THE BEHAVIOUR OF *SCHLAGMETAL* APPLIED ON DIFFERENT TYPES OF GILDING SIZES

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O COMPORTAMENTO DO *SHLAGMETAL* APLICADO A DIFERENTES TIPOS DE MORDENTES

Resumo

O *schlagmetal* (ouro holandês), obtido a partir de uma liga de cobre e zinco é uma alternativa barata à folha de ouro, mas sua cor muda devido a oxidações que aparecem como resultado de manipulações incorrectas ou de exposição a fatores ambientais (temperatura, umidade). Este artigo apresenta a influência da película de proteção à base de goma-laca e de dois aglutinantes (à base de óleo e água) no *schlagmeta*l. aplicado sobre um suporte de madeira com preparação de gesso. Para avaliar o comportamento da folha de metal nas estruturas apresentadas no presente estudo, as amostras foram expostas a envelhecimento artificial e as alterações registadas foram analisadas por microscopia óptica (MO), SEM-EDX e colorimetria em sistema CIE L*a*b*.

Palavras-chave

Schlagmetal, mordente, envelhecimento artificial, OM, SEM-EDX, Colorimetria em sistema CIE L*a*b*.

EL COMPORTAMIENTO DEL *SCHLAGMETAL* APLICADO A DISTINTOS TIPOS DE MORDIENTES.

Resumen

El *schlagmetal* (Oro holandés) , obtenido a partir de una aleación de cobre y zinc es una alternativa barata a la hoja de oro , pero su color cambia debido a la oxidación que aparece a consecuencia de una manipulación incorrecta o de la exposición a factores ambientales (temperatura, humedad). En este artículo se estudia la influencia de la película de protección a base de goma laca y de dos aglutinantes (a base de agua y aceite) en el *schlagmetal*, aplicada sobre un soporte de madera con preparación de yeso.

Para evaluar el comportamiento de la hoja metálica en las estructuras que se presentan en este estudio, se expusieron las muestras a un envejecimiento artificial y los cambios

registrados se analizaron por microscopía óptica (MO), SEM-EDX, por y colorimetría en sistema CIE L*a*b*.

Palabras clave

Schlagmetal, mordiente, envejecimiento artificial, MO, MEV-EDX, colorimetría en sistema CIE L*a*b*.

Abstract

The *schlagmetal* (Dutch Gold), obtained from an alloy of copper and zinc is a cheap alternative for gold leaf, but its colour changes due to oxidations which appear as a result of incorrect manipulations or of exposure to environmental factors (temperature, humidity). This paper presents the influence of shellac based protection film and of two mixtion binders (water and oil based size) on the schlagmetal applied on a gessoed wooden support. To evaluate the behaviour of the metal sheet in the structures presented in this study, the samples were exposed to artificial ageing, and the occurred changes were analyzed by optical microscopy (OM), SEM-EDX and colorimetry in CIE L*a*b* system.

Keywords

Schlagmetal, gilding size, artificial aging, OM, SEM-EDX, colorimetry CIE L*a*b*.

Introduction

Schlagmetal or Dutch Gold has reduced considerably the price of gilding technique. This 19th century innovation is based on a copper and zinc alloy that has similar properties to gold leaf. *Schlagmetal* is an advantage for artists because is easier to manipulate, as is thicker than the gold leaf. Nowadays is frequently used to decorate new art objects and redecorate old ones: altars, sculptures, furniture ornaments, cassettes, frames, and easel and mural paintings (Jenkins, 2007; Sandu et al, 2011:1171-1181).

Among the gilding techniques, the gilding with oil based sizes was taken and adapted to *schlagmetal* (Bigelow and Hutchins, 1991; Guerra, 2008:317-324; Oddy, 1981:75-79; Sandu et al, 2010:47-63; Sandu et al, 2011:1171-1181).Thus, one of the modern materials used in *schlagmetal* gilding was „mixtion" (mordant gilding size), a viscous lake that contains linseed oil, litharge (PbO) and turpentine, and can be used on different supports like stone, metal, textiles or wood (Sardela, 2008:86).

There is a large range of gilding sizes on the market nowadays, based on linseed oil, natural resins or polymers (Andreotti et al, 2014:300-307; Budu et al, 2015:1212-1216; Hutanu et al, 2015:895-900; de la Torre-Lopez et al, 2014:1052-1058; Rydlova and Kopecka, 2012:250-257; Sandu et al, 2011:1171-1181).

