ORIGINAL ARTICLE

Degradation marks of phonograph cylinders from Tesař's opera collection

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Phonograph cylinders are the oldest commercially available recording media and are an valuable part of cultural institutions' collections. Even though they are an essential part of sound history, they have long been overlooked as a relatively chemically stable media. The only degradation mentioned in the literature was a whitish cover, traditionally appointed to microbiological growth. In our study, we focused on unravelling the issue of these degradation products. A selected collection from the National Museum – Czech Museum of Music was subjected to microbiological and analytical examination via digital microscopy, FTIR spectroscopy, colourimetry and SEM-EDS analysis. From the results, some of the degradation pathways were proposed. The results also help us to focus future research and suppress the degradation so that the cylinders stay longer in conditions fit for digitisation and overall archivation.

INTRODUCTION

Phonograph cylinders represent the earliest mass-produced sound recording medium. However, despite forming part of numerous museum, library, and archive collections, they have not been paid due attention by researchers. The sound is captured in a helical groove cut on the outer side of the cylinder by a stylus placed perpendicular to the cylinder surface, creating a monophonic recording. This process is also referred to as the "hill and dale process". The principle of phonograph cylinder recording was patented at the turn of the years in 1877–1878 by Thomas Alva Edison, who followed up on acoustic experiments carried out by various experimenters (such as Édouard-Léon Scott de Martinville) in the previous decades. [1]. Initially, Edison's phonograph captured sound by pressing cutting soundwaves into a tin foil wrapped around a rotating metal cylinder. Problems with manipulation and reproduction triggered the search for more efficient methods and suitable recording materials. Moreover, the significant progress made by Charles S. Tainter and Chichester Bell, Edison's rivals from the Volta Laboratory and inventors of a wax-coated cardboard cylinder, inspired the latter to innovate his patent [2].

Phonograph cylinders were slowly distributed for commercial use after 1887-88 and were then mass-produced from the 1890s for almost another 40 years (until 1929). Various types exist, which differ significantly in terms of material and physical and acoustic parameters. This paper focuses on the first type suitable for mass production – the brown-wax cylinder.

Despite being widely referred to as "brown-wax cylinders", they are not made purely out of wax – unlike the older, experimental type [3]. The mixture of animal, plant and mineral wax was replaced mainly by a mixture of fatty acids and their salts (metallic soaps). The most frequently used acids were stearic and palmitic acid and their sodium and aluminium salts. The wax constituent was preserved in the form of ceresin. Despite this, the word "wax" is still used to refer to the cylinders reflecting the fact that their organoleptic and processing properties, as well as their appearance, are similar to those of "true" wax cylinders [3-5].

Within our experiment, a unique set of brown-wax phonograph cylinders from the collection of the National Museum – Czech Museum of Music (NM-ČMH) was examined.

The cylinders contain one of the most valuable collections of recordings of this type in the Czech Republic, sometimes called "Tesař's Opera". The term "Tesař Opera" refers to an amateur and probably informal artist association founded in the late 1890s, the members of which convened in the pubs run by Josef Tesař (1844–1904). [6]. The association brought together amateur and professional singers and musicians from

The Prague National Theatre and its surroundings, among them prominent artists such as Emil Burian, a singer and the brother of the famous singer Karel Burian [6, 7].

While the origins are uncertain, the outcome is a unique set of recordings of Czech music of the early 20th century, featuring over a dozen male and female singers, famous and unknown. The collection is nowadays located in the NM-ČMH phonothèque. The phonothèque holds over 1300 phonograph cylinders; according to Meszner (1985), 179 contain so-called "Tesař's Opera" recordings. Recordings were probably made between 1902-1905; however, the brown wax cylinders' production dates cannot be ascertained, mainly because they were used as "blanks" and repeatedly, and the original recording could be overwritten. This means that the actual cylinder does not necessarily date from the same time as the recording.

The set primarily consists of brown-wax phonograph cylinders. However, some moulded cylinders from black wax can also be found. They were produced later (as the following commercial type of cylinders after 1902) and shared many technological parameters with their brown-wax counterparts.

This paper focuses on brown-wax cylinders and their degradation, troubling all major cultural institutions with phonographic cylinders in their collections. The characteristic whitish cover on the cylinder is usually looked upon as a microbiological contamination. The objects are therefore moved into a dry storage facility and not further examined. An exception in this approach was the material examination of Monroe et al. [5]. However, to our knowledge, no systematic study covering material and microbiological examination has been published. For the prolonged life of phonographic cylinders is vital to know the degradation process behind to suppress its rate. In this paper, we focused on the optical, material, and microbiological analysis of phonographic cylinders and their degradation products to bring closer the uncovering of the degradation process behind them.

