Klaas Jan van den Berg · Aviva Burnstock · Matthijs de Keijzer · Jay Krueger
Tom Learner · Alberto de Tagle · Gunnar Heydenreich *Editors*

Issues in Contemporary Oil Paint

This volume represents 27 peer-reviewed papers presented at the ICOP 2013 symposium which will help conservators and curators recognise problems and interpret visual changes on paintings, which in turn give a more solid basis for decisions on the treatment of these paintings. The subject matter ranges from developments of paint technology, working methods of individual artists, through characterisation of paints and paint surfaces, paint degradation vs. long time stability, to observations of issues in collections, cleaning and other treatment issues as well as new conservation approaches.

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Foreword

It is a great pleasure to present the Proceedings of the Issues in Contemporary Oil Paint (ICOP) Symposium that was held in 28–29 March, 2013, in Amersfoort at the headquarters of the Cultural Heritage Agency of the Netherlands, RCE.

Our Agency is at the heart of cultural heritage in the Netherlands. Our research group is concerned with the evaluation and preservation of our heritage in the broadest sense and the research directions we need to follow in order to guarantee a sustainable heritage. Along with national and international research partners at museums, universities and archives, we conduct research, characterise materials and analyse change processes. The Agency ensures that third parties can apply the knowledge that we can provide.

The ICOP symposium was the first symposium focused on modern paints since the Modern Paints Uncovered (MPU) conference held at Tate in 2005. Whereas MPU mainly presented research on modern synthetic paints especially on acrylics, ICOP chose to focus on modern oil paints entirely. Many modern artists continue to work with oil paints, and modern oil paints increasingly become a challenge for conservators and collection keepers. Therefore it was felt by the organisers that it was time to organise a meeting which could discuss these challenges by presenting information on historical and artistic production, scientific research on degradation phenomena, and developing alternative conservation approaches.

ICOP marked the end of a 4-year Research Agenda, for our Agency.[1] In one of the programmes in the Research Agenda, *Object in Context,* the RCE research group generated knowledge on the production of and changes in heritage objects in their artistic, cultural and social contexts. Under the leadership of Klaas Jan van den Berg, the '20th century oil paint project' contributed to the outcome of the Agenda. The project brought many institutions together and was a breeding ground for

[1] Outcomes of this Research Agenda are accessible on-line: http://www.kennisvoorcollecties.nl/en/researchagenda/.

Sensitivity of Modern Oil Paints to Solvents. Effects on Synthetic Organic Pigments

Diana Blumenroth, Stefan Zumbühl, Nadim C. Scherrer, and Wolfgang Müller

Abstract Synthetic organic pigments are widely used in contemporary artists- and house paints. They can be found in artworks since 1900. Because of their special particle properties and their solubility in solvents, however, synthetic organic pigments pose a special challenge in conservation treatments. Analyses have been carried out on 23 synthetic organic pigments in oil paint films with six representative solvents. Solubility of the pigments upon solvent exposure has been determined by UV-Vis-spectroscopy. Some pigments have shown high solubility and were even extracted out of the oil paint film. Detailed examination on the influence of pigment extractions from the oil paint film was carried out with light microscopy, Raman- and ATR-FTIR spectroscopy. Swab tests elucidated that all pigments are very sensitive to the combination of (mostly) polar solvents and mechanical stress. The study demonstrates that mechanically applying solvents to paint surfaces containing synthetic organic pigments is delicate due to (a) the solubility of the pigments themselves, (b) mechanical removal of pigment particles – likely supported by the temporary destabilization of the binder. These findings have important implications to conservation practice.

