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Original article

Analysis of efflorescence on surface of beeswax seals

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ABSTRACT

Thirteen samples of an efflorescence collected from the surface of beeswax seals or from beeswax used for their restoration were analysed in detail. The samples were of different origin, age and storage history. The composition and the incidence of the efflorescence was correlated with a chemical composition of historical and recent beeswax samples. The composition of the crystalline layer was found to be very similar in all the samples. Linear monounsaturated alkenes containing 31 and 33 carbon atoms, more precisely (*Z*)-hentriacont-10-ene and (*Z*)-tritriacont-10-ene, were identified as main components. The analyses of beeswax have revealed that all compounds identified in "wax bloom" naturally occur in recent beeswax. However, the historical beeswaxes contained only traces of unsaturated hydrocarbons, if anything. The efflorescence was observed primarily on the surfaces of those samples which contained larger proportion of alkenes. The presented results could be of particular importance not only for better understanding of the described phenomenon, but also for the development of new beeswax-based materials, which would be "bloom resistant" and useful for conservation purposes.

1. Research aims

The aim of this study was to analyse the efflorescence developed on the surface of beeswaxes of different origin, age and storage history. In order to correlate the occurrence and the composition of the efflorescence with chemical changes of beeswax, the analyses of samples of historic and recent beeswaxes were proposed as well. Such an approach would enable the generalization of the results to considerable extent and provide a reliable basis for a further research, focused on explanation and prevention of this phenomenon.

2. Experimental

2.1. Introduction

Conservators of historical seals made of beeswax-based mixtures often meet the problem of the so-called "wax bloom" (Fig. 1). This term is commonly used to describe either a white crystallic layer or aggregations of crystals, which can develop over weeks or months on the newly created surface of beeswax. It can be found primarily on beeswax used for the reconstruction of damaged seals in the course of a conservation treatment, and, less often, on historical seals themselves.

Conservators are often asked to remove it, as it can make the seal image illegible and is usually considered to be aesthetically unacceptable. In addition, the compounds forming the efflorescence are sometimes regarded as plastifiers of beeswax, and their depletion as a cause of embrittlement of the seals during ageing. However, the removal of the "wax bloom" is not always an easy task and can lead to damage of the original surface of the seal. The problem is not limited to seals; these crystals can develop also on the surface of beeswax plastics, candles, encaustic paintings, beeswax-based protective layers, and more [1–4].

The mechanism behind the formation of this efflorescence is still a matter of discussion. Most often, it is considered to be a product of a physicochemical process, rather than of a chemical reaction [5,6]. Various factors are suspected to promote its development: extreme temperatures [2,7,8], fluctuations in temperature [1,3,5], higher cooling rates during solidification [5], lower or higher relative humidity [2,6], the presence of impurities in the beeswax [9], degradation processes [4] and the presence of admixtures such as stearine or resins [1,3,10], to name a few.

Several authors have published the results of analyses of these crystals, which were collected from the surfaces of historical seals or various types of wax museum artifacts. However, almost all of these studies dealt with only a few samples, leaving uncertain the extent to which the generalization of their results is possible.