MATERIALS AND METHODS

Samples selection

A total of 20 samples from the collection of the so-called "Tesař's opera" were selected for examination (Table 1). The selected cylinders were of different shades of brown. Degradation products had whitish colour and different macroscopic appearances.

Selected cylinders have been categorised according to their macroscopic degradation pattern. In some cases, different-looking degradations could be found on one cylinder.

Tab. 1. Selected phonographic cylinders of the Czech museum of music phonotèque from the collection of the so-called "Tesař's opera"

Call number	Title	Interpreter
MH 0051	Faust Duetto	Krampera, Čihák – singing
MH 0052	Kolíne…	Krampera – singing
MH 0053	Mý zlatý rodiče	Krampera – singing
MH 0054	Chodíval nám – Kamaráde nepí	Krampera – singing
MH 0055	V tom našem sadečku	Krampera – singing
MH 0066	Teče voda proti vodě	Mařák – singing
MH 0089	Probuzení jara 4	–
MH 0094	Andantino 2. concert	–
MH 0095	Martha	–
MH 0150	Píšťadlička	Geitler
MH 0231	Cant Ruse	–
MH 0267	Werther – Osiana	Tondl
MH 0459	Šveska – Wolfiáda	–
MH 0571	Rigoletto	–
MH 0613	„Už mou milou"	Viktorin
MH 0710	Hej slované, Na Marjánce	–
MH 0711	Lysistrata: Píseň o světlušce	Guthová
MH 0727	Čarostřelec	–
MH 0750	Santa Lucia	Mottas
MH 0970	Na Bledsko jezero	Bráč; Vítek

Microbiological examination

The microbiological examination proceeded with a specific swabbing method developed for abrasion-sensitive materials [8]. The polyurethane swabbing sponge (PUR-Blue Swab Sampler with Dry Large Tip Swab, World Bioproducts, USA) was gently rolled over the whole surface of the phonographic cylinder. The sponge was then gently rolled crosswise over the surface of Petri plates (diameter 90 mm) filled with solidified culture media, selected for fungi, bacteria and yeast cultivation.

The cultivation was performed on MEA (Malt extract agar, Oxoid, UK) for yeasts and fungi. For the xerophilic fungi and osmophilic yeasts (for material with a water activity of 0.95 or lower), the DG18 was used (Dichloran-glycerol agar base, Oxoid, UK). The cultivation of these media proceeded at 25 °C for 10-30 days. The primary cultivation for bacteria was performed on PCA (Plate Count Agar, Merck, DE) at 30 °C for 3-5 days.

After this primary cultivation, the colony-forming units (CFU) of fungi grown on MEA and DG18 and CFU of bacteria grown on PCA were identified based on their phenotype: colony morphology, microscopic characteristics and basic biochemical tests.

Digital microscopy

All cylinders with their degradation patterns were examined by digital microscopy (VHX 950, Keyence, Japan) in part and full illum light modes. Microscopic documentation was made at the magnification of 250×, 500× and 1000×. For 500× and 1000×, 3D topography was measured.

Scanning Electron Microscopy (SEM)

For the examination of degradation products under electron microscopy, the scanning electron microscope JEOL 6460 was used. Ca 5 mg sample was taken from one phonographic cylinder from each degradation pattern group. These micro-samples were placed onto carbon tape on an aluminium carrier. The surface gilding (99.99% Au) of the samples was carried out by sputtering on a JEOL JFC-1300 automated sputter coater. The sputtering produced a 10-15 nm thick metal layer on the sample's surface. Documentation of the samples was taken in secondary and backscattered electron (SE, BSE) mode using magnifications of 500×, 1000×, 2000× and 5000× in high vacuum mode with an accelerating voltage of 15 kV. The surface morphology was observed in the secondary electron mode. The backscattered electron mode provided information about the elemental composition of the surface in addition to the morphology.

Colourimetry

The brown wax phonographic cylinders exist in many shades of brown. Even though they were roughly divided as light/dark, the exact colourimetric measurements were made to see if there was any correlation between shade and type/extent of degradation.

The colour was measured at ten places on the cylinder avoiding the degradation patterns using a spectrometer Konica Minolta CM-700d in the CIELAB colour space. The geometry of cylinders was taken into account – the opening of 5 mm in diameter was chosen and always centered for the contact to be as good as possible.

