Ill. 1. Enclosed Garden with representation of the Calvary, Hunt of the Unicorn and the Immaculate Conception, after the conservation and restoration treatment ©KIK-IRPA, Brussels (x103024)

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The Enclosed Gardens of Mechelen: Revealed from Beneath the Dust

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Abstract: The Enclosed Gardens of Mechelen are extremely fragile historical mixed-media objects. To limit further degradation and to preserve their splendor, they must be exhibited in well-considered conditions. One of the challenges encountered in the Enclosed Gardens is the high level of accumulated dust. The deleterious effects of dust on the degradation of heritage items are well recognized. Dust induces visual, mechanical, biological, and chemical damage. First, the composition of the accumulated dust was determined to estimate its deleterious effects. Single particle analysis was performed and the water-soluble fraction was identified. Moreover, a dry cleaning treatment was performed to assess the level to which the accumulated dust could be removed. Lastly, the appropriateness of a common solution to protect the Enclosed Gardens from future dust accumulation is discussed, i.e., an airtight showcase. Such a solution should only be applied when the emission of adverse pollutant gases by the Enclosed Gardens is limited. Visual inspection and experiments have confirmed low off-gassing.

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Introduction

For almost 500 years, the seven Enclosed Gardens of Mechelen (ill. 1) were conserved in often precarious environmental conditions. Therefore, these precious medieval cabinets have reached an advanced state of deterioration. A thorough conservation and restoration treatment was required, which started in 2014. From 2018 on, all seven Enclosed Gardens will be permanently exhibited in the renovated museum Court of Busleyden in Mechelen. To limit further degradation and ensure their preservation, these heritage items should be exhibited in well-considered conditions.

Optimal conditions are being determined for both the long-term exhibition in Mechelen, and for a possible transportation of the Enclosed Gardens to temporary exhibitions. This is done by means of an adapted risk assessment, scientific research, and simulations. Several environmental factors are considered such as relative humidity, temperature, light, pollution, and vibrations. The complexity of this preventive conservation trajectory[1] is due to the diversity of materials used in these so-called historical mixed-media objects (panel paintings, wood, textile, metal, paper, parchment, polychromies, vegetal materials, glass, wax, etc.). Each material has its own optimal preservation conditions, but in an exhibition room human access and comfort have to be considered as well. Therefore compromises have to be found on different levels.

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One of the challenges encountered in the Enclosed Gardens is the high level of accumulated dust. Therefore, dust is one of the risk factors incorporated in the advice for future conservation conditions. The deleterious effects of dust particles for the conservation of cultural heritage are well recognized.[2] Four different types of damage are generally distinguished: visual, mechanical, biological, and chemical damage.

Visual damage is the most obvious type of damage: particle accumulation on objects alters their aesthetic appearance. For some people, soiling is perceived as disturbing. For others, it is appreciated as an aesthetic patina that evokes the feeling of an object to appear 'old' or 'antique'.[3] While soiling is about human perception, mechanical, biological, and chemical damage induce degradation of the art object.

Mechanical damage especially occurs with particles of high hardness (e.g., silicates), which lead to abrasion. Once such particles deposit, surface cleaning is considered as a hazardous treatment.[4] Increased dust deposition requires more frequent cleaning. This also increases the risk of material deterioration. Since dust becomes more strongly bound to surfaces over time, more invasive cleaning treatments might be required.[5]

Dust also enhances biological damage. On the one hand, airborne dust could contain particles from biological origin, such as bacterial and fungal spores. Once such particles settle on surfaces that are sensitive to biological attack (e.g., textile), they give rise to fungal growth, provided favorable microclimatic conditions such as high humidity levels.[6] On the other hand, deposited dust could create a humid micro-environment at the object's surface, creating a nutritional resource for fungi. Additionally, microorganisms could metabolize adhesive exopolymers that induce a more severe dust adhesion.[7]

Lastly, chemical reactions between deposited dust and the underlying surfaces hasten the deterioration of materials. This is due to the chemical composition and/or the moisture absorption capacity of the dust. Dust also accelerates the deterioration caused by gases, and particles could even transport harmful substances to indoor surfaces.[8] An example are soot particles that absorb reactive gases, bringing them in close proximity to the surface interface.[9]

The current study (1) explores the composition of the accumulated dust on the Enclosed Gardens, and discusses it in relation to the aforementioned damage types. This supports the risk estimation for the accumulated dust. (2) Second, the study assesses the degree of dust adherence and the possibility of dust removal. For this, the effectivity of a dry cleaning treatment of a silk object is evaluated. (3) Finally, a common protection against future dust accumulation is discussed, i.e., an airtight showcase. The current study tests whether this is an appropriate option for the Enclosed Gardens.