Because the gilding process leads to the formation of a stratified, sandwich-like structure, in which gilding metal sheet must be compatible with contact components its behavior in time was studied using artificial ageing. For this purpose, the involved structures are exposed to harsh environmental conditions, which lead to the alteration or micro-structural destruction of the components. Their evolution can be determined by certain chemometric characteristics (Sandu et al, 2000:532-542; Sandu et al, 2001:485-490).

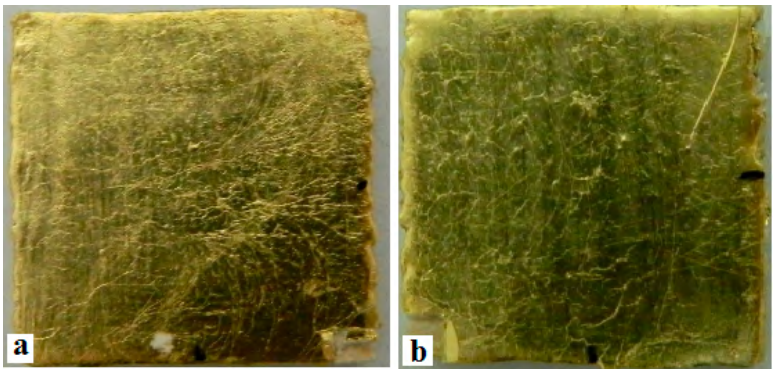
Depending on the materials' nature (organic or inorganic), the artificial ageing uses high temperature (60°-110°C) and high relative humidity (85-99% RH) conditions or UV exposure for short periods of time, equivalent to natural ageing during long time spans (Sandu et al, 2000:532-542; Sandu et al, 2001:485-490).

Excepting UV irradiation, which is very aggressive, with irreversible and un-controlled effects, the other procedures are moderated, and their effects can be easily stopped. At high temperatures there are dehydration processes which lead to structural modifications, redox, acid-base, and complexation reactions, depending on the nature of the component materials. Similarly, the atmospheres with high hygroscopic humidity affect especially the hydrophilic organic materials. Thus, at humidity higher than 85%, the films or the thin layers are transformed from un-membrane or semi-membrane systems in swollen membranes which allow air oxygen diffusion. The oxygen interacts at interface level, leading to chemical reactions and degradation of component materials (Sandu et al, 2000:532-542; Sandu et al, 2001:485-490).

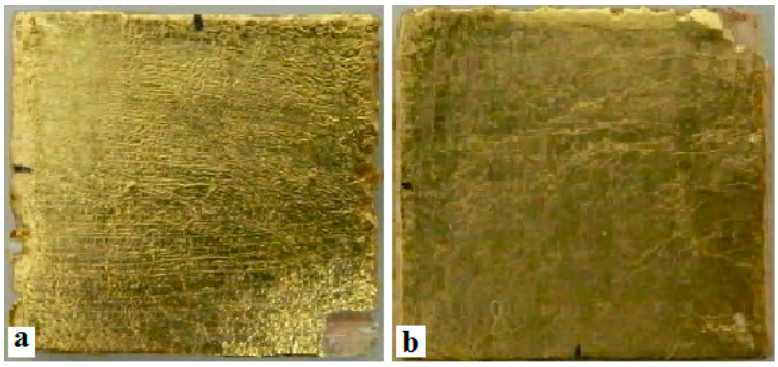
This paper presents the influence of shellac based protection film and of two "mixtion" binders on the *schlagmetal* applied on a gessoed wooden support, using artificial ageing with two different conditions: high temperature (65°C) and hygroscopic humidity (90%).

Materials and techniques

The samples were made of Balsa wood (20.5 mm x 20.5 mm x 5mm), following a traditional recipe of mordant gilding. Thus, a few layers of ground made of $CaCO_3$ and hide glue 10% were brushed on the surface of the wood samples. After drying the ground was polished with sand paper and isolated with shellac 20%. Drops of gilding sizes were spread by brush on samples' surface, and let to dry, respecting the time recommended by the manufacturer. The metal sheet was applied and isolated with shellac 20%. 2 types of "mixtion", with different compositions, produced by Masserini S.r.l and Maimeri were used. They were noted as follows: water based "mixtion" – M-H_2O, and the linseed oil based "mixtion", with a 12 hours drying time, was noted M-12h. 2 samples were used for each type of "mixtion". The gilding sizes and shellac were applied by brush in a thin layer, respecting the drying time. The 4 samples were exposed to artificial ageing. Two of them were placed in a thermoregulation oven AIR Concept (FIRLABO) type at 65°C, for 192 hours (the equivalent of 6 months ageing at 20°C), while the other two were exposed to extreme humidity (90% RH) also for 192 hours, using an air-tight humidifier. The evolution of ageing effects were determined by means of CIE L*a*b* colorimetry, every 48 hours.