Fourier-transform infrared spectroscopy (FTIR)

From each cylinder, five samples of a matrix (size ca 1-2 mm^3 without degradation signs – usually from the inside helix) were taken. To analyse the degradation

Tab. 2. Microbiological contamination of phonographic cylinders

Call number	DG18	MEA	PCA	Fungi (CFU)	Bacteria (CFU)
MH 0051	–	–	Staphylococcus epidermidis	0	2
MH 0052	–	–	Sporulating bacteria Staphylococcus epidermidis	0	5
MH 0053	–	–	Sporulating bacteria Staphylococcus epidermidis	0	7
MH 0054	–	–	Sporulating bacteria	0	2
MH 0055	–	–	Sporulating bacteria	0	1
MH 0066	–	–	Sporulating bacteria	0	3
MH 0089	–	–	Sporulating bacteria	0	5
MH 0094	–	–	Sporulating bacteria	0	3
MH 0095	–	–	Sporulating bacteria Micrococci	0	3
MH 0150	–	–	–	0	0
MH 0231	Cladosporium sp.	Cladosporium sp.	–	2	0
MH 0267	–		–	0	0
MH 0459	–	Cladosporium sp.	–	3	0
MH 0571	–	Cladosporium sp.	Micrococci	4	3
MH 0613	–		–	0	0
MH 0710	Penicillium sp.	Cladosporium sp.	Cladosporium sp.	7	0
MH 0711	Penicillium sp.	Cladosporium sp.	Sporulating bacteria	5	10
MH 0727	–	–	–	0	0
MH 0750	–	–	–	0	0
MH 0970	–	–	Micrococci	0	2

products, five samples (if possible) of degradation products were taken (always outside the space where the sound is recorded).

Analysis was performed by the FTIR spectrometer Nicolet iN10 in combination with the iZ10 module with ATR adapter (with a single reflection diamond crystal), detector MCT-A cooled by liquid nitrogen. Measurement parameters: spectral range 4000-650 cm^{-1} resolution 4 cm^{-1} number of accumulated spectres 64, apodisation N-B strong. All spectra were evaluated using the Omnic 32 programme.

