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The Analysis of Red Lead Endsheets in Rare Books from the Fung Ping Shan Library at the University of Hong Kong

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Abstract: Stored under optimal conditions, paper can last for hundreds of years; however, paper is also susceptible to numerous agents of deterioration as it is primarily made of cellulose fibre derived from plant cell walls. Paper is especially vulnerable under conditions with high temperature and humidity, which together can encourage mould growth and foster insect pests. Pest infestation is a common type of deterioration responsible for significant damage to paper and books. Endsheets prepared with red lead and sewn into traditional Chinese bookbindings are a historical method used to minimise insect damage to books in southern China. Samples of red-coloured papers from the Fung Ping Shan Library at the University of Hong Kong were assessed. Two types of so called *wan nian hong* papers (萬年紅紙), (1) damaged and (2) undamaged by insects, were analysed with a scanning electron microscope equipped with an energy dispersive X-ray fluorescence detector. The chemical analyses reveal high concentrations of red lead (lead tetroxide, Pb_3O_4) in the undamaged papers, and only very few traces of lead in the damaged papers. The orange papers with insect feeding tunnels seem to have been only coloured in orange, and not treated with red lead as a biocide. These results are discussed and compared with modern treatment methods in the integrated pest management of books, archive, and library materials in (a) Europe and (b) Asia.

Keywords: IPM; insect damage; orange paper; red lead; prevention; biocide

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1. Introduction

Library and archive materials, commonly made from paper, parchment, cloth, leather, and wood, are at constant risk of being damaged. Stored under optimal conditions, paper can last for hundreds of years, but it is also susceptible to numerous agents of deterioration. Paper is especially vulnerable under conditions with high temperature and humidity, which together can encourage mould growth and insect pests. The material is primarily made of cellulose fibres derived from plants. Pest infestation is a common type of deterioration responsible for significant damage to paper and books [1]. In the following section the most important insect pests of paper and books are described.

1.1. Insect Pests in Libraries and Archives

Book, paper, and archive materials are at constant risk of being damaged by a variety of insect species [2–8]. Three types of paper damaging pests can be differentiated: the first group are beetles (Coleoptera), whose larvae feed inside the books and damage the object by tunnelling through the material. Most common among these is the biscuit beetle (*Stegobium paniceum*), cigarette beetle (*Lasioderma serricorne*), and spider beetle, such as the Australian spider beetle *(Ptinus tectus)* and the white-marked spider beetle *(Ptinus fur).* The larvae especially like to feed on the starch-based adhesives that are used to bind books. The larvae live inside the books and only the beetles can be found when they are reproducing

outside the substrate. Within historic collections in Europe that have had less stable climate control systems, the furniture beetle (*Anobium punctatum*) and other woodborers also can be found feeding on books [1,5,8]. Today the furniture beetle has largely been replaced by the biscuit and spider beetles, as these species are more tolerant of a greater range of humidity and temperature levels. Biscuit beetles can be particularly destructive because of their high rate of reproduction. The same is true for cigarette beetles and other species of anobiids [9–16], which are more common in warm environments and tropical climates.

The second key groups of pests found among books and paper are silverfish (*Zygentoma*). Several different species of silverfish can be found within collections: the common silverfish (*Lepisma saccharinum*), the grey silverfish (*Ctenolepisma longicaudatum*), the "ghost" silverfish (*Ctenolepisma calvum*), and the four-lined silverfish (*Ctenolepisma quadriseriatum*) are found widely across Europe [17–22]. In Asia the oriental silverfish (*Ctenolepisma villosa)* is also found, along with the firebrat (*Thermobia domestica*) in dryer climates. Each of these species feed primarily on cellulose materials and will damage paper, bindings, wallpaper, papier-mâché, and starch-based adhesives; they also feed on detritus, mould, human skin or hair, textiles, protein-based glue, cotton, and silk. The animals live in the cracks and underneath floorboards during the day and infest and damage the materials at night. They feed in the surface of the paper resulting in irregular damage and holes. Additionally, the book louse (*Psocoptera*) is found in particularly high numbers in humid conditions [5,9]. Species such as *Liposcelis* sp. generally eat mould and starch, but again, they also will infest paper, books, herbal specimens, wallpaper, and even stuffed animals.

The third group includes pests such as termites, which in the tropics prove to be particularly damaging. Drywood termites and soil termites both invade libraries and damage books, bookshelves, or the entire building [1,5,9]. Due to the high levels of humidity and temperature found across the tropics, pest damage from termites can be extremely severe and can occur in a very short period of time. Termite activity can be detected by the galleries, but infestations are often overlooked.

1.2. Control of Insect Pests in Libraries and Archives

1.2.1. Chemical Methods

In the past, chemicals were the preferred method for treating active infestations in museums, archives, and libraries. Since the prohibition of DDT, methyl bromide, and hydrogen cyanide, very few European libraries have continued to employ pesticides [5]. Sulfuryl fluoride is the sole toxic gas still occasionally used in collection environments; however, it is costly and the related restrictions on its use within urban environments and residential areas are limited. Pyrethroid fumigations are not suggested, as they do not kill all insect stages, especially the larvae inside the material or the silverfish hiding in the cracks in the floor and corners during the day. Rather, the vast majority of libraries and museums in Europe, North America, or Australia now utilise a series of non-chemical methods as part of an integrated pest management program (IPM). See the following list of alternative treatment methods.

