

Investigations of the Influence of LED Light on the Colour Stability of Mineral Pigments in Cellulose Binder

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Abstract: Main goal of this study is to develop and establish an adequate method to investigate mineral pigments of their phenomenological behavior by optical irradiation in the low-intensity field what is typical for indoor situations with artificial radiation. This is of urgent need as there is no standardized and differentiated method available until now. The method is based on the description of the induced colour change by applying the remission spectroscopy. Gaining curves of colour distance for each specific sample material, it is possible to conduct qualitative analyses and to categorize the optical radiation stability. Besides, spectral object sensitivity and validity of the reciprocity low concerning the group of mineral pigments are also investigated. Further the influence of light, respectively the wavelength range from 380 nm to 780 nm is the primary focus of the investigation. This particular interest follows from an increasing application of LEDs which mainly emit visible radiation. The results are essentially aimed to provide practical advices for conservators and peoples in charge for valuable objects like they are situated in museums and exhibitions. As pigments are almost exclusively applied in binders, for the irradiation experiments exemplary a modern binder is chosen. This binder is a cellulose binder, which is amongst other fields, nowadays used for restauration of historical wall paintings.

Keywords: Optical Radiation Stability, Colour Change, Categorization System, LED Light, Mineral Pigments, Cellulose Binder

1. Introduction

The here presented work is an excerpt of a large-scaled scientific study concerning a comprehensive investigation of optical radiation induced colour change in mineral pigments. Primarily, new investigation methods had to be developed as there are no standardized and sophisticated methods for specification of optical radiation stability of colourants available. Further, in literature optical radiation stability is mostly declared very imprecisely and does not include pigments environment (binder, underground, climate, etc.) and radiation parameters (exposure time, spectral distribution, intensity). Based on that circumstances the study started to develop methods exemplarily for the group of mineral pigments. This group of colourants is very interesting as it is generally assumed to be very stable

against optical radiation. Although mineral pigments are widely used in all sorts of paintings their optical radiation stability are not investigated extensively yet. So a representative palette of mineral pigments was chosen and their phenomenological behaviour under irradiation was investigated. The results achieved for mineral pigments embedded in a modern binder (hydroxypropylcellulose) will presented in the following. Thereby the investigation of the influence of (LED) light as a main part of the optical radiation wavelength range is a focal point.

2. State of the Art

At first it is necessary to differentiate the terms light and optical radiation. The definition of light is the for human eyes visible wavelength range from 380 nm to 780 nm. Added the ultraviolet radiation (100 nm - 380 nm) and near infrared radiation from 780 nm up to 1000 nm, this whole wavelength range is called optical radiation. Standardly when light or optical radiation stability of textiles, coating or synthetic material is examined testing devices like the so called Xenontest (Atlas GmbH) are used with the intension to accelerate the irradiation process by using very high intensity [1]. This testing methods contain the assumption that high intensity in short time period leads to the same result than low intensity does in a long time period [2]. This correlation is named reciprocity law. As the approximately validity of this law is only investigated for organic materials and high intensity irradiation as created by xenon vapour lamps the application of this law has to be proven for inorganic pigments under artificial irradiation which acts usually in the low intensity region. As own previous studies inter alia lead to the result, that earth pigments are extremely stable against optical irradiation in contrast to mineral pigments which show very different behavior, the focus of the following study lies on the investigation of mineral pigments [3, 4]. The urgent need of verification of the described topic is that valuable coloured objects are predominantly stored, preserved and presented in indoor rooms with artificial light environment.

The first publication of an overview concerning the optical radiation stability of chromophoric substances was made by Thomson in 1986 [5]. Already there, although being a very simple and not specific categorization, it becomes obvious that mineral pigments are not easily to classify and tend to vary in their stability between the generally very stable earth pigments and the only little resistant organic dyes. Thomsons overview lacks the inclusion of the parameter of the pigments environment, i. e. binder as well as the parameters of the specific irradiation. Following studies tried to overcome these problems by either regarding only a single parameter or by generalizing whole groups of materials [6]. Today the so called *blue wool scale* is a standard tool for indicating optical radiation stability especially in the textile industry but this scale is also used in other fields like conservation [1]. The problem of the categorization after the blue wool scale is that the evaluation of colour changes is based on human eye determination. As this is a very subjective and thus unsatisfying evaluation it is absolutely necessary to develop a new classification system based on physical measurable values. An introduction to this topic is the task of the following presented part of our studies.