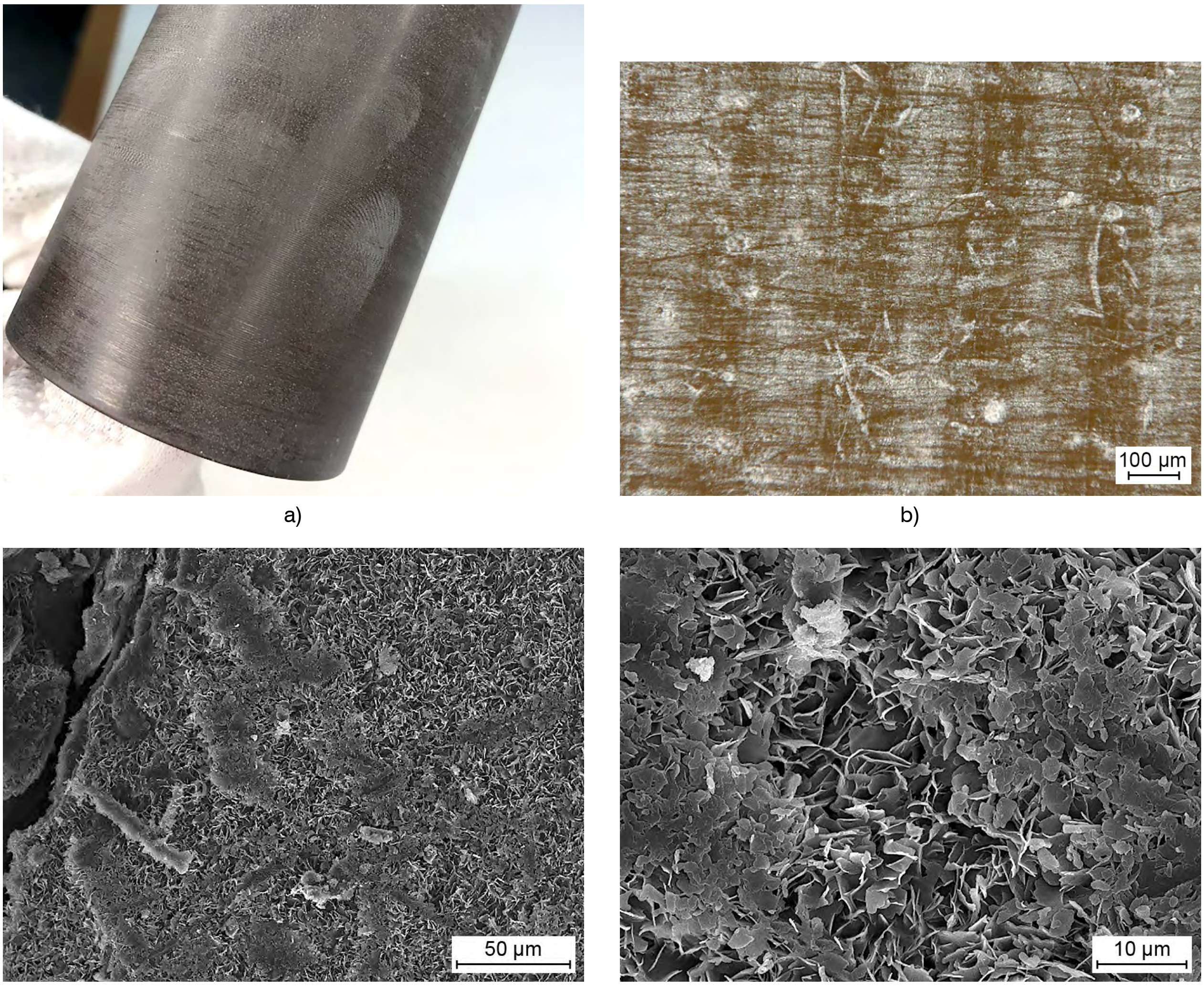
RESULTS AND DISCUSSION

Microbiological examination

Three species belonging to the regnum Fungi were isolated – *Cladosporium*, *Penicillium* and *Aspergillus*. These species are commonly isolated from both depositories and surfaces of archival and museum objects. They pose a potential threat when exposed to elevated humidity.

As for the regnum Bacteria, species associated with the human epidermis were found – mainly from improper manipulation by bare hands: micrococci and staphylococci. They are insignificant from the point of material degradation. Among sporulating bacteria, mainly the *Bacillus* species were found. It is commonly found in the overall environment, earth and dust. They have a long lifespan thanks to sporulating and high resistance against disinfection. When exposed to elevated humidity and proper nutrients, they can enter a vegetative state and cause material degradation. Specific findings and their amount (Colony forming units – CFU) are listed in Table 2.

In all cases, the microbiological contamination did not exceed the usual levels found in cultural institutions.

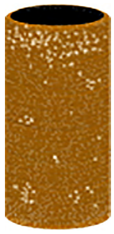
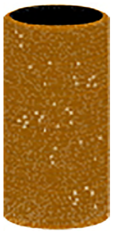


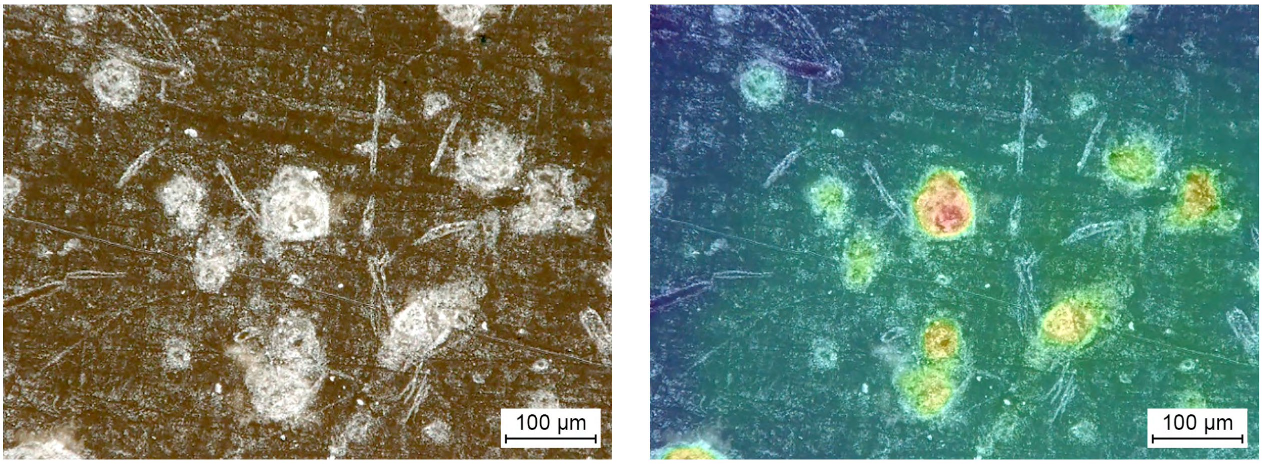
c) d)

Fig. 1. Examples of haze pattern at the sample MH 267: a) macroscopic photo, b) photo from the digital microscope under the magnification of 300×, c) SEM image (SE) under the magnification of 500×, d) SEM image (SE) under the magnification of 2000×

None of the cylinders was in a state of active microbial infestation; detected CFU numbers are very low and therefore do not pose a threat to the material or personnel. From the microbiological results, the white degradation product does not seem to be the product of microbiological contamination.