Keywords Synthetic organic pigments • Solvents • PY 3 • Oil paint • Solubility • Cleaning • Raman • UV-Vis spectroscopy • ATR-FTIR

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Introduction

The conservation and restoration of modern art works show a new complexity of performance and sensitivity as a result of the huge variety of the materials used. Modern oil paints cannot simply be compared with "classic" oil paints, because of their different composition and thus different physical and chemical properties. An important part of this new characteristic is the wide use of synthetic organic pigments. The development of these pigments started already in the middle of the nineteenth century (Heumann and Friedänder 1898; Hübner 2006; Zerr and Rübenkamp 1921) and they can be present in works of art since the last quarter of the nineteenth century. Many new pigments have acquired a central role in paints due to their light fastness and high color intensity. The on-going development in this field has recently led to a huge variety of new products (Faulkner 2009; Smith 2002). Inorganic pigments are increasingly being displaced by synthetic organic pigments and they are now the most important group of pigments in the bright hues of modern artist's paints (Scherrer 2009). The interest in the properties of synthetic organic pigments has increased significantly in recent years, both in the historical (de Keijzer 1988, 1989, 1999, 2002; Frowein 2004; Fritsch 2006) and technological context (Lomax 2006; Lutzenberger 2009; Strauss 1984), as well as in relation to the specific requirements of the material analysis (Strauss 1984; Vandenabeele 2000; Kalsbeek 2005; Schäning 2005; Fritsch 2006; Lomax 2006; Ropret 2008; Schulte 2008; Frowein 2004; Scherrer 2009; Fremout 2012). As versatile as the applications of these pigments are, there also are many associated risks. New demands are therefore arising continuously with respect to the conservation of modern artists' paints, made with synthetic organic pigments. Some synthetic organic pigments found in "classic modernism" paintings of the early twentieth century, proved to be highly soluble (Zumbühl 2008). As a consequence, the specific particle properties as well as the sensitivity to solvents pose a particular challenge in conservation treatments of modern oil paints. In this context, the solvent sensitivity of 23 synthetic organic pigments in oil paints was tested under the various aspects (Blumenroth 2010).

Solvent Sensitivity of Organic Pigments and Their Modifications

Synthetic organic pigments consist of low molecular weight compounds (M <2,000 Da). In theory, a good solvatisation and solubility of these pigments in organic solvents can be predicted. The solvent sensitivity of these pigments is not evident in their chemical structure, as the properties are altered by selective modifications of the structure and encapsulation. Surface modifications are possible by physical or chemical adsorption of organic and metal-organic compounds or by in-situ polymerization of coating systems (Fu 2007; Lelu 2003; Sirikittikul 2004; Viala 2002). Resins have long been used for modifying the pigment properties, e.g. through a process such as rosination (Schröder 1988). In this case, the resin acts as a growth inhibitor and is used to control the crystallization process. The cohesion of a pigment is reduced by coating the pigment surface and therefore improving its dispersibility (Schröder 1988; Sirikittikul 2004). An overview on the various derivatization types has not been attempted, as each class of pigments has another application system (Bugnon 1995). Next to organic additives, other types of coatings (encapsulation) are industrially possible. The stability of organic pigments can be achieved and optimized by the application of nano-layers of silicon dioxide (Yuan et al. 2005, 2008) or other metal oxides (Binkowski et al. 2000; Bugnon 1995; Tiarks 2001; Yuan et al. 2006). Through such inorganic coatings, the thermal stability, wettability, dispersibility, chemical stability and light and weather fastness are improved (Yuan et al. 2006, 2008). In order to ensure good processing properties, such encapsulated pigments – similar to inorganic pigments – can be polymerized with organic coupling agents (Tiarks 2001). Accordingly, the pigment properties will vary considerably depending on the surface modifications. Sometimes the modification of one property can lead to a decline of another. No general statement can be made, as the pigment processing can be material specific and vary from manufacturer to manufacturer. Accordingly, it is difficult to derive theoretical predictions for the resistance against solvents solely based on chemical composition. It is thus likely that early pigments may behave different to contemporary ones (Herbst and Hunger 1995). Properties of dissolution are thus influenced by the chemical nature, crystal structure, particle size, as well as surface modifications of the pigment products. The focus of this project thus was to determine solvent sensitivity of synthetic organic pigments and the paints produced with them experimentally (Blumenroth 2010).