3. Phenomenological Description of Colour Change Induced by Optical Irradiation

A very good and common phenomenological parameter to describe the damage induced by optical irradiation is the colour change. The colour change which is determined by default with the so called colour distance ΔE , is useful as it is often the first visible reaction of a colored object to optical radiation exposure. The colour distance is defined for the

three-dimensional CIEL*a*b* colour space. This Euclidean space consists of a two-dimensional colour field a*b* and a third dimension for brightness L*. The basic function to describe the colour distance between two different colour coordinates P_1 (L*₁, a*₁, b*₁) and P_2 (L*₂, a*₂, b*₂) shows the following CIE-Lab formula [6].



Figure 1. Three-dimensional colour space of the CIE-Lab colour system [7].

Experiments with test persons proved that the colour distances achieved with this formula could not display the perceived colour distance. So the basic function was evolved for better fitting to human sense. To the present day there exist several functions which work for a determined case like low value of colour distance or only red colored objects. But there is no common method to use for all issues. As this problem further on exists and as for this study primarily a comparable physical value for colour changing is needed, the colour distance is calculated with the original CIE-Lab function.

After CIE 157:2004 the colour distance follows in first approximation the function (Figure 2)

$$\Delta E = m \cdot (1 - e^{-n \cdot H_{dm}}) \tag{2}$$

where by Hdm is called the damaging irradiation and is generally the product of irradiance and exposure time [9].

$$H_{dm} = \int_{t_0}^{t_1} E_{dm} \cdot dt \tag{3}$$

Until today damaging irradiance inducing light damage on painted objects is calculated with the by *Krochmann* and *Aydinli* introduced exponential function including a material-specific parameter b [10, 2, 11].

$$E_{dm} = \int_{380}^{780} E_{e,\lambda} \cdot s(\lambda)_{dm,rel} \, d\lambda = \int_{380}^{780} E_{e,\lambda} \cdot e^{-b(\lambda - 300nm)} \, d\lambda \tag{4}$$

For the purpose of investigation of mineral pigments this equation has to be questioned because it has only be proven to be applicable for dyes and some paper materials [2]. As we don't know the function of the spectral radiation stability of mineral pigments and for determination of the damaging wavelength range of affecting irradiation it was used the energy density instead of damaging irradiation. The energy density is the product of intensity (or irradiance) of the whole irradiating wavelength range and the exposure time.

$$\omega = \int_{\lambda_{start}}^{\lambda_{end}} E_{e,\lambda} \, d\lambda \cdot t = E_e \cdot t = I \cdot t \tag{5}$$

energy density	ω in Wh/m ²
exposure time	t in h
intensity	I in W/m ²
irradiance	E_e in W/m ²

The equation for the damaging irradiation (equation 4) is known as the already mentioned reciprocity law which means that comparable irradiation values lead to same degree of colour change. So whether low intensity and high exposure time or high intensity and low exposure time are given the same effect is achieved. This relation is generally assumed to be valid for the influence of optical radiation on colored objects [2]. To verify this postulated relation between energy density and resulting damage for artificial irradiation (with low intensity) own preliminary studies were conducted. The investigations were realized by comparison experiments with a xenon vapour lamp simulating sun irradiation on earth with a relatively high intensity (approximately 500 W/m²) and standard artificial light, mainly LED light, which is used in interior spaces.

The investigation of the reciprocity law in the field of optical radiation damage on mineral pigments in interiors reveals two principal results (Figure 3). At first the effects resulting from low intensity (<< 100 W/m²) are not comparable to these from high intensity (>> 100 W/m²) irradiation. Secondly it can be proved that the reciprocity law is not valid in the field of low intensity optical radiation. No solid relation between colour distance and affecting energy density could be found. Therefore, all results achieved in this study are exclusively valid for artificial optical radiation with low intensity.



Figure 2. Curve of colour distance in dependence of exposure time after [8] with positions of appearance of significant damage $x_{th}(t_{th}) = 4$ and the beginning of equilibrium state $\Delta E_{max}(t_{max})$.



Figure 3. (left) Smoothed curve of colour distance for red lead in 4%-hydroxypropylcellulose exposed to high and low daylight irradiation. (right) Smoothed Curve of colour distance for ultramarine blue in 4%-hydroxypropylcellulose exposed to standardized daylight and cold-white LED irradiation with different intensities.