Materials and Methods

The results discussed in this paper are related to four Enclosed Gardens. To simplify the text, short references to these Gardens are used, i.e., the abbreviation EG followed by their number in the Court of Busleyden collection (table 1).

Before the start of the restoration treatment, accumulated dust was collected. Double-sided carbon tape was gently pressed on several dusted surfaces and carefully removed. Seven sampling

Table 1:
Abbreviations used to refer to a specific Enclosed Garden.

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locations were selected. In EG1, samples were collected at the ceiling, the side wall, the bottom part, the altar, and the head of the Lady with the Unicorn. In both EG2 and 6, dust was collected at the bottom area.

The sampled tape was analyzed with a scanning electron microscope coupled to an energy dispersive X-ray detector (ESEM-EDX, QUANTA 250 FEG, FEI, Hillsboro, Oregon, USA). The morphology of the sampled dust was studied based on secondary and backscattered electron images. Subsequently, single particle analysis was performed. The elemental composition of a total of 157 individual particles was determined (high vacuum, 20 keV, 30 s acquisition time). Backscattered images were used to generate contrast between the dust particles and the carbon tape. All spectra were evaluated and quantified, using a built-in standardless ZAF correction (Inca software package, Oxford instruments). Finally, k-means clustering was performed. The elemental particle composition used for the clustering was based on atomic percentages. Carbon and oxygen were excluded since their content cannot be accurately determined due to the influence of the carbon tape substrate. All other elements were normalized in order to get their sum equal to 1 for each dust particle. The clustering algorithm was performed for k values from 2 to 8. Based on the physical meaning of the cluster content, k=6 was selected as the most relevant distribution for classifying the data set.

During the restoration treatment, additional dust samples were collected to determine the water-soluble dust fraction. Dust was recuperated from a museum vacuum cleaner used for textile cleaning (555-MU-E HEPA). Two samples of 0.03600 and 0.01789 g, respectively, were mixed with 7 mL of milli-Q water, and subsequently vigorously stirred to dissolve the water-soluble fraction. The insoluble fraction was removed by passing the sample through a filter with a 0.2 μm pore diameter. Subsequently, the ion-rich solution was analyzed with an 833 Basic Plus Ion Chromatograph (Metrohm, Antwerp, Belgium) for both anions and cations. Concentrations were determined from calibration curves set up with standard solutions in the 1 to 20 ppm range.

To evaluate the effectivity of a dry textile cleaning treatment, a small silk fragment with the representation of a wine leaf was partially cleaned. The cleaning was performed with the museum vacuum cleaner with adapted suction. The nozzle was adjusted with a plastic pipette tip (Ø 1.5 mm). The dust was gently moved to the nozzle with a fine synthetic brush (Panduro Hobby 1/0 and 2/0). The partially cleaned silk fragment was subsequently analyzed with an environmental scanning electron microscope in low vacuum conditions (ESEM, QUANTA 250 FEG, FEI, Hillsboro, Oregon, USA). This allows the capture of secondary electron images without the need of sample coating.