Analysis

The sensitivity of artist's paints containing organic pigments to solvent action arises not only from the solubility of the pigment itself, but also from the characteristics of the surrounding binder matrix and the swelling behavior of the whole system. The solvent resistance of 23 organic pigments in oil paint was tested with the six solvents n-hexane, toluene, chloroform, diethyl ether, acetone and ethanol - comprising a spectrum of different interaction properties and cavitation energies (Marcus 1998, Zumbühl 2011). The solubility of the pigments was determined quantitatively on pigment/solvent mixtures as well as by immersion of artificially aged oil paint films.[1] The time-resolved extraction was determined by UV-Vis

[1] Mussini and Norma (H. Schmincke & Co. GmbH & Co. KG) paints were applied in a wet film thickness of 100 μm with a film applicator Model 360 (Erichsen GmbH & Co. KG)

spectroscopy. Following solvent treatment by immersion of oil paint films, the influence of pigment extraction along the surface was examined applying infrared spectroscopy ATR-FTIR. Raman spectroscopy was applied to study morphological effects of extraction on polished cross-sections of embedded oil paint films. Structural damage was examined under practical conditions with cotton swabs using the six solvents.

Results and Discussion

The Solubility of Synthetic Organic Pigments

The solubility of the organic pigments was defined according to the industry standard by immersion in solvent. Properties of dissolution are influenced by the chemical nature, crystal structure, particle size, particle size distribution, as well as surface modifications of the pigment products and the reaction temperature (Herbst and Hunger 1995). Almost all pigments of the series exhibited discoloration of the liquid. Suspensions were found especially in solvents with high density such as chloroform. In particular, pigments with very small particle sizes resulted in stable suspensions where the particle sedimentation was very slow. After filtration with a 0.2 μm syringe filter, the molecularly soluble pigments became apparent. In addition, the filtered solutions were evaporated to quantitatively detect the pigment residues. Pigments exhibiting solubility within the tested set were the yellow and orange azo pigments PY 3, PY 97, PY 153, PO 5, the orange pyrazolochinazolon PO 67, the red anthraquinone PR 83:1, phthalocyanine blue PB 15:6, dioxazine violet PV 23, and to a lesser extent the red azo pigments PR 188, PR 177, as well as the anthraquinone perylene PR 179, as summarized in Table 1. The solubility is similar to the sensitivities known from literature and industry. The main focus was thus set on the behavior of these pigments bound in oil. Time-resolved quantitative extractions were determined on artificially aged oil paint films with UV-Vis spectroscopy.[2] For eight pigments, the extraction of pigments from the oil paint film was determined. The azo PY 3, PY 97 and PO 5 exhibited particularly high extractability. This is of course partly due to the solubility of the pigments, but it is also influenced by the swelling behavior of the surrounding binder matrix (Zumbühl 2011). According to the strong dispersive force of interaction, chloroform often showed the highest extraction capacity. Good solubility usually is encountered along the polarity scale from the polarizable toluene up to the dipolar acetone. Towards the non-polar end of the polarity scale, the solubility decreases in general. The non-polar n-hexane is a poor solvent with synthetic organic pigments. The blue phthalocyanine PB 15:6 also exhibits high solvent sensitivity, with the highest extraction rate observed in toluene. In contrast to the well-soluble azo pigments, the solubility of the PV 23 and PY 153 proved modest and amounted to less than 0.005 mmol/l. The quantitative effect of extraction of the soluble PY 3 after immersion of the paint film in chloroform is presented in Fig. 1.

on glass (Slides Assistent Elka 2400) and silicon Hostaphanfoil® RNT 36 (Kremer GmbH & Co. KG). After a preliminary drying of 7 days under room conditions, the samples were artificially aged. The lighting was done with the fluorescent tubes True Lite® 5,500 K and Philips® UV–20 W/08 F20 T12 BLB with ≈5,800 lm/m^2 and 557 mW lm^{-1}/≈3,200 mW/m^2 at ≈35 °C/≈45–50 %rH for a minimum of 6 months.

[2]Perkin Elmer Lambda 650 UV/VIS/NIR spectrometer in quartz cuvettes (10 mm deep K282) over the spectral range of 200–1,000 nm at a resolution of 1 nm. Quantification was carried out in relation to the specific pigment absorption λ max (Blumenroth 2010). It was measured at a time interval of 1 min over a period of 16 min.

