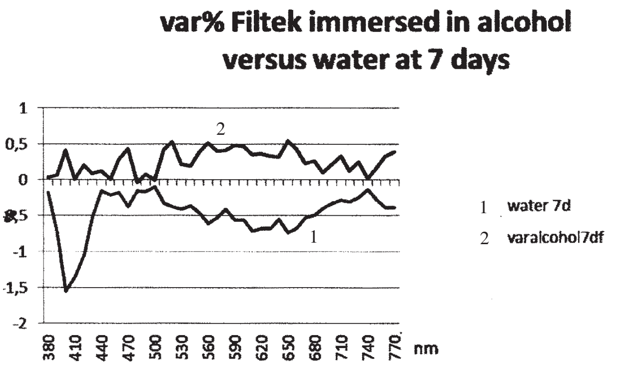

Fig. 7. Comparative representation of the reflectance changes for the Filtek samples immersed in water after 7 days and the effect of ethylic alcohol on the same material

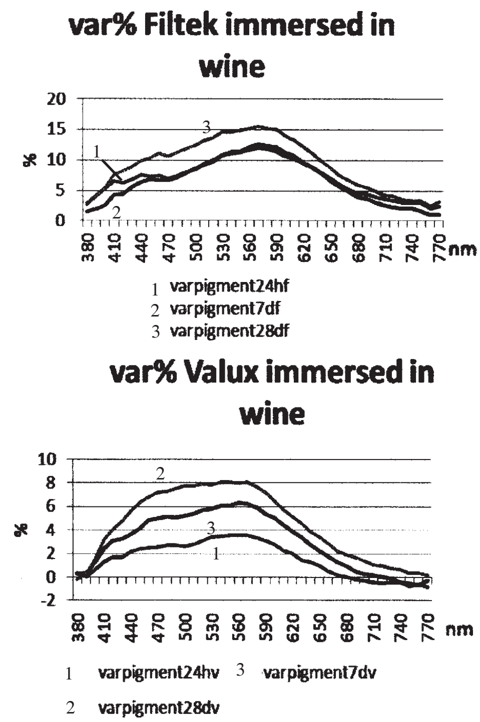

Fig. 8. Comparative representation of the specific effect of wine pigments on the studied dental composite materials

reflection modifications caused by the cofactors from wine and not the colour change of the dental materials we did not consider it however necessary to use these theories in our study.

In the clinical practice the last steps in realizing a composite filling are polishing and finishing [1]. All composites will roughen in time as the surface is exposed to the erosive and abrasive effect of food or drink [24]. As the roughness increases there occur modifications in color at the immersion in liquids [12]. The increase of roughness can be caused by the acid from drinks [25, 26]. An increased wear of composite materials was observed after the immersion in alcoholic beverages with over 9 vol% ethanol. In case of wine it looks that the wear is due to the content of alcohol [27]. In our study the effects caused by alcohol with a concentration of 13% and acid with a ph of 3,45 on the color of the composites are significant, a part being caused by these surface modifications. The maximum modifications of the degree of reflectance were measured at wine.

There could be observed an increased susceptibility of the dental composites to colour modifications in the presence of alcohol and acids. The results of the tests showed a close dependency on the materials used in the studies [28]. Our tests revealed that both the ethyl alcohol and the tartaric acid have the power to influence the reflection capacity of the dental composites. Interesting is the fact that in the case of some tested combinations (Filtek&wine) the cofactors from the solution seem to reduce the amplitude of the colour changes of the material.

Studies were performed on whether the dimension of the filling particles influence the results of the colouring tests of the composite materials but no consensus was reached [29]. In our study we identified statistically significant differences in the behaviour of the two materials at the immersion in different solutions but we could not specify whether the dimension of the filling particles is the cause for the different behaviours. The fact that there exist differences in the composition between the two materials studied makes the different behaviour of the materials even more difficult to be identified.

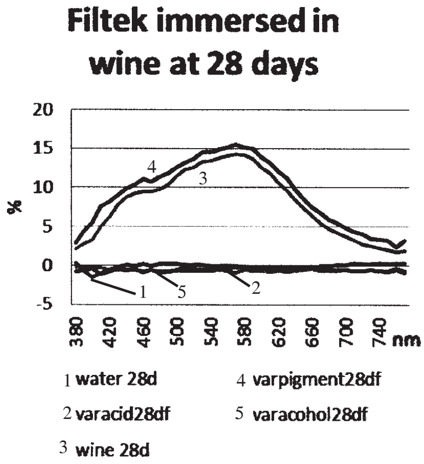

Fig. 9. Graphic representation of reflectance changes 28 days after the immersion of the Filtek samples in red wine versus samples immersed in water and the effect of tartaric acid, ethylic alcohol and wine pigments

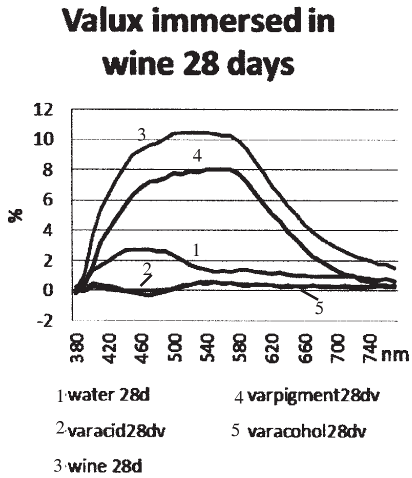

Fig. 10. Graphic representation of reflectance changes after 28 days of the Valux samples immersed in red wine versus the samples immersed in water and the effect of tartaric acid, ethylic alcohol and wine pigments

The colorants modify the colour of the surfaces by changing the spectrum of absorption/ refraction. As expected, the red colorant from wine determines an increased absorption of light in the green field of the spectrum (500-600 nm) and a somewhat more reduced absorption of light in the blue spectrum (400-500 nm). The closer we get to the red end of the spectrum the more reduced is the absorption effect of the colorant. As a result of these phenomena the composite material introduced in wine will get a reddish tinge and a reduced luminosity (because of the decrease of the entire reflected energy). The modification graph of the reflectance for the pigments from wine comes close in form to the malvidin 3-glucoside graph of absorbency, the main anthocyanin pigment from wine. The absorption wave that we have obtained is not completely superposing to the one from the studies because we have not studied the absorption spectrum of the pure anthocyanin colorant but rather its effect on the reflectance of the composite materials (which can differ). Moreover there are also other colorants in wine which influence the final colour of the wine. The colour of the anthocyanin pigments from wine changes according to the *p*H of wine and to its age, having shades of red, blue up to brick-red [30, 31].

Conclusions

The effect of the wine cofactors (tartaric acid and ethanol) on the surface properties of composite dental materials depends on the studied material. The studied materials have shown different responses to the immersion solutions. We expect that changes in the composition of the composite materials, including activation systems, inorganic filler, resin matrix, silane will modify the response of the composite materials regarding the solutions of immersion.

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