M-H_2O samples



M-12h samples

Figure 1 – The samples, coated with shellac, before the artificial ageing at a temperature of 65° C (a) and 90% RH (b).

The samples morphology was analyzed using a reflection microscope CARL ZEISS AXIO IMAGER A1m, having attached a photocamera AXIOCAM. Also, for the analysis of elemental composition we used a scanning electron microscope, model VEGA II LSH, produced by TESCAN Czech Republic, coupled with a spectrometer QUANTAX QX2, made by BRULER/ PROENTEC Germany. *Schlagmetal*'s colour changes were analyzed every 48 hours with a spectrophotometer Lovibond RT 300. The color change was calculated for each color coordinate (L*, a* and b*) as related to its initial value on the same sample and the same point. Finally, the total color change (D*E**) was calculated in each point (Atodiresei et al, 2013:165-169; Cristache et al, 2015:348-352; Daher et al, 2014:1207-1214; Le Gac et al, 2013:242-257; Saviuc-Paval et al, 2012:170-178 and 2013:564-571; Sandu et al, 1999:902-908; 2007:879-886 and 2010:752-760; Schanda, 2007) using the equation

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

where D*L** is the luminosity change in the respective point at different time intervals, by artificial aging, comparative to initial value:

$$\Delta L^* = L^*_{s.aged} - L^*_{initial}$$

D*a** is the change of the red (+a*) – green (-a*) coordinate in the respective point and at different time intervals by artificial aging, compared to initial value

$$\Delta a^* = a^*_{s.aged} - a^*_{initial}$$

and D*b** is the change of the yellow (+b*) – blue (-b*) coordinate in the respective point and at different time intervals, by artificial aging, compared to initial value

$$\Delta b^* = b^*_{s.aged} - b^*_{initial}$$

s.aged – aged samples ; *initial – initial samples.*

Results and discussions

Initially the elemental compositions of the 2 types of "mixtion" were determined by means of scanning electron microscopy, coupled with X-Ray spectrometry, SEM-EDX (table 1).

Table 1. The elemental composition of the "mixtions" used

Element (norm.wt%) / Mixtion	S	C	O
M-H_2O	0.664887	27.81474	71.52037
M-12h	-	29.57225	70.42775

The sulfur present in M- H_2O composition was used to enhance "mixtion" adhesive properties (Voitovich, 2010:133-136). The high quantity of carbon and oxygen is a result of organic composition of the gilding sizes and of graphite conductive adhesive, used in microscopy.

The alcoholic solution of shellac 20% used to coat the ground layer and *schlagmetal* was made of ethanol and refined natural resin, which contains 95% resin, 0.5% dyestuff (laccaic acid and eritrolaccin), 4% wax and 1.8% volatile compounds. The resin contains sesquiterpenic acids pertaining to cedrene, mostly jalaric and laccijalaric acids, and hidroxy fatty acids, mainly threoaleuritic acid (Derry, 2012; Wang et al, 1999:1316-1322).

The 4 samples from figure 1 were studied by means of OM and SEM-EDX. Therefore in figures 2 and 3 are presented the microphotographs made using the two techniques.