Future dust accumulation would be highly reduced by exhibiting the Enclosed Gardens in airtight showcases. However, off-gassing of the materials used in the Enclosed Gardens could cause a pollution buildup inside the showcase. This should be avoided since it enhances further chemical degradation. An experiment was set up to evaluate the extent of such pollution buildup. EG3 was placed in an airtight box constructed with low-emitting materials. The box was made of polycarbonate plates and assembled with metal screws and bolts. The joins were covered with aluminum tape of archival quality, which is expected to have negligible emission (Lineco, self-adhesive frame sealing tape). On top of this tape, a broader aluminum tape without archival label was applied to ensure the air tightness. An air exchange rate of less than 0.05 air changes per day was reached in the box. The box was sized to enclose an air volume of around 150% of that of the Enclosed Garden. Gaseous pollutants were measured through passive sampling with Radiello diffusion tubes. The tubes were mounted both inside and outside the box with EG3 for a two week sampling period (ill. 2). The experiment was repeated in the same box without the Enclosed Garden. Active cartridges for NO_2

Ill. 2. Experimental setup to test the extent of pollution buildup in an airtight box (± 100 × 115 × 40 cm) with EG3 (± 90 x 109 x 30 cm) ©W. Anaf

and SO_2 were used. These also allow the detection of formate and acetate. After sampling, the tubes were analyzed with ion chromatography (833 Basic Plus, Metrohm, Antwerp, Belgium). Formate and acetate were analyzed according to the method of Kontozova-Deutsch et al.[10] and Stranger et al.[11]

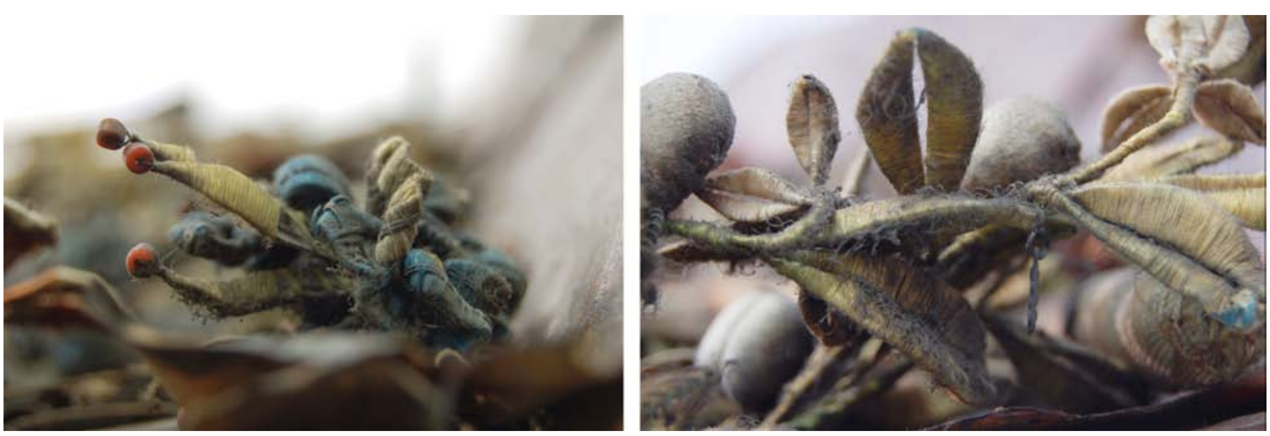
Results

Ill. 3 shows the dusty appearance of the Enclosed Gardens. The photographs illustrate the aesthetic nuisance that dust causes on the detailed silk flowers and vegetal motifs. Moreover, the dust further obscures the already faded colors of the Enclosed Gardens.

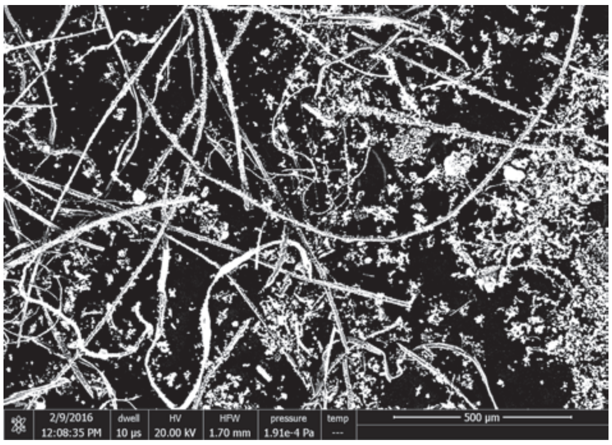
The dust samples collected on the carbon tape were subjected to a basic morphological analysis with secondary and backscattered electron images (ill. 4). It was remarkable to detect textile fibers on all horizontal sampling locations, even when the sampled surface was not made of textile. Due to the fragile state of the Enclosed Gardens, it was expected that the detached fibers mainly originated from the silk flowers and vegetal motifs in the Gardens themselves. Due to gravitational settling, such large particles mainly deposit on horizontal surfaces.