1.2.2. Non-Chemical Methods

Insect pests can be eradicated using a variety of non-chemical methods. These methods are preferred within museums, libraries, and archives, as they pose no harm to the objects or staff. Successful treatment can be achieved by using physical methods like freezing [23] or humidity regulated heating to kill all stages of the pests. For more delicate objects and mixed materials, anoxic treatments are the preferred method [24]. A low oxygen atmosphere can be achieved using nitrogen, argon, or oxygen scavengers in small bags. CO_2 is sometimes applied within museums, as it is also not harmful to objects. A relatively new method is biological control by using parasitoid wasps, for example, against biscuit or furniture beetles [25]. Initial results show that this method will work, if the location of the infestation is known at an early enough stage, and if the parasitoid wasps are commercially available [25]. Gamma radiation [26] was used for some time against fungi and insect

pests, but was found to be damaging to the paper material. Therefore, it is only used in countries where no alternatives are available. See Querner and Kjerulff [27] for an overview of treatment methods used in museums.

No single treatment method is perfect and the chosen method must be applied based on the available timeframe. Heat treatment only takes 24 h, while nitrogen treatments last from three up to five weeks. Other considerations are availability, financial resources, biocidal regulation (by the EU for example), the type of pest species, and the specific materials to be treated. Integrated pest management is now the standard across the GLAM sector (Galleries, Libraries, Archives, and Museums) [2–9].

1.3. Materials Used for Paper and Books in China

The earliest fibre used to make paper in China was hemp. *Xuan* (宣紙), mulberry, and bamboo are the most common papers produced over the past few centuries. In the Tang dynasty (618–907) *Xuan* paper made from blue sandalwood bark (*Pteroceltis tatarinowii*) and rice straw (*Oryza sativa*) was considered to be of high quality and became popular for calligraphy and painting. This type of paper is still produced in Anhui province. Mulberry fibres produce a strong paper that became popular in China during the Song dynasty (960–1279). The first known use of bamboo for papermaking was during the Tang dynasty. The raw materials for bamboo paper (grasses from the *Poaceae* or *Gramineae* family) are inexpensive, sustainable, and easy to harvest. They are primarily produced in Fujian, Sichuan, and Zhejiang provinces with much of the production located in Southern China. Xuan, mulberry, and bamboo papers have all been used for manuscript and printed books.

1.4. Traditional Methods to Protect Papers in China from Insect Attack

Dying paper yellow to keep insects away is a treatment method documented and used in China as early as the second or third century [28]. This dye was made from the Amur cork tree and is known as *huang bo* (黃柏) in Chinese. Applied to the entire surface of the paper, numerous examples which date from the fourth to eleventh century were discovered in caves in Dunhuang, China in the 20th century. These findings were flat or rolled and preceded the current side-sewn type of book binding in China. As printing and binding technologies evolved, a different type of coloured paper was used to prevent insect damage.

1.5. Introduction to RED–ORANGE Papers in China

During the Ming (1368–1644) and Qing (1644–1912) dynasties, red lead was placed into a solution and applied to paper that was used to wrap books or was bound into the front and back as endsheets [28–32]. The Chinese name for these endsheets is *wan nian hong* paper (萬年紅紙). Literally translated, *wan nian hong* paper means 10,000-year red paper, though most of the papers are actually a bright orange colour. The sheets are made through the addition of red lead or litharge *qian-dan* (鉛丹) powder, which is mixed with water and vegetable glue and then brushed onto the paper. The majority of the books that contain these papers date to the Ming and Qing dynasties and are found in collections in southern China (Figure 1).

Bamboo paper has been shown to be more susceptible to insect damage, and consequently the orange-coloured insecticidal paper is found primarily in volumes from southern China, where bamboo paper production has been historically more widespread. The orange paper does not actually prevent damage, as the chemical compounds in the paper only kill the insects after contact and/or ingestion. In Joseph Needham's *Science & Civilisation in China* is a description of the traditional process of preparing red lead for use on paper ([33], p. 76). Chinese thread bound books are printed or written on one side of a sheet of paper that is folded at the foredge and stab sewn along the long cut edge. The insecticidal solution is applied to only one side of the paper and is most often folded and sewn into the text block.