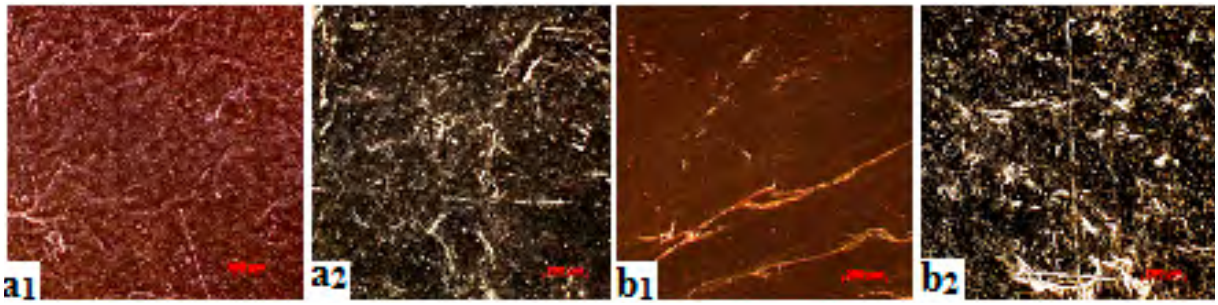


Figure 2 – Microphotographs of *schlagmetal* samples, with shellac and M-H_2O (OM-100x): a – before (1) and after (2) artificial ageing at 65°C,
b – before (1) and after (2) artificial ageing at 90% RH

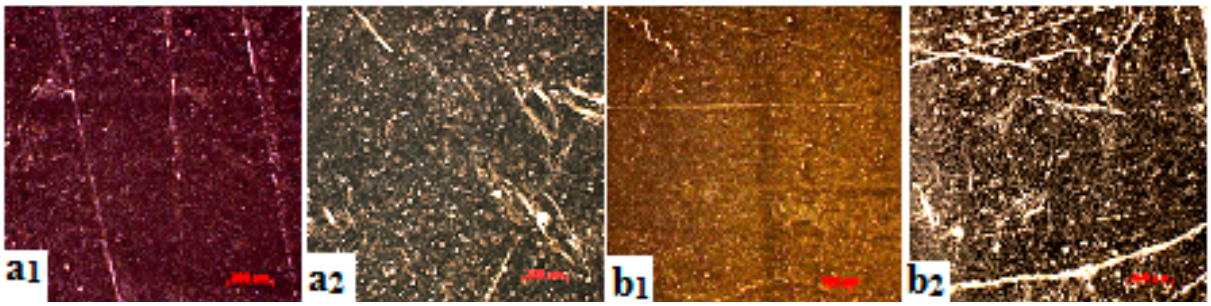


Figure 3 – Microphotographs of *schlagmetal* samples, with shellac and M-12h (OM-100x): a – before (1) and after (2) artificial ageing at 65°C,
b – before (1) and after (2) artificial ageing at 90% RH

The analysis by means of optical microscopy did not reveal notable changes of the *schlagmetal*'s surface between the shellac and "mixtion" films. The wrinkles and folds that can be observed on some samples' surface are a result of metal sheet spreading and irregular drying of "mixtion" layer.

In table 2 is presented the elemental composition of *schlagmetal*.

Table 2 – Elemental composition of *schlagmetal*

Element	[norm. wt.-%]	[norm. at.-%]	Error in %
Copper	31.2674	9.647535	1.002499
Zinc	5.280557	1.583365	0.218092
Carbon	27.05418	44.16396	4.138933
Oxygen	36.39786	44.60514	8.5215

The *schlagmetal* composition contains, beside brass' metals (Cu, Zn) a significant quantity of carbon and oxygen from the graphite conductive adhesive, used in microscopy.

In figures 4 and 5 the SEM microphotographs are presented for the 4 samples, before (1) and after (2) artificial ageing.

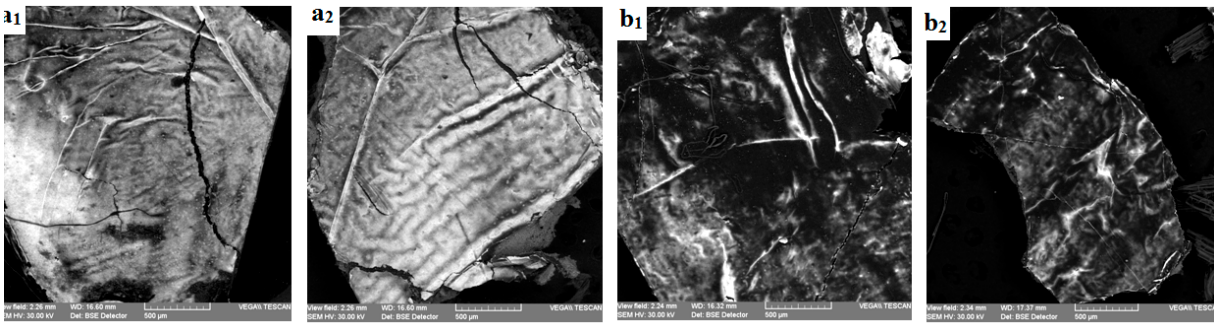


Figure 4 – Microphotographs of *schlagmetal* samples, with shellac and M-H_2O (bse 100x): a – before (1) and after (2) artificial ageing at 65°C, b – before (1) and after (2) artificial ageing in humid atmosphere (90% RH)