Table 1 Overview on the results comprising all pigment/solvent combinations tested in the series

Pigment				Paint samples						Cleaning samples					
C.I.	Pigment class	Sensitivity		Pigment solubility						Solvent sensitivity					
		Literature	Pigment pure	Hexane	Diethyl ether	Toluene	Chloroform	Acetone	Ethanol	Hexane	Diethyl ether	Toluene	Chloroform	Acetone	Ethanol
PY3	Monoazo	***	***	o	*	**	***	***	*	*	**	**	***	***	***
PY97	Monoazo		***	o	o	*	***	**	o	*	**	**	***	***	***
PO5	β-Naphthol	***	***	o	*	*	**	*	o	*	*	*	***	***	**
PR188	Naphthol AS		*	o	o	o	o	o	o	*	**	***	***	***	***
PY151	Benzimidazolone	o	o	o	o	o	o	o	o	o	*	*	**	***	***
PY155	Bisacetessigarylide	o	o	o	o	o	o	o	o	o	*	**	***	***	***
PR242	Disazo condensation	*	o	o	o	o	o	o	o	*	**	**	***	***	***
PY153	Metall complex	*	*	*	*	*	*	*	o	o	*	*	***	***	**
PY139	Isoindolinone	o	o	o	o	o	o	o	o	o	*	**	***	***	***
PB15:6	Phthalocyanine	***	***	*	*	***	**	**	*	o	*	*	**	**	**
PG7	Phthalocyanine	o	o	o	o	o	o	o	o	o	*	*	**	**	*
PG36	Phthalocyanine	o	o	o	o	o	o	o	o	o	o	**	***	***	**
PV19	Quinacridone	o	o	o	o	o	o	o	o	o	**	**	***	***	***
PR122	Quinacridone	o	o	o	o	o	o	o	o	*	**	**	***	***	***
PR209	Quinacridone	o	o	o	o	o	o	o	o	*	**	***	***	***	***
PR179	Perylene	o	*	o	o	o	o	o	o	*	**	**	***	***	***
PO43	Perinone	o	o	o	o	o	o	o	o	*	**	***	***	***	***
PR177	Antraquinone	o	*	o	o	o	o	o	o	o	**	***	***	***	***
PR83:1	Antraquinone CA	***	**	*	*	*	*	*	*	o	*	*	**	***	**
PB60	Indanthrene	o	o	o	o	o	o	o	o	*	**	**	***	***	***
PV23	Dioxazine	*	*	o	*	*	*	*	o	o	*	*	***	***	**
PR264	Diketopyrrolo-pyrrole		o	o	o	o	o	o	o	o	*	***	***	***	***
PO67	Pyrazolochinazolone	***	***	o	o	*	***	*	o	o	*	**	***	**	*
PW4	Zinc white		o	o	o	o	o	o	o	o	o	*	*	*	*