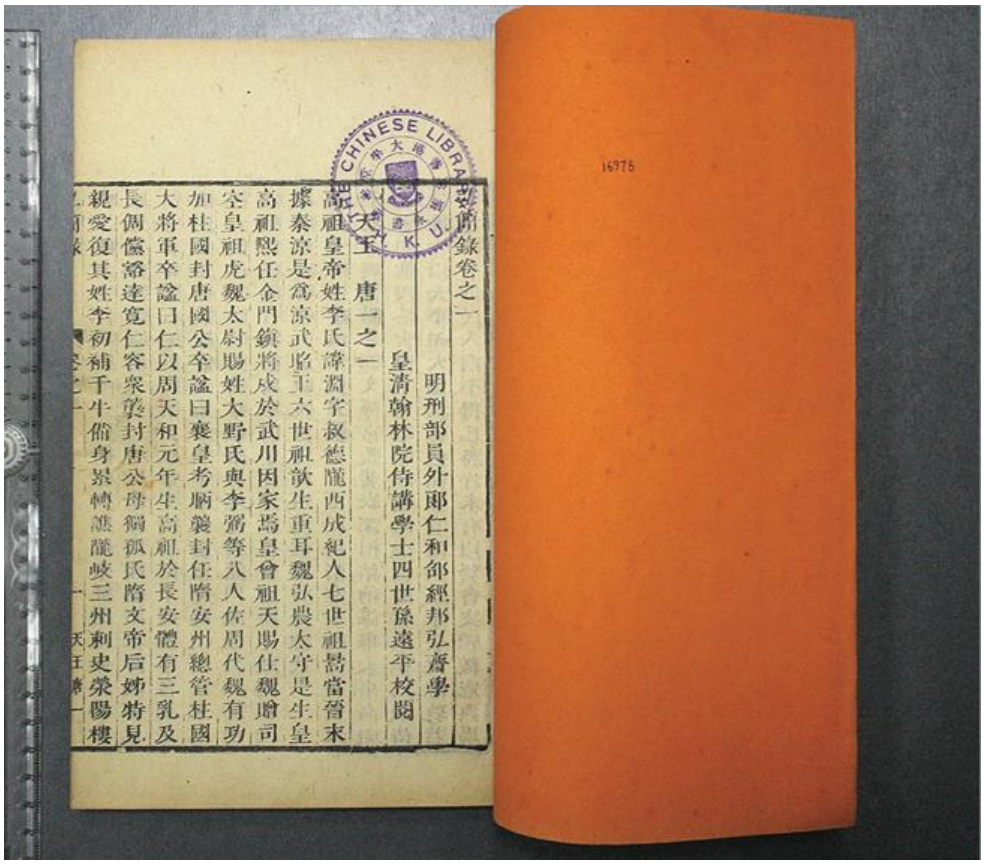
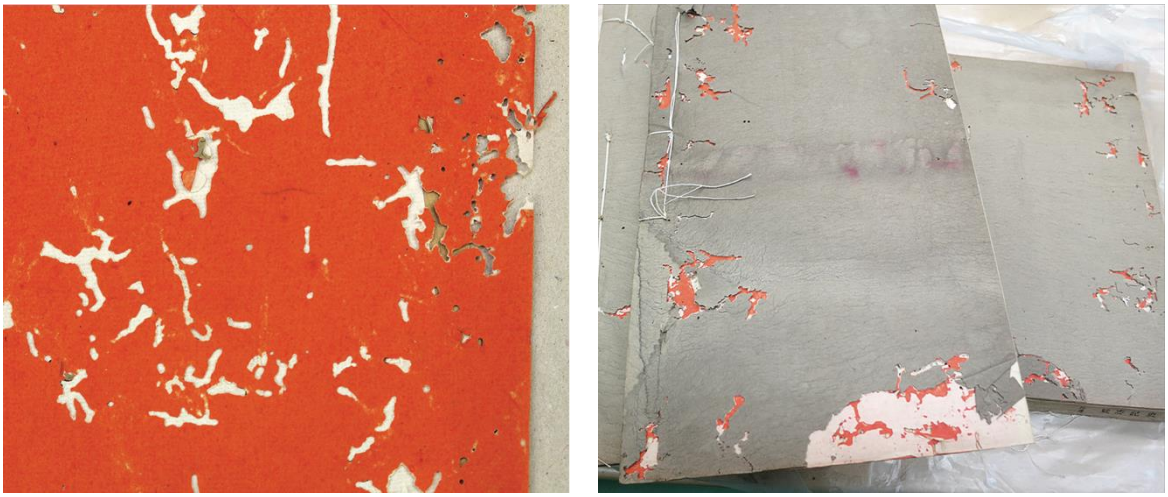


Figure 1. Undamaged orange paper in the Fung Ping Shan Library at the University of Hong Kong. Photo Jody Beenk.

These orange papers have been the subject of research by various institutions across China [28,34,35], and the presence of red lead has been verified by multiple studies. Tests have also been carried out to prove the effectiveness of the insecticidal properties of the prepared papers. Application of the insecticidal solution to the paper is not always done evenly and does not always extend to the ends of the paper. It is not uncommon to find an endsheet that is only partially covered in the orange colour, resulting in examples where the uncoloured area has been severely damaged by insects, while the orange-coloured areas remain undamaged.