Moreover, the dust particles were identified through chemical analysis. The 157 analyzed particles were classified into 6 clusters, each corresponding to a specific particle type. Three of the clusters contain mineral particles: calcium-rich particles, silicon-rich particles and particles with a mix of various mineral-related elements. These three mineral-related clusters cover 75% of the analyzed particles. Two other clusters contain either iron-rich or copper-rich particles. The last cluster is characterized by a high nitrogen content.

The risk of mechanical damage is increased in the presence of mineral-related particles. Silicon-rich particles, in particular, could have a high hardness which promotes abrasion processes. At high



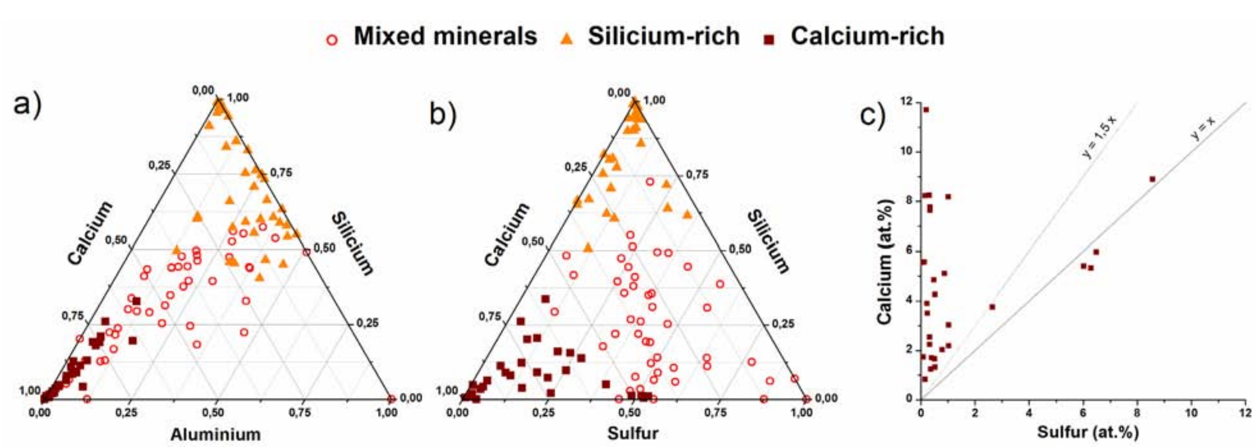
Ill. 3. Dust deposition on silk flowers in EG1, before restoration ©W. Anaf



Ill. 4. Backscattered electron image of a dust sample collected on carbon tape (sample location: EG1 - bottom part, on top of green paper)

humidity conditions, mineral particles and especially calcium-rich particles could form microcrystalline calcite cements.[12] Such a cementation process causes a stronger attachment of the dust particles to the underlying surface, impeding easy dust removal. The composition of the mineral particles found in the Enclosed Gardens is visualized in ill. 5. The ternary plots in ill. 5 a) and b) show that the silicon-rich particles have a low calcium and sulfur content. Their aluminum content is rather low as well. The mixed mineral particles are distributed between the silicon-rich and calcium-rich particles. They have the highest sulfur content and an intermediate silicon and calcium content. Lastly, the calcium-rich particles have a low silicon and aluminum content. Most common environmental calcium-rich particles are carbonates, sulfates and nitrates. Nitrogen was not detected in any of the analyzed calcium-rich particles, which excludes nitrates. The carbon content cannot be interpreted due to the influence of the carbon substrate. However, the calcium-to-sulfur atomic ratio is higher than 1.5 for 25 out of the 30 calcium-rich particles (ill. 5c). Therefore, an important contribution of carbonates is expected.