To consider the degree of optical radiation damage in respect to colour changing of pigment, in this study a new system of categorization was developed. Based on the whole achieved data material it was possible to determine a threshold above which a significant damage is recognizable. This empirically founded threshold x_{th} is determined at a colour distance of $\Delta E = 4$. All values equal or above that threshold are categorized as a significant damage on the pigment. Values underneath $\Delta E = 2$ cannot counted to damage, as the errors of the measurement device or its application can accumulate to that size. Is there a colour change over $\Delta E = 40$, the damage can be classified as extremely high. Overall, there are eight categories, from zero to seven to describe the optical radiation stability (ORS) of a non-organic pigment sample, evaluating the maximal colour distance ΔE_{max} when equilibrium state is attained. Before starting the evaluation of the measured curve of colour distance, it is necessary to compare this curve with the obtained curve for not-exposed samples in the same time period. If the curve of colour distance for not-exposed samples lies under a threshold of $\Delta E = 2$ (no damage) no colour change is observed under dark condition and the developed categorization system can be applied without difficulty on the measured values for irradiated samples (Table 1).

Table 1. Developed basic categorization system of maximal optical radiation damage to determine an optical radiation stability (ORS) of mineral pigments.

maximal colour distance	description	optical radiation stability (ORS)	
< 2	no damage	7	
< 4	no significant damage damage	6	
< 6	low damage	5	
< 10	medium damage	4	
< 20	high damage	3	
< 30	strong damage	2	
< 40	very strong damage	1	
\geq 40	extreme damage	0	

4. Experiments

4.1. Experimental Setup

In principal two different kinds of samples were produced. Pure pigment samples without binder for reference purpose and samples with pigments embedded in cellulose binder. This binder was chosen because it possesses lots of advantages, i. e. it is a low cost product, simple to handle and non-poisonous. Moreover, this binder material already entered in the conservation and restauration business but hasn't been investigated in that context yet. Representatively a hydroxypropylcellulose (hpc) C_3H_7O with low viscosity (< 150 mPas) in a concentration of 4% was used for sample production.



Figure 4. Experimental setup in a black box with different layers of irradiation settings at Interdisciplinary Research Center of Material Science, Martin - Luther - University, Halle/Saale, Germany.

The finished samples were put in a particularly built light chamber which is completely matt black coloured inside. In this chamber the artificial lamps are installed in separate layers. In a determined distance to the specific lamps, the samples are horizontally arranged and continuously irradiated. Thereby in principle two different types of lamps are used. On the one hand LEDs with different wavelength range were installed covering the whole light wavelength range from 380 nm to 780 nm (Figure 5). Among these LEDs the damaging potential of cold-white LEDs was of special interest. For comparison a D65 standardized daylight lamp (justdaylight) representing the type of fluorescent light with a discrete spectral distribution and a colour temperature similar to sun light was investigated. In contrast to the cold-white LED the D65 daylight lamp possesses a full spectrum including UVand IR-radiation from 300 nm up to 1000 nm. This is the maximal spectral range which is usually produced by indoor artificial radiation. The irradiation experiments were run under clean room conditions (classification ISO 7) and steady climate with relative humidity of 37% and room temperature of 22°C [12].

Table 2. Overview of investigated lamps and their radiometric (photometric) properties measured with the spectroradiometer specbos 1211 UV (JETI).

illuminants	spectral range in nm	Intensity in W/m ²	colour rendering index Ra	colour temperature CCT in K
D65 standardized daylight lamp	300 - 1000	18 / 36	96	6324
LED cold-white	410 - 780	0,5 / 6	80	6159
LED blue	420 - 530	1,5 / 4,5		
LED violet/blue	380 - 740	0,5	73	6269





Figure 5. (above) Covering wavelength range in the visible wavelength region and (below) the spectral distribution of applied LED lamps.

4.2. Experimental Procedure

Each single irradiation experiments last non-stop over a period of about 14,000 hours. The measurement of the colour change was conducted in defined intervals of time which

increase with increasing irradiation period as the pigment reaction on irradiation is highest in the beginning. In the end, for each sample-irradiation-setting a characteristic curve of colour change was obtained. Including the information whether a significant damage occurs or not and the information of the maximal value of colour change when the state of equilibrium was reached (no further colour change). As it was figured out that the value of intensity in the low intensity field is in first approach negligible, all curves of colour change were plotted in dependence of exposure time. Consequently, the achieved information of a specific pigment depends on the spectral function of impacting radiation (i.e. cold - white LED) and the exposure time. For reference purpose, accordingly experiments were run with same climate - temperature and relative humidity - conditions but no irradiation. Comparing the curves of colour change for irradiated and for not-exposed samples, optical radiation influence is significantly evident.