1.6. RED-ORANGE Papers at the Fung Ping Shan Collection, Library of the University of Hong Kong (HKUL)

Because of geographic proximity and economic factors, bamboo paper is found in many of the books at HKUL [28]. According to a survey completed by the author, this type of red-orange endsheet is found in 65% of the rare books in the Fung Ping Shan Library at the University of Hong Kong. The bulk of the historical rare book collections were acquired in the 20th century from libraries and collectors who housed them in environmental conditions similar to Hong Kong. Located on the southern coast of China, the climate in Hong Kong is subtropical and insect damage to paper, books and museum objects is a constant challenge. Interestingly, some of the orange papers in the Fung Ping Shan collection have not proven effective, as is evidenced by the tunnels of insect damage across the orange sheets (Figure 2a,b). We aim to investigate the chemical compounds of the damaged and undamaged orange papers from the Fung Ping Shan Library at the University of Hong Kong and discuss this method of pest prevention or intervention with other modern day IPM methods used in Europe and Asia.



(**a**) (**b**)

Figure 2. (**a**,**b**): Damaged orange paper with insect feeding tunnels in the Fung Ping Shan Library at the University of Hong Kong. Photos Pascal Querner.

2. Materials and Methods

We sampled ten red-orange endsheets from late 19th-century books with broken bindings that were part of a recent donation to the Fung Ping Shan Library at the University of Hong Kong (HKUL). Non-destructive or micro-sampling was not available at the library. The samples came from multi-volume titles and the remaining endsheets will be housed with the original texts when conservation treatment is completed. Five of the endsheets were damaged by unknown insect pests, and the other five were without visible pest damage. From each endsheet, three 1 × 1 cm samples (pseudo replicates; n = 3) were selected for further study. The endsheet samples were transported from Hong Kong to Vienna (Austria) for analysis on 25 January 2017, at the Federal Monuments Authority of Austria.

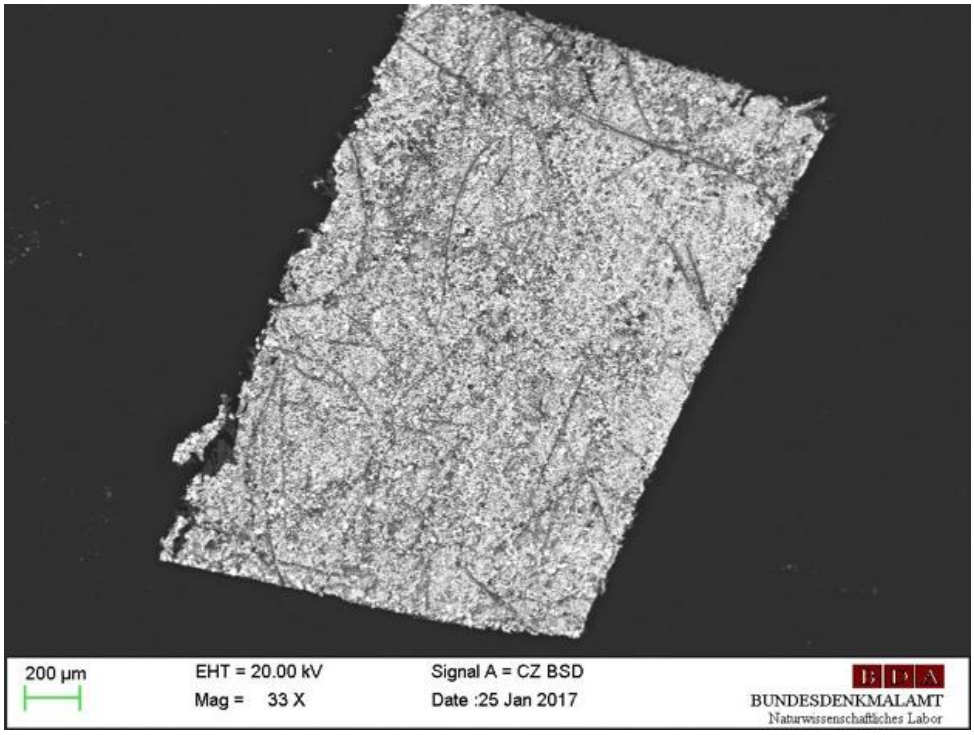
Analysis was carried out by means of scanning electron microscopy (Zeiss, EVO MA 15) equipped with an energy dispersive X-ray fluorescence detector (Oxford, INCA dry-cool) in order to study the application of the red pigment, as well as its chemical composition.

The precise pest species that caused the damage cannot currently be determined, but the tunnelling clearly indicates beetle larvae feeding within the book [36–42]. A seven-year study by the National Archives of China documenting the distribution and types of insects posing threats to library collections in China notes that Guangdong province, located across from Hong Kong's northern border, is especially vulnerable, with 21 different insect types threatening archives and museums.