Tab. 3. Degradation of phonographic cylinders – visual description and sorting by the degradation pattern

Degradation pattern name	Description	Layout	Degradation pattern name	Description	Layout
Clean	Cylinder of one colour without any visible deviations.		Fibrous	White particles with one dimension exceeding significantly two others (by a naked eye comparison), located without any macroscopic patterns, commonly aggregated into clusters. The rim is usually more affected than other parts. The degradation is located beneath the plane of the original surface (Figure 3).	
Haze	White fog-like look all over the surface, with no distinctive particles observable by digital microscopy. Easy to remove mechanically, all behaviours show a parallel with „wax bloom" (Figure 1). The degradation found on beeswax heritage objects – extensively studied by Bartl et al. [9-11].		Circular	White particles arranged in a circular or radial pattern. Under the digital and electron microscope share the morphology with the "Fibrous" type, located beneath the plane of the original surface (Figure 4).	
Grain	Single rounded particles of white colour above the original surface of a phonographic cylinder. Located without any macroscopic pattern (Figure 2).		Compact	White particles forming a compact cover without distinguishable particles or patterns. From a light microscope not recognisable whether the layer is above or beneath the plane of the original surface of a phonographic cylinder (Figure 5). In some cases, easy to substitute for the overgrowth of one of the previously mentioned degradation patterns.	



a) b)

Fig. 2. Examples of Grain pattern: a) MH267 under the magnification of 500×, b) MH267 under the magnification of 500× with the colour visualisation of height – red part corresponds with the highest point, blue with the lowest – the Grain degradation was found only on the area with sound recording and, therefore, could not be taken as a sample to SEM analysis

a)

Visual and microscopic characterisation

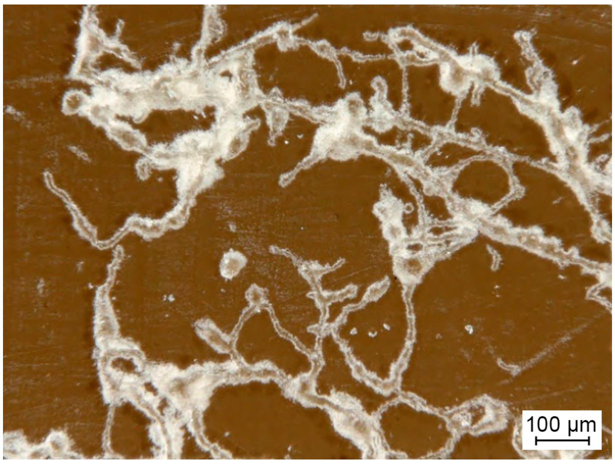
Under the digital and electron microscope observation, degradation patterns were assigned to five categories (Table 3).

The microscopic documentation shows that the degradation patterns are distinguishable and suggest different origins and pathways.

The HAZE is formed by particles similar to the wax bloom phenomena; therefore, separation of the phases with the migration of less soluble ones to the surface is suggested. As for the beeswax, the trouble may have a solution in appropriate storing.

The GRAIN degradation is detectable by circular bulks of the matrix being separated. As in HAZE, the degradation product is clearly above the plane of the original surface and, therefore, may be considered removed if necessary.

The FIBROUS and CIRCULAR degradations show several similar characteristics under the microscopic



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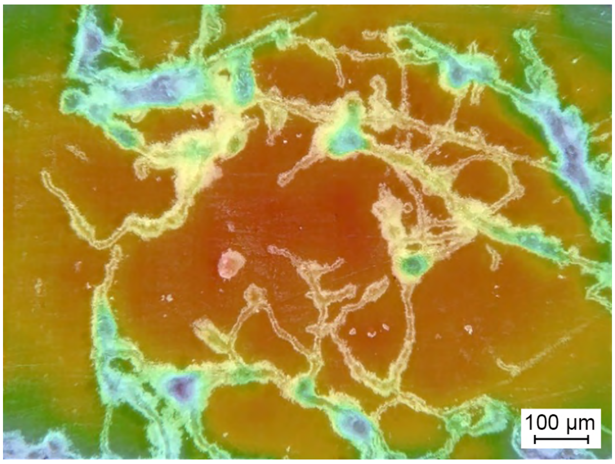