The mixed minerals and calcium-rich particles were detected at all sampling locations in the three studied Enclosed Gardens. However, the silicon-rich particles were detected in all samples collected in EG1, but not in the samples collected in EG2 and EG6. Several hypotheses are formulated for this finding. All three Enclosed Gardens were closed with glass panes. At the start of the restoration project, the protective windows were removed. In EG2 and 6, the glass panes were fixed in a metal frame, which was sealed to the cabinet with mastic. This created a more closed environment inside the cabinet, but also complicated the window's removal. EG1 had a wooden framed glass pane that could be easily detached by removing two dowels. This wooden frame provided a less close fit, with small openings near the cabinet. Have the Enclosed Gardens at one point been exposed to an environ-



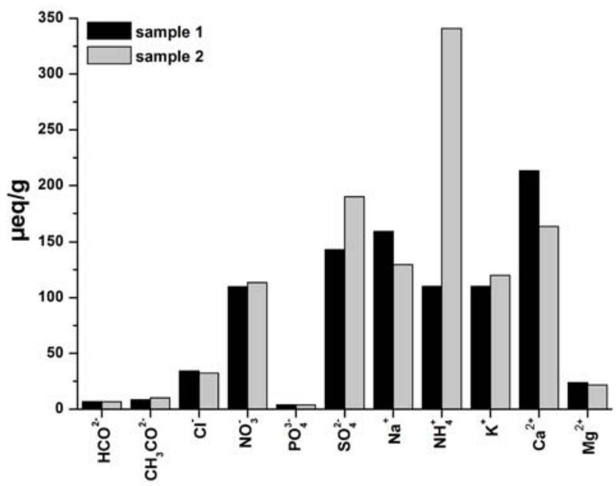
Ill. 5 a-b-c. Ternary plots visualizing the three clusters of mineral-related dust particles; c) Scatter plot of sulfur to calcium for the calcium-rich particles ment with a high level of silicon-rich particles? Was EG1 opened more often than the others? These are questions for further research. It is not expected that the silicon-rich particles originated from deteriorated glass fragments in the Enclosed Gardens, such as the glass beads. If originating from these glass fragments, silicon-rich particles would be expected mainly on horizontal surfaces, but they were found at different orientations. In addition, EG2 and 6 showed a higher level of glass deterioration.

Iron-rich particles are known to catalyze the reaction from SO_2 to H_2SO_4.[13] Moreover, Grau-Bové et al.[14] proved that the presence of transition metals such as copper and iron hastens the degradation of cellulose fibers and thus increases the risk of chemical degradation. Nine of the eleven copper-rich particles were detected at the bottom part of EG2. Since this area is painted in green, there is a high probability that small flaked off copper-based paint fragments were collected. Since only 5 iron-rich particles were detected, their source is hard to determine.

The nitrogen-containing particles mainly consist of carbon, oxygen and nitrogen, with traces of sulfur, sodium, and calcium. They probably have an organic origin. It could be small fragments of for example silk fibers. Another option are human skin flakes. Biological particles such as spores have a similar chemical composition, but often have a characteristic morphology. Particles with such a morphology were not observed. Moreover, signs of (historical) mold growth were not detected in the Enclosed Gardens.

Chemical degradation promoted by dust particles is linked to the chemical composition of the dust, but also to its hygroscopic properties. Once dust particles deposit on heritage materials, the adverse effects largely depend on their hygroscopic characteristics. Water often plays a crucial role in degradation phenomena in general, and it probably promotes chemical reactions between particles and their deposition surface. Also the cementation process is promoted in the presence of hygroscopic dust. All particles, even inert ones, act as condensation nuclei. They adsorb humidity from the ambient air, thus increasing their water content.[15] However, for insoluble particles such as silica, the water uptake is only significant in the extreme conditions of a supersaturated vapor. Water-soluble particles, on the other hand, have a moisture absorption capacity and already show considerable water uptake under moderate conditions.[16] These soluble particles have a strong affinity to water and possess the ability to attract and hold water from the environment. When deposited, such hygroscopic particles attract water on the deposition surface, accelerating any degradation process favored by humid conditions such as oxidative degradation of paper and textile, corrosion of metals, discoloration of pigments, etc.[17] Moreover, when a surface is soiled with a hygroscopic contaminant, the liquid film easily adheres other particles that touch the surface, increasing the rate of particle deposition.[18]