Solvent sensitivity: o insoluble, *low, **high, ***very high

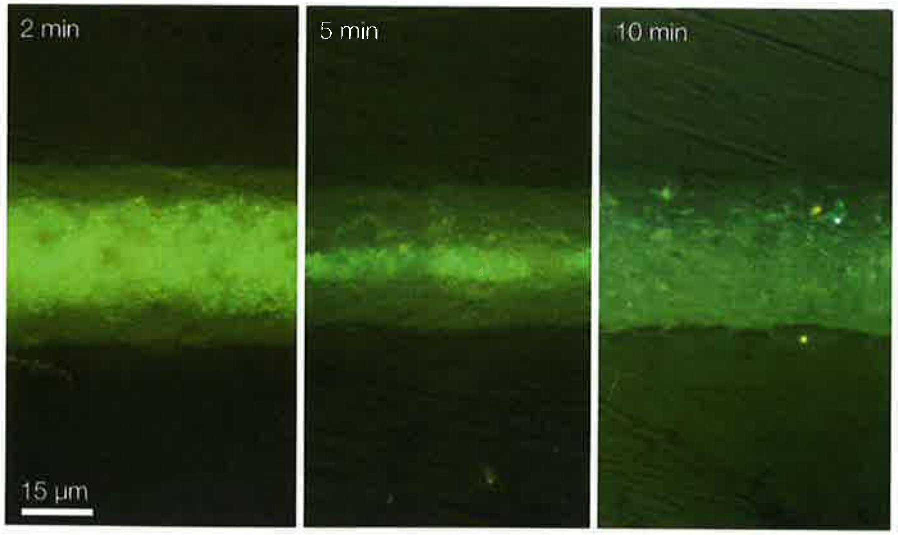

Fig. 1 Embedded PY 3 paint film cross-sections showing the time-dependent leaching front after immersion in chloroform. Bright field illumination with crossed polarization filters

The Influence of Solubility on the Oil Paint Film

The structural changes within the paint film were investigated using an artificially aged oil paint film containing the yellow azo pigment PY 3, which showed a strong solubility in the tests before. Applying infrared spectroscopy ATR-FTIR[3] to the surface of the leached films, the surface pigment response was tested upon solvent exposure of 1, 2, 5, 10 and 15 min (Fig. 2). After an immersion time of 1 min almost no pigment could be detected on the surface of the PY 3 oil paint film, even though no significant color changes can be observed visually.

With Raman spectroscopy,[4] the pigment concentration was examined along profiles across the embedded paint films on polished cross-sections.[5] All cases showed a steep concentration gradient (Fig. 3). The pigment was leached progressively from the exposed surfaces. Immediate leaching of the pigment occurs within the swollen parts of the film. This suggests very fast solubilization of the pigment with the solubility of the pigment being greater than the rate of diffusion of the solvent

[3]Avatar 360 FT-IR (Thermo Nicolet) ATR ZnSe-crystal Avatar Smart Miracle with 32 Scans and resolution of 4 cm^{-1}.

[4]Renishaw InVia Raman-spectrometer: 785 nm excitation, <12,1 mW on sample, 24 s per spot cumulative mode 6 × 4s, 1 µm step size, 100× objective, slit 65 µm standard in static mode, grating 1,200 l/mm, pinhole IN.

[5]Carbon evaporation coater Cressington Carbon Coater 108; embedding medium Technovit 2000LC.

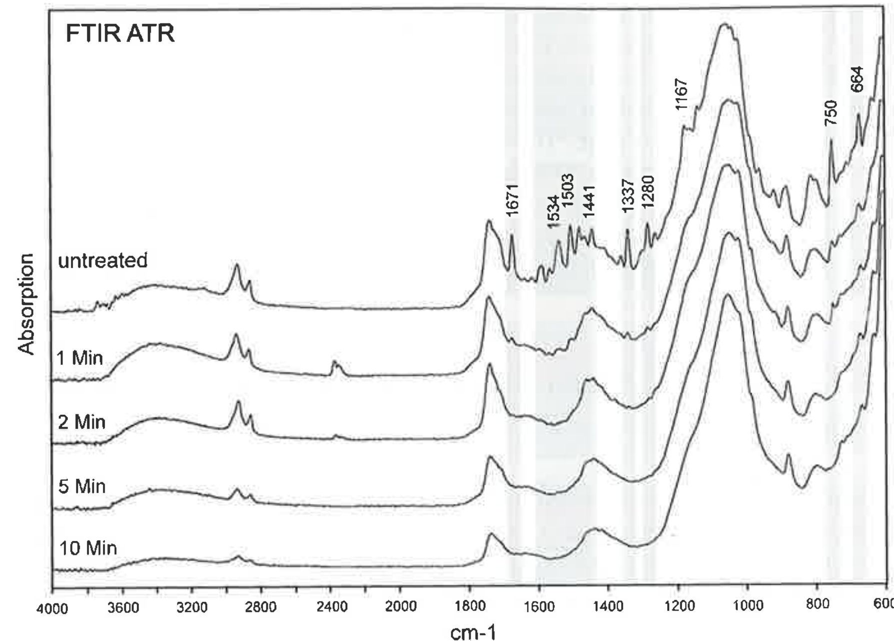

Fig. 2 Timed sequence of ATR-FTIR spectra on the PY 3 oil paint film after chloroform immersion: the pigment relevant response at the surface is lost within the first minute of solvent exposure