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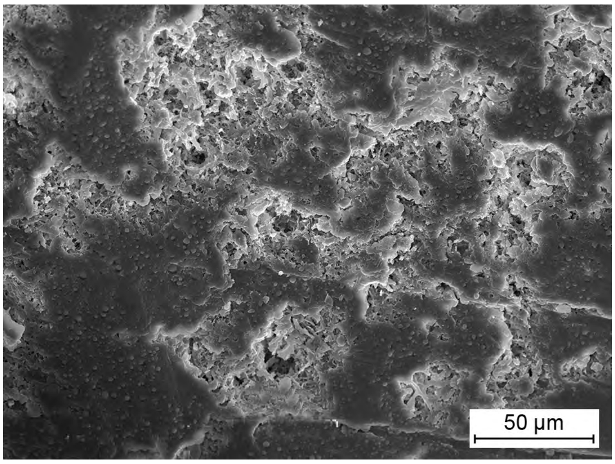

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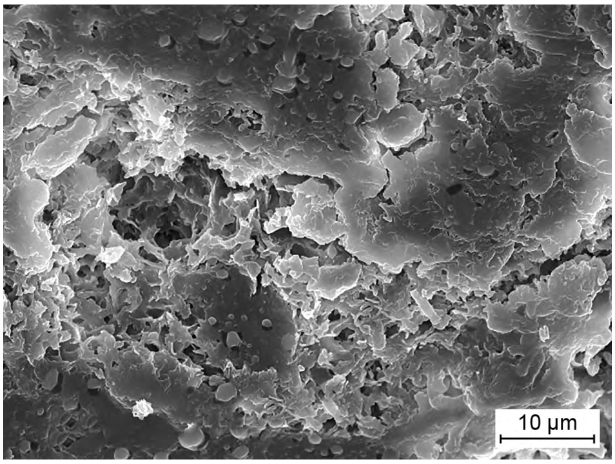


e)

Fig. 3. Examples of Fibrous pattern: a) MH 0095 macroscopic photo, b) MH 0459 under the magnification of 500×, c) MH 0459 under the magnification of 500× with the colour visualisation of height – red part corresponds with the highest point, blue with the lowest, d) SEM image (SE) under the magnification of 500×, e) SEM image (SE) under the magnification of 2000×

view. However, they are distinguishable by their macroscopic patterns. Both form cracks in the matrix; therefore, it is impossible to remove them. Around and inside the cracks, the scaling of the material is visible. In some cases, the spheric/globular bulk separation was also observed.

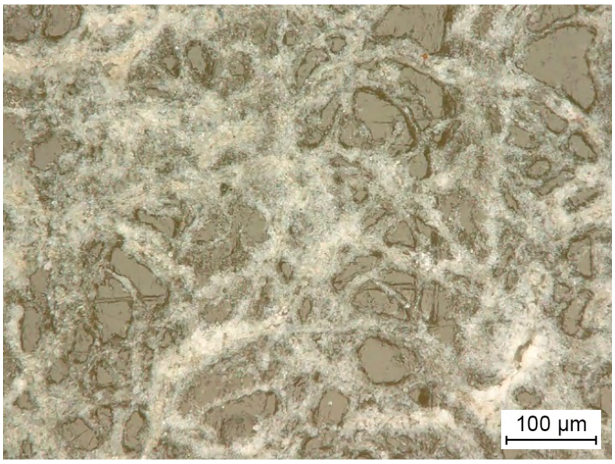
The COMPACT pattern is sometimes harder to distinguish from the other patterns simply overgrowing into the dense crust but is distinguishable under the light or electron microscope. Its origin seems tied with the lamellar crystallisation of a matrix's component and spheric/globular bulk separation and loss.

Colourimetry

The impact of the cylinder's colour on the degradation pattern was tested. Cylinders were assigned to four different colour-shade categories (Table 4).

No observable trend of the colour shade and degradation pattern was observed in the selected phonographic cylinders. Since the colour shade is more associated with the heating of the material during casting than with the change in the material formulation, it is safe to say that temperature fluctuations during production are not the case of particular degradation. The degradation is,

a)



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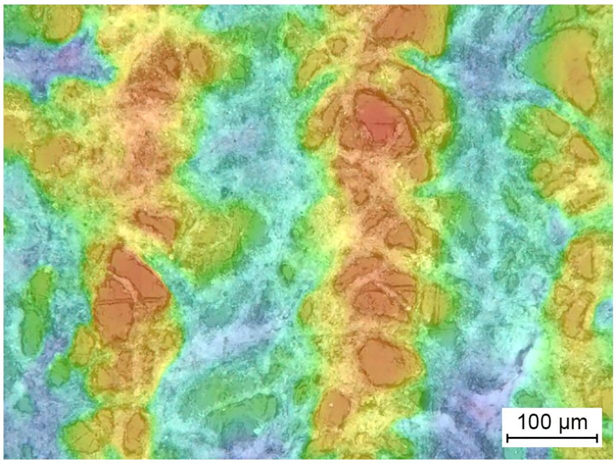