Two dust samples were analyzed for their anions and cations to gain insights in the water-soluble and thus hygroscopic fraction of the accumulated dust on the Enclosed Gardens. The samples were collected with a museum vacuum cleaner by the textile conservator. The samples had a mass of 0.03600 and 0.01789 g, of which respectively 7 and 8 wt% was water-soluble. The measured ion concentrations were recalculated to μequivalents per gram. The latter unit better reflects the chemical meaning of the ion concentrations: one equivalent of an anion could react with one equivalent of a cation. The results of the analysis should give equal anion and cation concentrations. However, a clear anion deficit is present. This is commonly observed, certainly for coarse dust particles. Carbonate (CO_3^{2-}) was not measured, but is expected to be present mainly as a mineral component.[19] This confirms the results of the single particle analysis, where the calcium-rich particles were found to be rich in carbonates. Ill. 6 shows the equivalent concentrations of all measured ions. The ion concentrations are similar in both samples, except for ammonium, which is far more abundant in sample



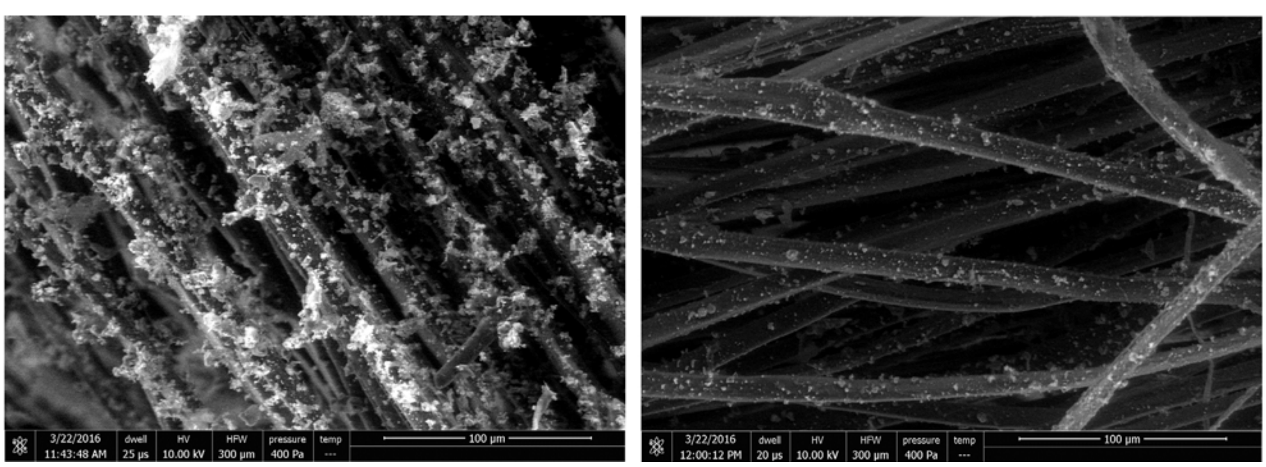
Ill. 6. Concentrations of water-soluble ions in two dust samples removed by the textile restorer. Results are given in µequivalents per gram

2. Sulfate and nitrate are the most abundant anions. For the cations, high abundance of sodium, ammonium, potassium and calcium is observed. The hygroscopic properties of the dust depend on the type of salt present in the dust. Although the exact salt compositions cannot be deduced from the current analysis, humidity levels are given at which certain salts form a saturated salt droplet, i.e., their deliquescence relative humidity (DRH). Calcium carbonate ($CaCO_3$) has a DRH of 97% and is therefore of lower risk regarding its hygroscopic properties. Sodium chloride (NaCl) deliquesces at a relative humidity of around 75-78%, $(NH_4)_2SO_4$ at 80-81% and NH_4NO_3 at 61-62%. Certain salts have very low DRHs, such as $MgCl_2$ (33%).[20]

To assess the effectivity of the dry cleaning treatment, an original wine leaf was partially cleaned by the textile restorer. Ill. 7 shows secondary electron images of the silk fibers without and with cleaning, respectively. The cleaning removed a large part of the dust particles, but even after the cleaning, small dust particles (mainly < 5 µm) stay attached to the silk fibers. Judging by visual observation, the dusty appearance is highly reduced after cleaning. However, dust is still perceptible. Deeper areas are more difficult to clean, leaving larger dust particles that could not be removed.