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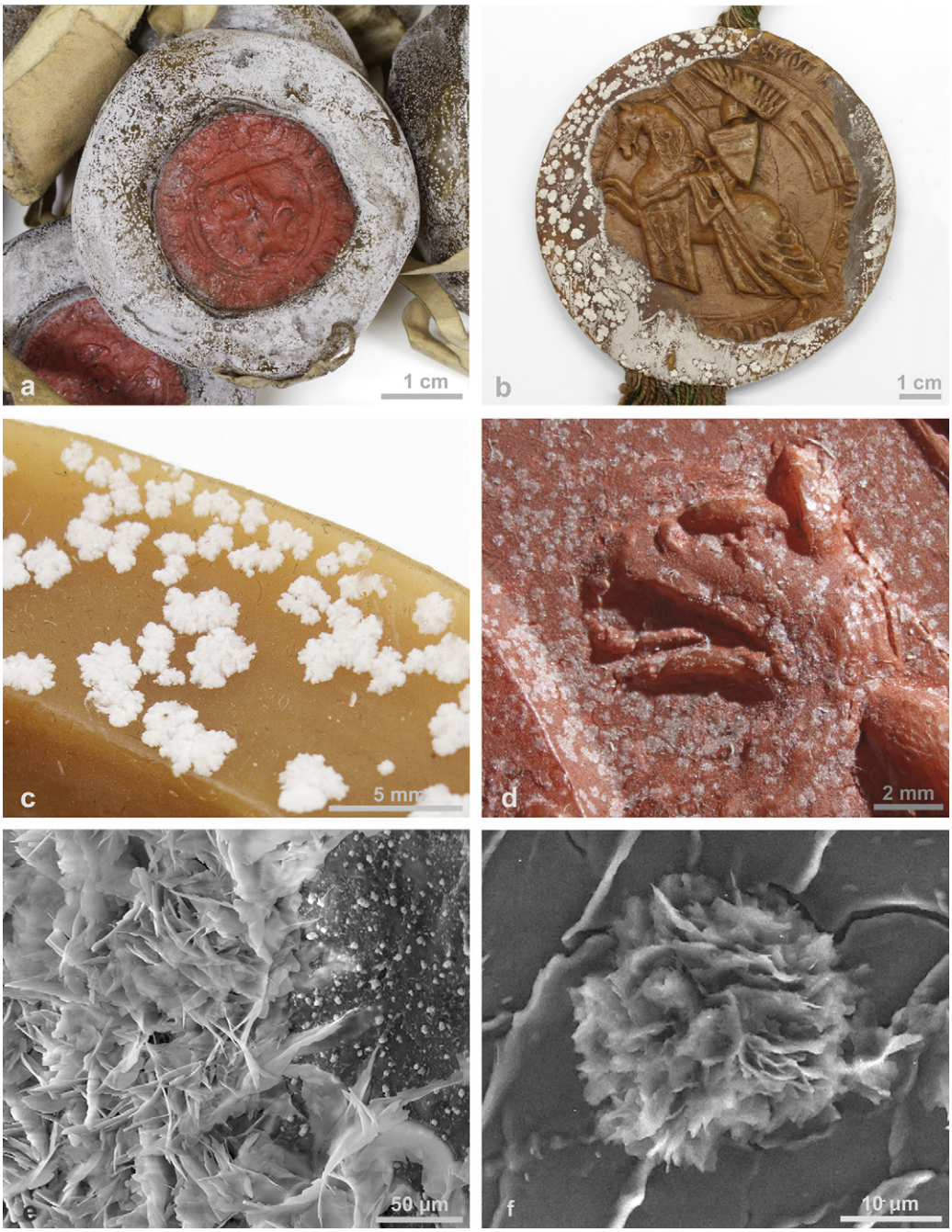

Fig. 1. a: efflorescence on the surface of beeswax seals dated from 1374; b: efflorescence on the surface of beeswax repair made in 1987; c: detail of crystal development on the surface of repair made in 1990; d: detail of crystal growth on the surface of a red-coloured beeswax seal dated from 1867; e and f: scanning electron microscope images of crystals on the surface of recent beeswax (TESCAN VEGA3, SEM HV: 20 kV, BSE mode).

In some cases, aliphatic hydrocarbons naturally occurring in beeswax itself were identified as the main components of the "wax bloom" [3,7]. Novotná specified these compounds as alkenes containing 31, 32, 33 and 35 carbon atoms. It was suggested that their migration to the surface could be promoted by the recrystallization of beeswax at lower temperatures, especially below a "critical" temperature interval of 12 to 16 °C [7]. Similarly, Bacílek described the efflorescence process as an outflow of more volatile unsaturated compounds characterised by the melting point about 38 °C [11].

However, in other samples, almost exclusively fatty acids, especially palmitic and stearic acids [1–3], have been identified. These compounds can also be detected in pure beeswax. According to some experts, their higher content can be a consequence of the hydrolysis of wax esters during the natural ageing of beeswax or due to stearine admixtures [3,12].

Some authors have also reported the occurrence of efflorescence which consists mainly of beeswax itself, sometimes in combination with a small amount of water [6,8].

Considering the variability of these results, the question arises whether the composition of crystals from similar material can actually vary: for example, whether it depends on the storage conditions, or whether it is connected mainly with a content of specific admixtures [3,8]. The primary aim of this study is to gain deeper insight on the differences between the chemical composition of various samples of efflorescence on beeswax