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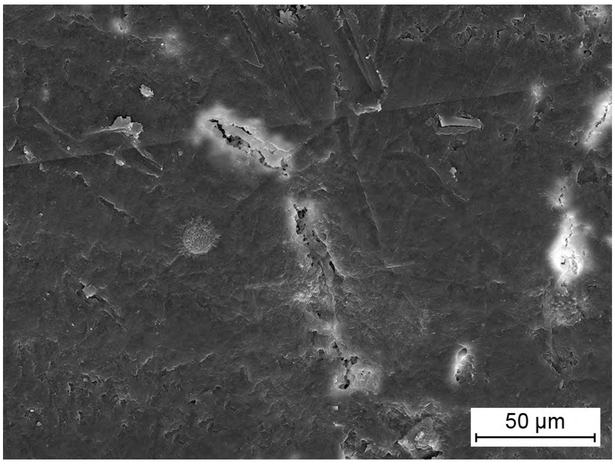

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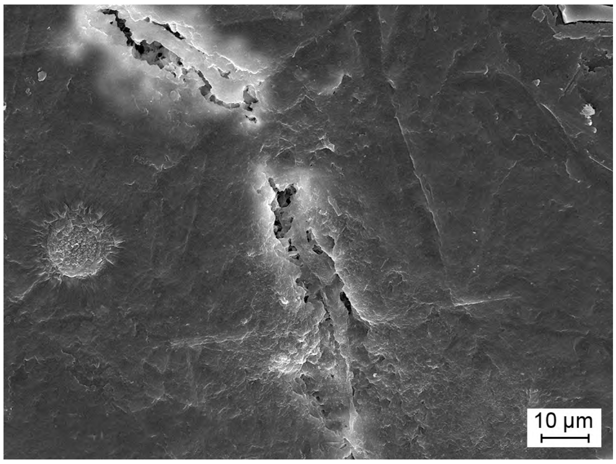

e)

Fig. 4. Examples of Circular pattern: a) MH 0054 and MH 0089 macroscopic photo, b) MH 0054 under the magnification of 500×, c) MH 0054 under the magnification of 500× with the colour visualisation of height – red part corresponds with the highest point, blue with the lowest – expressing the degradation is under the plane of the original surface, d) SEM image (SE) under the magnification of 500×, e) SEM image (SE) under the magnification of 2000×

therefore, more possible to connect with the change in chemical composition or storage conditions. Cylinder's colour and degradation pattern are filled in in the following table (Table 5).

Tab. 4. Colour shade categories of "brown wax "phonographic cylinders

D*	a*	b*	Category
> 40	> 8	> 15	A
40 – 35	8 – 6	15 – 10	B
35 – 30	6 – 4	10 – 6	C
< 30	< 4	< 6	D

Infrared spectroscopy with Fourier transformation (FTIR)

The spectroscopic analysis of matrices corresponded with the literature findings [3-5]. The results of the analysis confirmed mainly the presence of fatty acids and their salts – metallic soaps (Figure 6).

The differences in the matrix of selected cylinders lay mainly in the degree and type of saponification of fatty acid to metallic soap, with a shift in wavelength positions confirmed by several research groups [12-14]. These differences were also visible between the samples taken from one cylinder (Figure 7). Generally, the spectra of matrix exhibit the main differences between 1800 and 1480 cm^{-1} (Figure 8). The molecule we derive from is stearic acid, which shows the carbonyl peak around 1700 cm^{-1}. However, this band shifts towards higher frequentions as is typical for alkali metal acid soaps [12, 13]. This corresponds with the samples of matrix having the C=O peak in range of 1714-24 cm^{-1}. The region of following bands (1620-1510 cm^{-1}) changes distinctively with the structure of fatty acid soap – depending on the ratio of alkali metal ions, hydrogen and carboxylates in one molecule [14]. Since the input material was not possible to prepare within todays purity standards and since we can assume their non-ideal homogenisation it is only logical that the saponification proceeded in many different pathways.