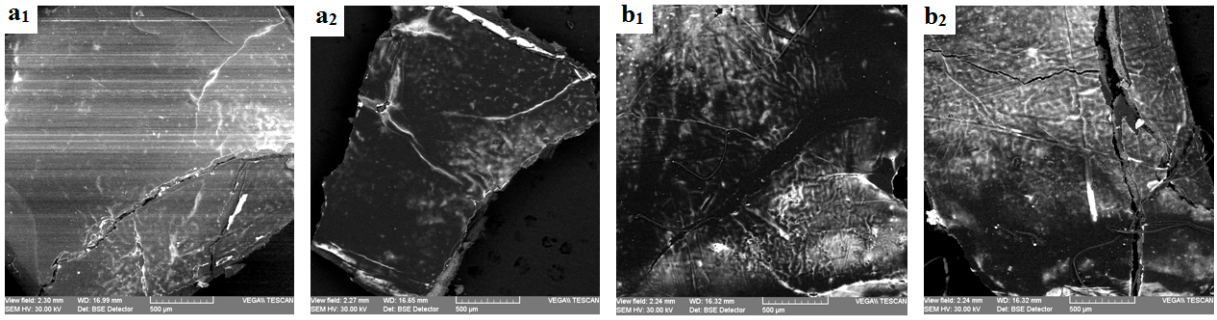


Figure 5 – Microphotographs of *schlagmetal* samples, with shellac and M-12h (bse 100x): a – before (1) and after (2) artificial ageing at 65°C, b – before (1) and after (2) artificial ageing in humid atmosphere (90% RH)

The L*, a*, b* values (Table 3) obtained by colorimetric analysis of the artificially aged samples at 65°C, were used to evaluate the colour differences ΔE* at different ageing stages, according to the above formulae (Fig. 6).

Table 3. L*, a*, b* values of *schlagmetal* samples after temperature exposure

time	CIEL*a*b*	M-H_2O	M-12h
t=0	L*	86.61	86.85
	a*	+9.77	+9.30
	b*	+35.50	+34.68
t= 48h	L*	87.03	87.59
	a*	+9.80	+9.47
	b*	+36.67	+35.61
t=96h	L*	87.44	88.40
	a*	+9.84	+9.60
	b*	+37.40	+36.97
t= 144h	L*	87.83	89.66
	a*	+9.93	+9.80
	b*	+37.83	+37.78
t=192h	L*	87.79	88.67
	a*	+10.01	+9.72
	b*	+38.17	+37.59

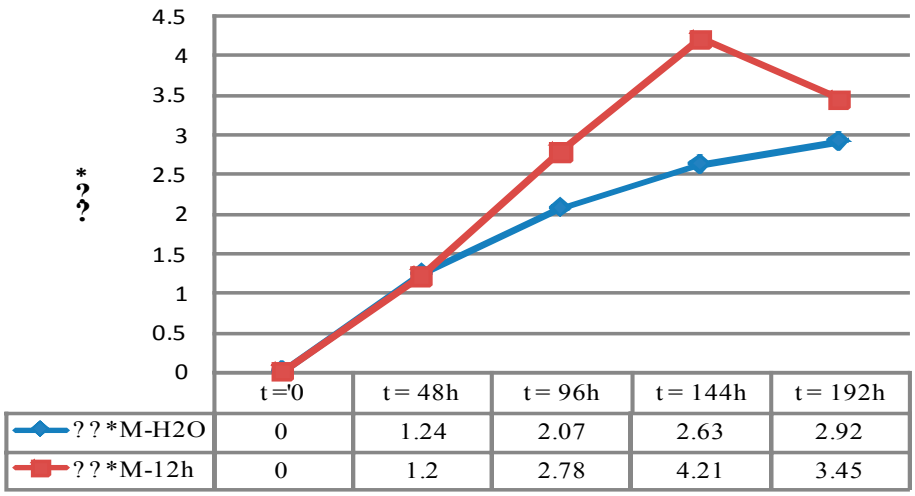


Figure 6 – The graphic for ΔE* colour differences of the two samples at different time spans during thermal aging

Analyzing the samples exposed to high temperature, we observed an increment of ΔE* values until t = 144h, the greatest variations occurring in the case of M-12h sample. After 144 hours the value of chromatic changes decrease only in the case of the M-12h sample, while for the M-H_2O sample slightly increases with a small slope. The biggest difference between ΔE* values at t = 144h appear in the case of M-12h, 4.21 units, while the M-H_2O sample has a variation of 2.63 units, for the same time span.