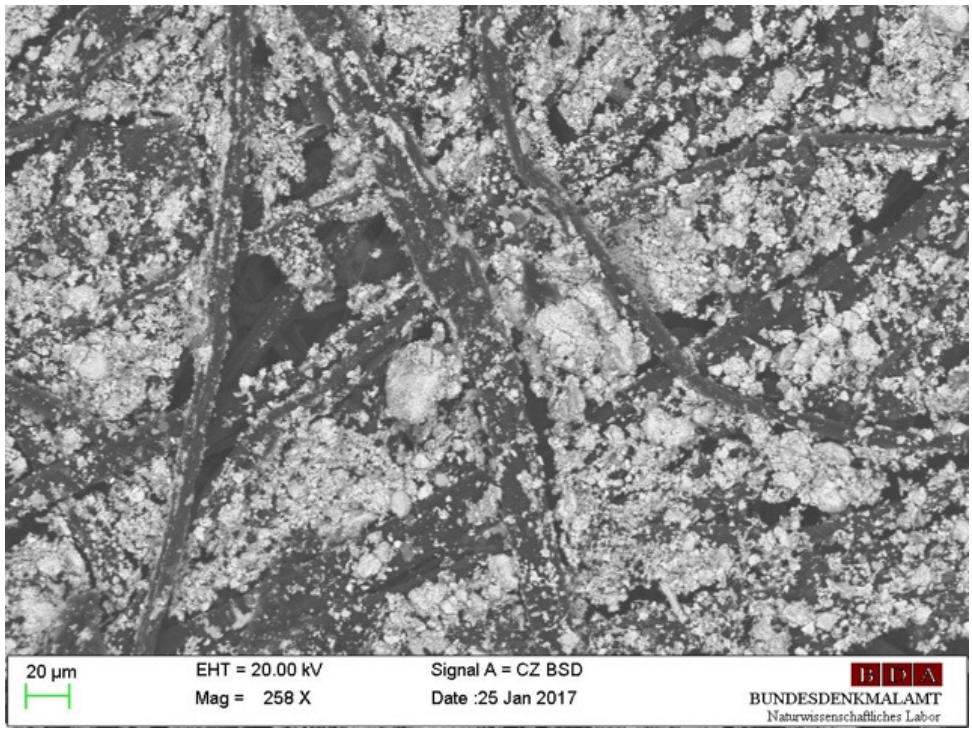
3. Results

3.1. Undamaged ORANGE Papers

The results reveal clear differences in the chemical composition between damaged and undamaged paper sheets. The chemical analysis shows high concentrations of lead only in the undamaged orange papers (Figures 3 and 4), which could be attributed to read lead or lead tetroxide (Pb_3O_4). Additionally, minor amounts of barite ($BaSO_4$) and calcium carbonate ($CaCO_3$) were found. These materials were most likely used as filling material. Trace elements such as Cu (copper) and Sn (tin) were also identified, but their exact purpose has not yet been interpreted. Figure 3a–c show SEM pictures at different magnifications of two nearly identical samples. In Figure 3c, crystals of red lead mixed with calcium carbonate and barite are visible. All undamaged samples revealed the same chemical composition and surface structure.

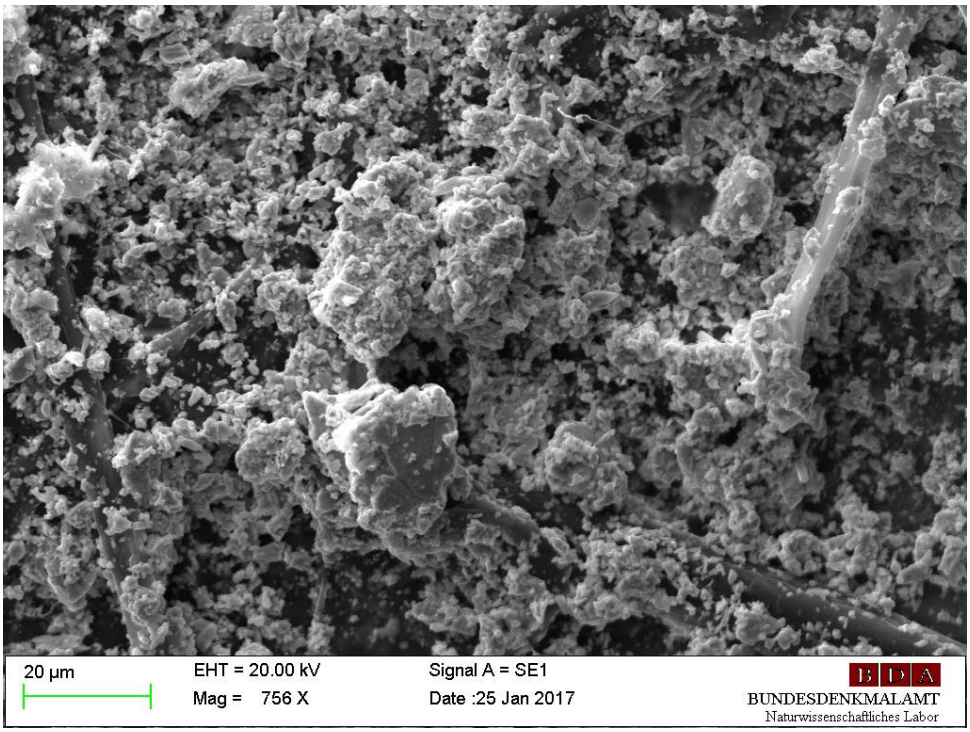


(**a**)



(**b**)

Figure 3. *Cont.*



(**c**)

Figure 3. (**a**,**b**): SEM-BE images of samples no. 84 and 85. (**c**) The SEM-SE image shows a particle size up to 10 µm, sample no. 84.