Although the cleaning appears relatively effective, its damaging effect should not be underestimated. An important fraction of the removed dust was identified as detached (silk) fibers. This again confirms the extremely fragile state of the silk. The silk could have been broken and detached in the past, but could also have been broken during the cleaning treatment. It is almost impossible to clean strongly degraded silk without additional material loss.[21]

To protect the Enclosed Gardens from future dust accumulation in the permanent exhibition, a commonly used solution would be airtight showcases. Due to the air tightness, all external



Ill. 7. Secondary electron images of the wine leaf. Left: before cleaning; right: after dry cleaning.

influences such as incorrect or fluctuating relative humidity, external pollutant gases and dust are blocked.[22] However, pollutants generated inside the showcase cannot escape, and high pollutant levels might arise. Internal pollution sources could be the materials used to construct the showcase. By using appropriate materials, this source could be easily avoided. However, the Enclosed Gardens themselves could also be a source of harmful compounds. Oak, for example, as used for the cabinet, is a well-known source of acetic and formic acid.[23] These pollutant gases enhance the degradation of other materials present in the Enclosed Gardens. It is well-known that material off-gassing reduces over time, but there is a lack of published data on oak emission rates on the long-term. Therefore, an experiment has been set up to examine if there is still substantial off-gassing from the materials used in the Enclosed Gardens. The results of the diffusion tube analysis demonstrate that no elevated formic and acetic acid concentrations were present in the airtight test box with EG3 compared to the restoration workshop. This result is confirmed by a long-term test that was unknowingly performed in EG3. The protective glazing that was present in this Enclosed Garden was made of lead pane windows. Lead is known to be highly reactive to volatile organic compounds such as formic and acetic acid. These gases hasten lead corrosion, and the process is promoted by high humidity conditions.[24] However, the lead does not show an advanced state of corrosion, not even the inner side that was exposed to the cabinet.

Discussion

The deleterious effects of the accumulated dust on the Enclosed Gardens are well demonstrated. Visual damage creates an aesthetic nuisance and obscures the remaining colors of the Enclosed Gardens. Mineral particles and especially particles of high hardness cause mechanical damage by abrasion. A cementation process increases the adherence between the dust particles and their underlying surface. This increases the need for more invasive cleaning treatments, which is problematic due to the fragile state of the Enclosed Gardens. It is therefore not surprising that many broken and detached silk fibers were found in the accumulated dust that was removed during the cleaning treatment. Moreover, it is demonstrated that a dry cleaning treatment does not remove all dust particles. Hygroscopic dust particles promote the cementation process, but also hasten chemical degradation reactions by their ability to hold water at their deposition surface. Based on the morphological analysis, no biological dust particles were detected. However, this does not mean that the Enclosed Gardens are exempted from biological damage. Insects could feed on materials present in the Enclosed Gardens such as the textile, wood and paper. Insect remnants were found in the Enclosed Gardens, evidencing insect attack in the past. Therefore, precautions should be taken to prevent future attack.

After summing up the damage phenomena enhanced or induced by dust deposition, it is clear that recommendations for future conservation conditions include low dust levels: the lower, the better. Low airborne dust concentrations cause lower dust deposition rates and thus decrease the frequency of cleaning treatments. More generally, the risk for enhanced degradation is reduced.

Visitors are well-known contributors of dust in collections. Mineral particles stick to their shoes and detach during the museum visit. Also (loose) cloth fibers are released. In the past, several studies were performed to investigate the relation between visitor behavior and deposited dust levels.[25] One option to reduce dust deposition is related to the location of the object in the visitors' route. The further away from the entrance, the less deposition is observed. The Enclosed Gardens will be exhibited at the end of the visitors' route through the museum Court of Busleyden. Therefore, reduced dust levels could be expected compared to the start of the route. A second option to reduce the level of deposited dust, is to increase the distance between the object and the visitor. However, to admire the

Enclosed Gardens in all their details, limited distance between the object and the visitor is required. Therefore, to optimally protect the Enclosed Gardens from dust accumulation, it was decided to exhibit them in individual showcases with a low air exchange rate. These passively climatized showcases will also protect the Enclosed Gardens from other agents of deterioration such as incorrect humidity, external pollution sources, theft and vandalism, insects etc. This study has demonstrated that internal pollution buildup due to emissions from the Enclosed Gardens themselves is limited.