From figure 6 we can observe that both colorimetric curves change their inclination after 144 hours of constant heating, which indicates a modification in the discolouration ageing mechanism, due to dehydration and compaction of shellac, followed by an interaction with the surface cations from the *schlagmetal*, which makes it darker.

After the measurements in CIEL*a*b* system of the samples exposed to humidity (table 4) and ΔE* calculations it was noticed that the samples presented an increase of the values from t=0 to t=48h, which was greater in the case of M-H_2O (4.29 units compared to 0.78 units in the case of M-12h), followed by a decrease of ΔE* values from t = 48h to t = 96h, the highest variation appearing in the case of the M-H_2O sample. Between t = 96h and t = 144h the ΔE* values increase, while at 192 hours, the slight change of ageing mechanism lead to a significantly growth for the M-H_2O sample and constant values for M-12h.

Table 4. L*, a*, b* values of *schlagmetal* samples after humidity exposure

time	CIEL*a*b*	M-H_2O	M-12h
t=0h	L*	84.92	89.15
	a*	+9.60	+8.44
	b*	+35.67	+34.74
t=48h	L*	88.86	89.82
	a*	+9.98	+8.55
	b*	+37.33	+35.13

time	CIEL*a*b*	M-H_2O	M-12h
t=96h	L*	86.46	89.37
	a*	+9.84	+8.48
	b*	+36.51	+34.93
t= 144h	L*	86.54	89.56
	a*	+9.83	+8.48
	b*	36.76	+35.08
t= 192	L*	87.68	89.56
	a*	+9.96	+8.52
	b*	+36.93	+35.08

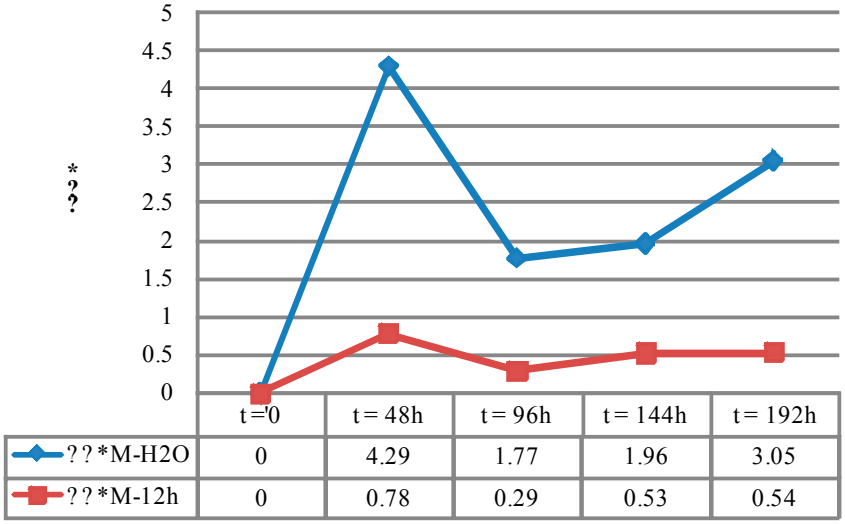


Figure 7 – The graphic for ΔE* colour differences of the two samples at different time spans during aging at high humidity

Conclusions

The samples with *schlagmetal* applied on water and oil based "mixtions" have suffered colour changes under the influence of temperature and humidity, the ΔE* variations being higher in the first case. The smallest variations determined by the temperature occurred in the case of M-H_2O sample, while the highest appeared in the M-12h sample. The situation changes for the samples exposed to humidity, the M-12h sample being the one with the smallest chromatic deviations, while the M-H_2O presents the highest differences. This data may help the artist/restorer to choose the right mixtion for gilding, also taking into account the microclimatic conditions for object's display.

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