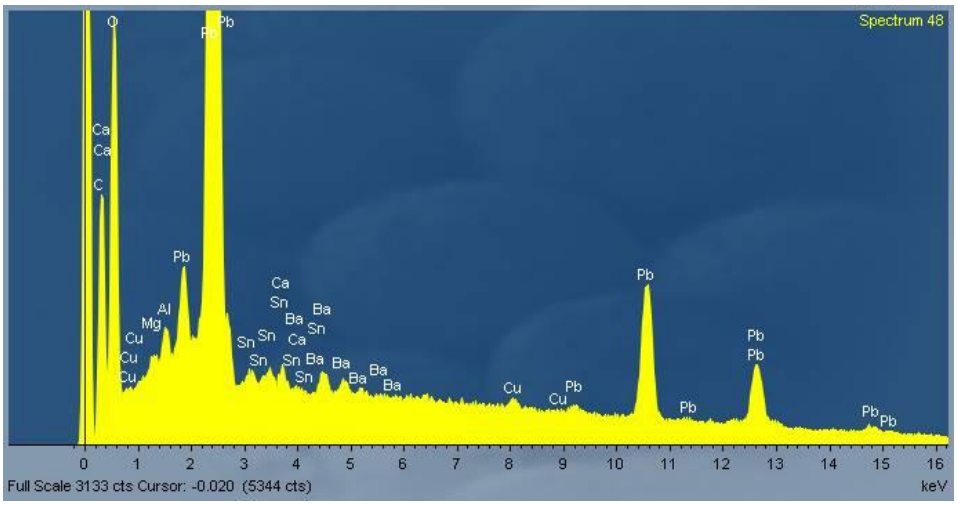


Figure 4. The EDX-spectrum of the surface of sample no. 84.

3.2. Damaged ORANGE Papers

Chemical analysis carried out on the surface of the damaged paper sheets shows an absence of lead or any other toxic element such as Hg (mercury) or As (arsenic) (Figure 5). No inorganic pigments were detected in these samples. Therefore, high concentrations of Ba (barium) suggest that barite was used as a substrate for an organic dye. Additionally, elements such as Na (sodium), Al (aluminium), K (potassium), and Ca (calcium) were found in minor concentrations, which could be interpreted as natural impurities connected to the production of the paper sheets. The surface structure of the damaged orange paper (Figure 6b,c) looks very different for the undamaged one (Figure 3c), without crystals of red lead, calcium carbonate, and barite. All damaged samples revealed the same chemical composition and surface structure.

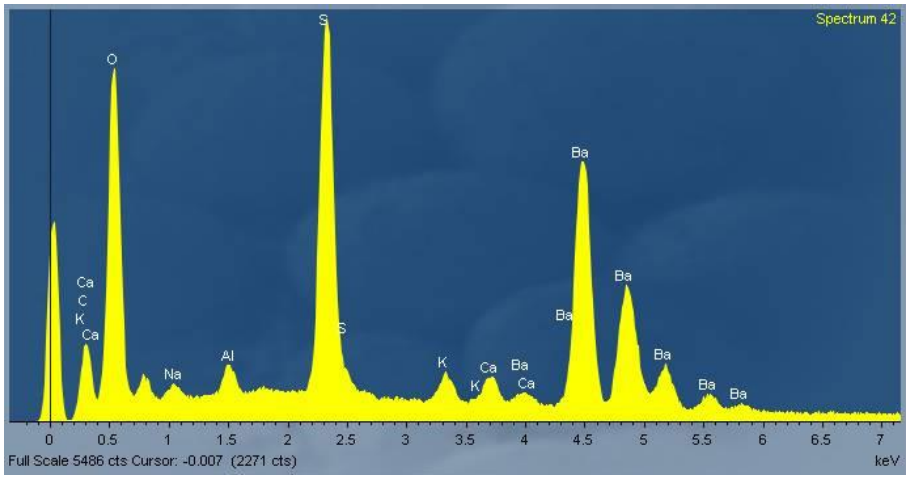
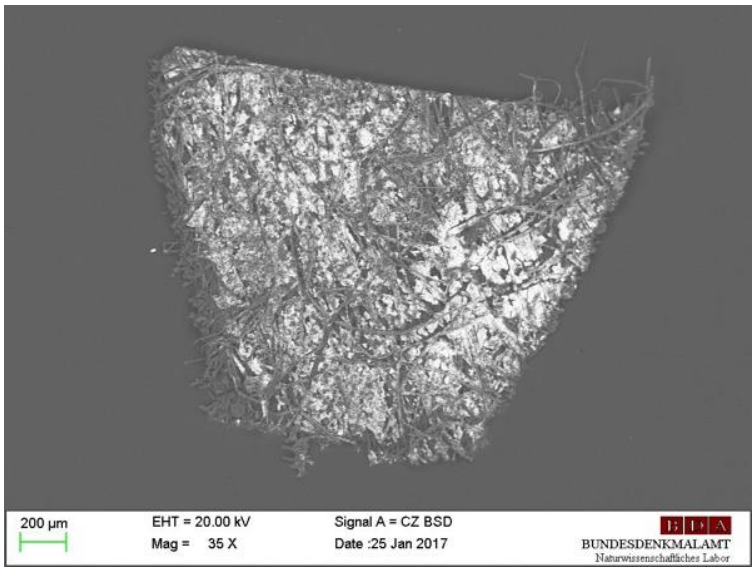
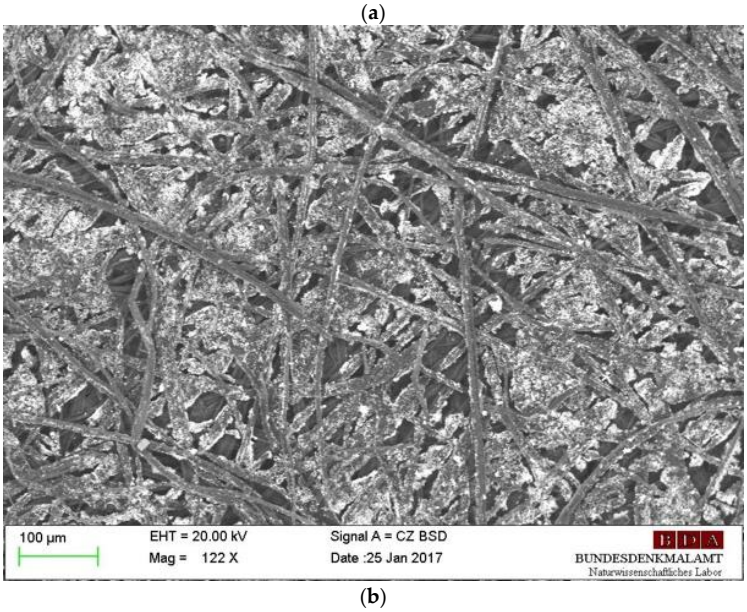