Conclusion

The extremely fragile Enclosed Gardens should be kept in appropriate environmental conditions to limit further degradation. A main problem encountered, was the high level of accumulated dust in the Enclosed Gardens. Dust induces visual, mechanical, biological and chemical damage. Based on the identification of the dust composition, the risk posed by the accumulated dust could be estimated. Dry cleaning of a silk fragment demonstrated that a large fraction of deposited dust could be removed. However, small dust particles stay attached to the silk fibers.

The Enclosed Gardens will be permanently exhibited in the Court of Busleyden. To protect them from future dust accumulation, they will be displayed in airtight showcases. The study demonstrated that pollution buildup within the showcase due to off-gassing of materials used in the Enclosed Gardens, will be limited. With a good control of all agents of deterioration, and a good follow-up, appropriate preservation conditions will be created. This ensures the preservation of the Enclosed Gardens for future generations.

Acknowledgements

Willemien Anaf thanks the Jean-Jacques Comhaire Fund and Prize, managed by the King Baudouin Foundation, for their financial support for the present study. The authors are grateful for the fruitful collaboration and help of the Enclosed Gardens restoration team, coordinated by Joke Vandermeersch and Hilde Weissenborn.

Notes

1 Preventive conservation: all measures and actions aimed at avoiding and minimizing future deterioration or loss. They are carried out within the context or on the surroundings of an item, but more often a group of items, whatever their age or condition. These measures and actions are indirect – they do not interfere with the materials and structures of the items. They do not modify their appearance (Definition as provided by ICOM-CC, 2008).

A major framework used in the preventive conservation field are the ten agents of deterioration. These are ten primary treats to collections, i.e., incorrect relative humidity, incorrect temperature, radiation, pollution, pests, physical forces, thieves and vandals, fire, water and dissociation. More information on the ten agents of deterioration can be found on the website of the Canadian Conservation Institute (https://www.canada.ca/en/conservation-institute/services/agents-deterioration.html)

2 Tétreault 2003.

3 Lithgow & Brimblecombe 2003: 47-49; Lloyd, Brimblecombe & Lithgow 2007: 135-146; Dillon *et al.* 2017.

4 Ioanid, Parpauta & Vlad 2005: 1643-1649; Wei *et al.* 2007: 261-269.

5 Spafford-Ricci & Graham 2000: 37-56; Brimblecombe, Thickett & Yoon 2009: 410-414.

6 Ioanid, Parpauta & Vlad 2005: 1643-1649; Prajapati 2003: 46-54; Szostak-Kotowa 2004: 165-170.

7 Appelbaum 1991; Caneva, Nugari & Salvadori 2008.

8 Walton, Johnson & Wood 1982: 59-64; Melcher, Schreiner & Kreislova 2008: 346-356.

9 Van Grieken *et al.* 2000: 215-228; Yoon & Brimblecombe 2001: 232-240.

10 Kontozova-Deutsch *et al.* 2008 : 418-423.

11 Stranger *et al.* 2009: 411-417.

12 Brimblecombe, Thickett & Yoon 2009: 410-414.

13 Camuffo *et al.* 1999 : 135-152.

14 Grau-Bové *et al.* 2016.

15 Brimblecombe, Thickett & Yoon 2009: 410-414.

16 Hinds 1982.

17 Feller 1994; Havlínová *et al.* 2009: 222-231; Saunders & Kirby 2004: 62-72.

18 Camuffo 1998.

19 Horemans et al. 2011: 429-438.

20 Anaf 2014.

21 MEMORI 2018.

22 Thickett, David & Luxford 2005: 19-34.

23 Gibson & Watt 2016: 172-178.

24 Tétreault, Sirois & Stamatopoulou 1998: 17-32.

25 Yoon & Brimblecombe 2001: 232-240; Yoon & Brimblecombe 2000a: 445-454; Yoon & Brimblecombe 2000b: 127-137.