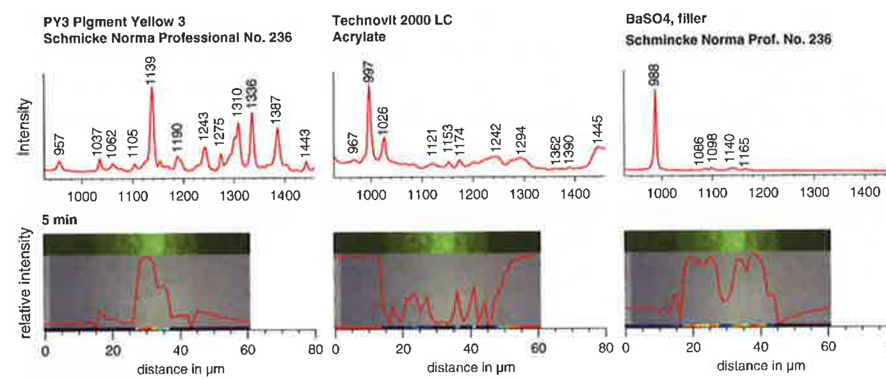

Fig. 3 The pigment loss, as observed in reflected light microscopy (Fig. 1), is also visible with Raman spectroscopy, documenting a sharp and clear delimitation of the solvent action horizon. Where the pigment and likely other paint additives are lost, the oil paint film becomes very porous and is infiltrated by the embedding medium

within the oil paint film. According to the signal response to ATR-FTIR, the pigment is being leached very efficiently from the surface (Figs. 2 and 3). Upon re-drying of the paint film, a porous film is left behind due to removal of pigment particles. This is also evident on embedded sample films, where the embedding resin was able to penetrate the leached zone. These results highlight the extremely delicate and fast response of paint layers with organic pigments to contact with solvents. The treatment of such materials thus sets a real problem to restoration practice. The same test procedure was also applied to the red 'insoluble' Disazo condensation pigment (PR 242). The paint film with this pigment, however, remained visibly unaffected.

Solvent Sensitivity of Paint Layers Containing Synthetic Organic Pigments

To test the sensitivity of paint layers containing synthetic organic pigments against solvents under real restoration conditions, cotton swab wipe tests with minimal pressure application were performed.[6] This allows the solvent to act in combination with light mechanical pressure on the surface. The tests were performed using commercially available solvent-moistened cotton swabs. They were wiped across the surface five times each as evenly as possible and with minimal contact pressure on the paint layer. While this test may not claim total objectivity, it nevertheless reproduces restoration practice and as such delivers useful information. The wiped cotton swab samples showed that all pigments within the series reacted very sensitively. Some exhibited pigment extraction at first contact with the cotton swab. This observation was independent from the pigment's classification with regard to the solubility examinations. In general, abrasion/extraction was lowest on cotton swabs soaked with non-polar solvents (Fig. 4). Solvents producing strong swelling of the oil binder caused an enhancement of this effect. Interestingly in contrast to the general solubility tests not only chloroform but also the polar solvents were most harmful. This may be explained by photochemical degradation and the change of the polarity of the binder in the paint layer's surface (Van den Berg 2002, Zumbühl et al. 2011). It also shows that the low wipe resistance is likely due to the small particle dimension of the pigments. By processing the pigments on a three-roll mill, the mean particle size of the tested pigments was between 8 and 12 μm. In contrast, the inorganic pigment zinc white was found to be significantly more resistant.

Shortly, as demonstrated in Fig. 4 and Table 1, these results show that the delicate behavior of paints with synthetic organic pigments against solvents is influenced by several factors. When considering the differences in solvent stability against the far less reactive classic oil paint made with inorganic pigments, one should bear in mind that the production of (metal) carboxylates as a factor of aging, has a stabilizing effect that is absent in modern paints with organic pigments.