Are there complete colour distance curves available, the optical radiation stability can be determined. If possible the damaging wavelength range was questioned. Therefore, a comparison method was developed to achieve qualitative results. For example, two different irradiation experiments, e I and e II were conducted. Each result is evaluated by applying the threshold $x_{th} = 4$ so that damage or no damage can be stated. The comparison of both results leads to the confinement of the damaging wavelength range. Repeating this procedure several times by comparing results of different experiments with each other, makes a determination of the damaging wavelength more exactly.



Figure 6. Principal method of experimental procedure by evaluation and comparing of measurement results.

5. Results

5.1. Stability of Cellulose Binder

Actually no colour change is expected for pure cellulose binder under optical irradiation as yellowing of materials containing cellulose or wood is due to its lignin content [13]. The results of this study show a very high light stability (ORS = 5) for hydroxypropylcellulose under irradiation with all examined light sources except for the so called violet/blue LED light possessing a wavelength range from 380 nm to 740 nm (Figure 7, above). As for blue LED light (420 nm – 530 nm) and cold-white LED light (410 nm – 780 nm) no damage occurs, the damaging wavelengths of LED light can be determined to a range from 380 nm to 410 nm.





Figure 7. (above) Smoothed curve of colour distance for 4%hydroxypropylcellulose and (below) in dependence of exposure time and (right) in dependence of energy density in the range from 380 nm to 410 nm.

A second valuable result which can be confirmed by the results for pigments in 4%-hydroxypropylcellulose is that the damage primarily depends on the spectral function of the acting irradiation. This effect becomes clearly for the damaging wavelength range of the violet/blue LED which is actually also present in the spectrum of D65 standardized daylight but does not lead to same colour change for the different irradiation sources. For illustration of this effect the curve of colour distance is plotted in dependence of energy density in the damaging range from 380 nm to 410 nm (Figure 7, below).

5.2. Stability of Mineral Pigments in Cellulose Binder

Except the pigment Red Lead which experienced also a significant colour change without irradiation (Figure 8), all investigated non-exposed samples show a curve of colour distance which lies beyond the threshold of $\Delta E = 2$ during the whole experimental period of 14,000 hours. ySo the complete detected colour change of the samples can be lead back solely to the influence of optical radiation.



Figure 8. Smoothed curve of colour distance for red lead in 4%hydroxypropylcellulose. Several effects are visible: invalidity of reciprocity law (compare curve for LED cold-white $I = 0,5 W/m^2$ and $I = 0,5 W/m^2$), unusual exalted colour change for non-exposed samples and significant high gradient of curve for LED violet/blue irradiation.

Basically this study of mineral pigments in cellulose binder reveals two main results. The first one is that green and blue pigments tend to experience less optical radiation damage than yellow to red ones do (Figure 9). So the most stable pigments were azurite and smalte (ORS = 7, for D65 standardized daylight as well as for cold-white LED), whereas the arsenic sulphide pigments like auripigment and realgar own the lowest optical radiation stability of all investigated pigments (see also [14]). Secondly the damaging effect of the D65 standardized daylight with full optical spectrum is generally higher than for cold-white LED with namely similar colour temperature but only radiation in the visible spectrum from 410 nm to 780 nm. Further significant damage ($\Delta E \ge x_{th}$) as well as the equilibrium state in colour change is achieved much faster by using the D65 standardized daylight (Table 4).



Figure 9. Light stability of investigated mineral pigments in 4%-hydroxypropylcellulose for irradiation with standardized D65 daylight ($I = 18 \text{ W/m}^2$) and for irradiation with cold-white LEDs ($I = 6 \text{ W/m}^2$).

Counting the colour changing effect of infrared radiation (radiation above 780 nm) as negligible it is assumed that the higher values of colour distance for the D65 standardized daylight are either caused by wavelengths beyond 410 nm or are issued by different spectral distributions as seen for the hydroxypropylcellulose.

For determining a possible blue light effect from 380 nm to 410 nm, experiments with LEDs of different wavelength ranges were conducted (Figure 5). For pigments with an optical radiation stability of at least ORS \leq 3 damaging light

regions could be found. Thereby the damaging wavelength ranges turned out to be very different and not even comparable for pigments of similar chemical composition.

The results also show that violet or blue light is not damaging generally and other colours of the visible radiation are equal in their damaging potential. Furthermore, there is no clear correlation between absorbed light and damaging light observable. Again an illuminant depending damage potential - like seen for pure 4%-hydroxypropylcellulose was detected.