Figure 5. EDX-spectrum of sample no. 88 shows high concentrations of Ba.

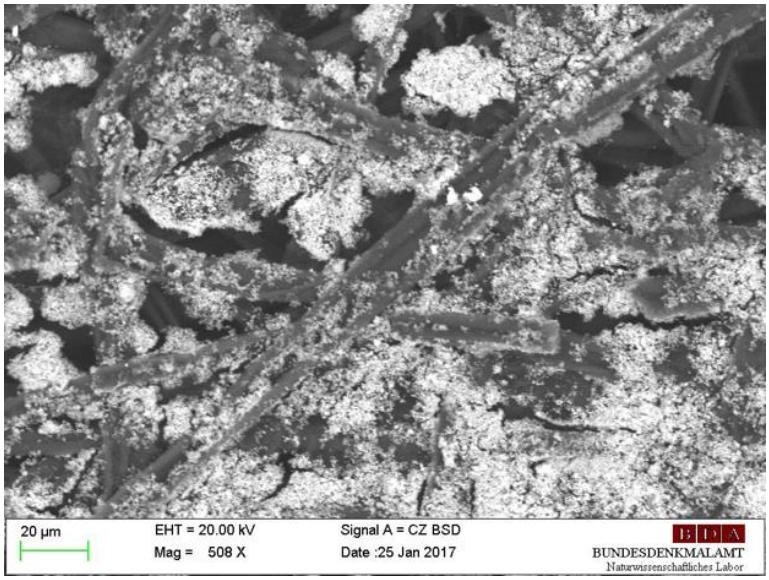


(**a**)



(**b**)

Figure 6. *Cont.*



(**c**)

Figure 6. (**a**,**b**): SEM-BE images of samples no. 89 and 88. (**c**): Compared to Figure 3c, the SEM-BE image of sample no. 89 shows a smaller grain size.

4. Discussion

Some of the orange papers in the Fung Ping Shan collection have not proven effective, as is evidenced by the tunnels of insect damage across the orange sheets. We can show that red lead was not used in the manufacture of these endsheets, which would seem to indicate that the manufacturers used other red pigments to produce a fraudulent range of insecticidal papers.

No conclusions yet have been drawn as to why a "non-toxic", and therefore ineffective, substitute was used. It is possible that the craftsmen knew about the toxicity of red lead and were attempting to protect themselves. At least in Europe, the toxicity of pigments that contain lead was well known in the nineteenth century [43]. It certainly would have been safer and less expensive to apply non-lead-based solutions to the paper.

Red lead is a highly toxic mineral that has been used as a pigment since ancient times—referenced by Dioscorides, Vitruvius, and Pliny the Elder [44]. The use of lead-containing pigments in China was reported as early as the 5th century BCE [45]. Remarkably, it has been proven that at that time in China, the pigment was artificially produced from heating white lead; there appears to be no evidence that the natural mineral was used [46]. Red lead is known to be carcinogenic, mutagenic, and toxic to reproduction, which has resulted in its present use being highly restricted. Italian Renaissance panel paintings had red lead applied on the reverse side as a protection against wood-boring insects. Panels sealed with red lead have survived in better condition than those left untreated [47]. The red insecticidal paper is limited in effectiveness, as most often it is only used on the endsheets, leaving the centre of the textblock vulnerable to attack. In addition, the preparation of the solution involves handling toxic elements harmful to humans.