[6] 100 μm oil paint films on slides, commercial cotton swabs.

Fig. 4 The action of solvents in combination with mechanical stress to oil paint films with organic pigments can be regarded as dramatic. Due to the small particle size, even "insoluble" pigments will be extracted. Polar solvents seem to cause the most damage. (H = n-hexane; D = diethyl ether; T = toluene; C = chloroform; A = acetone; E = ethanol, U = untreated)

Conclusions

Modern oil paints are very different to classical oil paint systems with inorganic pigments. This sets new problems to conservation treatments involving solvents. It was demonstrated that azo pigments tend to bleed in particular. Considering their wide distribution, there are many paints of the early twentieth century that are likely to react very sensitively to solvent exposure. Furthermore, other pigments from very different pigment classes were also found to be soluble. The pigments PY 3, PO 5 PO 67 and PY 97 were found to be completely soluble, while PY 153, PR 83:1, PV 23, PB 15:6 were partially soluble, and PR 188, PR 177, PR 179 were dissolved to a lesser degree. The susceptibility of a paint film to extraction upon solvent contact, however, depends not solely on the solubility of the pigment molecule. It is also highly influenced by the swelling properties of the binder matrix, controlling the stability of such heterogeneous structures like paint films. In combination with mechanical stress, the reaction speed was observed to be extremely fast, which makes it rather challenging when it comes to restoration treatment handling. Factors such as the very small particle size of these pigments will further contribute to the overall rather poor wipe resistance – regardless of the pigment solubility. A strong correlation between the polarity of the solvent and the power of material extraction was observed.

In conclusion, applying solvents for the cleaning of surfaces made from modern oil paints containing synthetic organic pigments is extremely delicate with a high potential for inducing unintended detrimental effects. It is important to know that pigments that are soluble won't lose this feature, even in old age. It means they stay soluble, even if the surrounding binder might become less sensitive towards solvents during aging, as known from analysis.

Synthetic organic pigmented oil paints can create problems in the preparation of cross sections, because bleeding may make interpretation of samples difficult.

The difficulty of assessing synthetic-organic pigments is their variability. The properties of a chemically identical pigment can differ by manufacturing and further modifications, this aspect was not included. The wipe samples should be repeated at a more advanced time with the aged oil. If possible quantitative aspects, which at this time were just qualitative, should be included.

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Dry Cleaning: Research and Practice

Maude Daudin-Schotte and Henk van Keulen

Abstract The Cultural Heritage Agency of the Netherlands (RCE) 2006–2009 dry cleaning research project investigated a broad range of dry cleaning materials (latex sponges, make-up sponges, PVC erasers, Factis erasers, Gum powders, microfiber cloths, mouldable materials), each with very variable affinities to dirt and paint surfaces, from efficient and safe cleaning to abrasion and residues hazards. This paper presents a follow-up to the RCE 2006–2009 research, focusing on the long-term effect of potential chemical residues, facilitated by our practical experience acquired in workshops since 2010 together with further scientific analysis.

Keywords Mercaptobenzothiazole • Plasticizers • Residues • Long-term effect • Dry cleaning • Make-up sponges • Micropore sponge • Sofft Tools® • Absorene® • Electrostatic roll

Introduction

After gentle brushing and vacuuming of the surface, unvarnished paintings can typically be cleaned by swab rolling the surface with aqueous solvents, except in cases where surfaces are sensitive to water or other solvents (Burnstock et al. 2006; Ormsby et al. 2006; Mills et al. 2008; Tempest et al. 2013). In such instances alternatives mainly based on mechanical action, such as dry cleaning, are mandatory (Estabrook 1989; Paerlstein et al. 1982). The RCE 2006–2009 dry cleaning research project investigated a broad range of materials with very variable affinities to dirt and paint surfaces (Fig. 1; Daudin et al. 2013, 2014). The use of these materials might imply friction and subsequent elevation of surface temperature, potential risks of abrasion and polishing, micro cracks or flattening, and particulate or chemical residues.

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