Table 4. Time of exposure in hours for pigments in 4%-hydroxypropylcellulose with standardized daylight ($I = 18 \text{ W/m}^2$) and cold-white LED ($I = 6 \text{ W/m}^2$) until a significant damage ($x_{th} = 4$) or an equilibrium state (ΔE_{max}) is reached.

pigments in 4%-	exposure time for significant damage in h		exposure time for equilibrium state in h	
hydroxypropylcellulose	D65 standardized daylight	cold-white LED	D65 standardized daylight	cold-white LED
azurite	no significant damage	no significant damage	500	500
malachite	> 14,000	no significant damage	> 14,000	1,000
cobalt blue	3,500	12,000	20,000	> 14,000
smalte	no significant damage	no significant damage	11,000	11,000
ultramarine blue	500	no significant damage	> 14,000	4,000
ultramarine red	no significant damage	no significant damage	3,000	2,000
lapis lazuli	4,500	14,000	> 14,000	> 14,000
auripigment	1,000	2,000	10,000	> 14,000
realgar	48	48	1,000	> 14,000
tin-lead yellow I	500	1,000	7,000	14,000
red lead	100	1.000	2,000	> 14,000
vermilion	3,000	12,000	> 14,000	12,000

6. Discussion

The here presented categorization system (Table 1) serves as a basic classification. Current work is to enlarge this categorization system by including the exposure time needed to reach a significant damage into the evaluation process. Further experiments are run to investigate pure pigment powder whereby initially an adequate method for sample preparation has to be found. Achieving the results for pure pigments it will be possible to determine the influence of (cellulose) binder on the spectral radiation stability of the specific pigment by comparative analysis. At last, applying the Raman spectroscopy at defined exposure intervals in where a significant damage occurs efforts are in progress to correlate measured colour change to changes in molecular vibration modes. Thereby the ability of Raman spectroscopy to prove a chemical reason for colour change will be possible. Conceivably further spectroscopic method must be used for a sufficient explanation of the chemical reasons.

7. Conclusion

First of all, it should be noted that the developed categorization system could be successfully applied for all investigated pigments. Apart from the pigment Red Lead significant colour change can be correlated distinctly to the influence of irradiation. Concerning the influence of optical radiation on mineral pigments two very important facts have been revealed which force to revise assumed facts. On the one hand the validity of the reciprocity law in the low intensity field as normally present for artificial radiation could be falsified for mineral pigments. This is of great importance as until now, in all fields of research and their implementation reaching from biomedicine to conservation technics, the reciprocity law is assumed uncritically. But our results show that a more profound investigation of the relation between irradiation and exposure time and their issued effects need to be done.

Secondly it turns out that the effect of damaging wavelengths depends on the whole impacting spectral function by means of the existence as well as the distribution of the entire wavelength spectrum. Analog to the behavior of acoustic waves the kind of superposition of spectral wavelengths effects on their damaging impact. Perchance a reasonable chosen spectral function could lead to a significant reduction of colour change caused by potential damaging wavelengths.

The experimental results have been shown that blue and green mineral pigments tend to be more stable against the influence of optical radiation, especially to light, than red to yellow pigments are. Further it has to be considered that a higher damage in less time is realized for irradiation with D65 standardized daylight in contrast to irradiation with cold-white LEDs. Nevertheless, it firmly needs to be pointed out that LED light causes significant damage on the majority of the investigated pigments. This aspect is remarkable regarding the increasing application of LED technology. Until today even leading light designers for museums and galleries consider the damaging effect to mainly UV radiation and assume an exponential decreasing damaging effect with increasing wavelength (formula 4). However, this study can state that damaging effects occur throughout the entire light spectrum depending on every single painting media (pigment - binder - combination). There is no clear relation between (absorbed) wavelengths and the degree of colour change at least for mineral pigments. Consequently, it is generally not possible to avoid a colour change by using LED light, even not by using additional blue light filters.

These scientific insights have to be taken into account in the work of conservators and exceedingly concerns the field of preservation of culture goods. Only the knowledge of the material composition and the specific information of its optical irradiation behaviour leads to a reliable object protection.

8. Outlook

Future studies should have the focus on the problem of the influence of the spectral distribution. Moreover, additional binders should be investigated to allow a classification of cellulose binder respectively hydroxypropylcellulose concerning its optical radiation stability in the context of painting material and to reveal possible advantages of the cellulose. The palette of mineral pigments also has to be extended to accomplish a general understanding of pigments behaviour under the influence of optical and especially light irradiation. Moreover, other climate parameter as there are temperature and relative humidity should be taken into account in respect of irradiation effects. At least the whole gained data matrix is considered to lead to a detailed handbook which finds application in the specific field of restauration. Besides conservation and these phenomenological considerations it is of great interest to understand the underlying processes may they be chemical or physical. First steps are already done with our running studies.

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