4.1. Traditional Treatment Methods and Current IPM Methods in China and Asia Today

In China, other methods of natural products were used over the centuries against insect pests (and fungi). See J. Beenk for an overview of methods that include camphor, pepper, and burnished wax [28]. However, orange papers are the most obvious and, especially in libraries in the south of China, very widespread. We assume that if the orange papers are still in place and the lead containing pigments were used, they still protect the books in a traditional way.

Another example for traditional methods to protect books from insect damage are libraries in Sri Lanka, India, and other Asian countries, that hold large collections of the

ancient traditional Sinhala writing. Here palm leaves are used for manuscripts [48–53] and a traditional method (yearly or regular application of oils) for the conservation of these manuscripts was applied. Plarre et al. analysed the biocidal properties of these natural and traditional [54] oils and found clear repellent and killing effects on the termites investigated.

Today in Asia, pyrethroid fumigations are used widely in urban areas, especially for pests like cockroaches, ants, mosquitos, or silverfish, and are therefore also used in many libraries and museums. There is also a need to control urban pests, like cockroaches or mosquitos, which are known vectors of tropical disease. However, pyrethroid fumigations are not 100% effective against silverfish or biscuit beetle infestations, and a re-infestation likely will occur.

Many Asian libraries and archives still lack an IPM program, and actions are focused primarily on treatment (reaction). We suggest putting more time, education, and resources into prevention in order to successfully protect Asia's valuable collections.

4.2. Current IPM Methods and Treatment of Books in Europe Today

In Europe, the treatment of paper against insect pests is not widely documented. Compared to wooden, ethnographic or natural history objects—which have been treated for many decades with numerous biocides (Lindane for example)—books and paper were largely untreated, resulting in a significant number of damaged books in historic libraries (personal observations). An exception to this is the use of mercury-based biocides in herbaria albums and archives [55]. During major infestations, the entire library, including the books and wooden shelves, were treated with methyl bromide and hydrogen cyanide in the past, or more recently by means of sulfuryl fluoride. One case is known in Austria where the so-called pigment paris green [56], in German "Schweinfurter Grün", was applied to passepartout cardboard in the Albertina collection. It is not yet known if it was applied to protect against fungi, insects, or for some alternative reason (Ida Rupp, Albertina collection, oral communication).

Integrated Pest Management (IPM) is now the standard method used across the GLAM sector (Galleries, Libraries, Archives, and Museums) in Europe, North America, and Australia [2–9]. Significant resources are spent on monitoring collections [57–64] and on preventive measures like sealing the building, good housekeeping, and quarantining infected objects or new acquisitions.

Insect pests still occur in European libraries. If an infestation is found, a variety of non-chemical methods are employed, such as freezing individual books or entire collections. For delicate and extremely precious books, anoxic treatments using nitrogen are the preferred method. If only a few books are infested, anoxic treatment with oxygen scavengers in small bags (Figure 7) is a good option (easy to use without an external pest control company). A biological control using parasitoid wasps against biscuit beetles was successfully documented in one museum storage depository [25], but not yet in a historic library [64]. Sulfuryl fluoride has been used twice in the last 15 years to treat two large historic libraries in Austria. To our knowledge, in Asia this biocide is only registered for use in Japan. Recently, an increased problem with grey silverfish has led to research [65] and the development of different poisonous bait gels. The ADVION gel, originally manufactured as a bait for ants and cockroaches, is now used to combat grey silverfish in European countries such as Norway, Austria, Germany, the Netherlands, and Switzerland [66,67].

Silverfish, book lice, and some of the beetles require humidity levels of 70% RH or higher. Regulating the building's climate is an IPM method to reduce pest development or the risk of infestation. Regularly cleaning objects and rooms (good housekeeping) in order to reduce dust, microscopic fungi, and other forms of organic matter are further key steps to halting the spread of pests like silverfish and booklice. It is inevitable that a small number of silverfish and book lice will be found in an interior space, and small numbers can be tolerated. However, any sighting of termite activity, biscuit-, cigarette-, or other beetles is cause for alarm; infested objects must be immediately quarantined and treated to prevent the spread of further damage.

Figure 7. Treatment of historic books in the library of the monastery Altenburg with oxygen scavengers (ZerO2) in 2018. Photo Pascal Querner.

5. Conclusions

Some of the orange papers in the Fung Ping Shan collection in Hong Kong have not proven effective, as is evidenced by the tunnels of insect damage across the orange sheets. We can show that red lead was not used in the manufacture of these endsheets. The undamaged paper on the contrary has insecticidal properties and protected the pages of the books over centuries. Today this protection is probably still active as the red lead was found by us in high concentrations. Libraries, archives, and museums in Europe today try to reduce the application of new biocides and follow an Integrated Pest Management (IPM) protocol that focuses on prevention, monitoring, and quarantine and uses primarily nonchemical treatment methods. However, tropical conditions pose additional challenges for the conservation of paper and books, as high temperature and humidity and the fast development of pests and species like termites can rapidly damage collections.

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