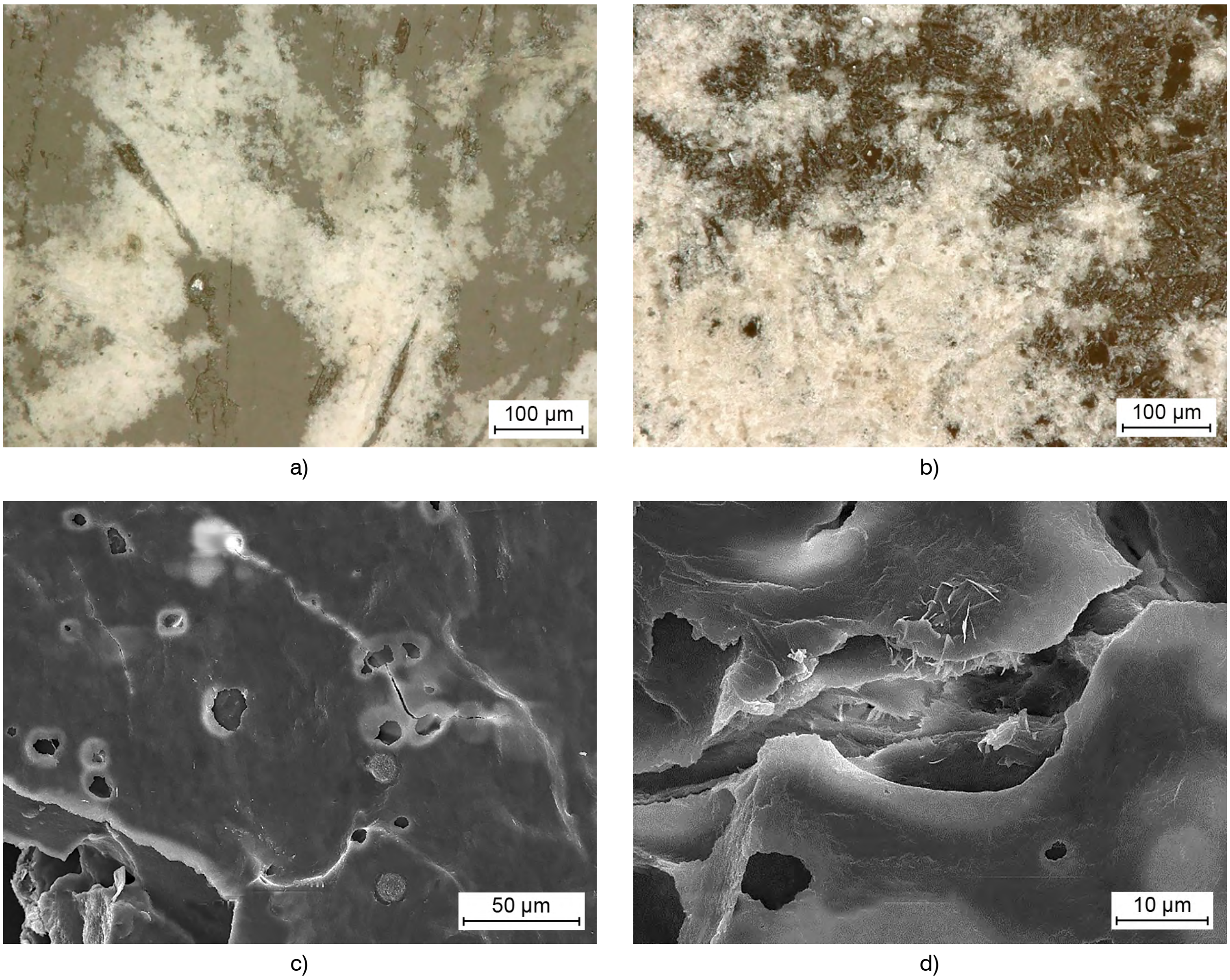

Fig. 5. Examples of Compact pattern: a) MH 0066 under the magnification of 500×, b) MH 0231 under the magnification of 500×, d) SEM image (SE) under the magnification of 500×, e) SEM image (SE) under the magnification of 2000×

Tab. 5. Selected phonographic cylinders assigned to colour and degradation pattern category

Degradation pattern	Colour shade	Call number
Haze	D	MH 0267
	A	MH 0613
Grain	D	MH 0267
Fibrous	D	MH 0051
	C	MH 0055
	B	MH 0095
	D	MH 0150
	B	MH 0459
	C	MH 0571
	A	MH 0710
	B	MH 0727
	C	MH 0750
	C	MH 0970
Circular	A	MH 0052
	C	MH 0053
	B	MH 0054
	B	MH 0089
Compact	B	MH 0066
	C	MH 0711
	B	MH 231
Clean	C	MH 0094

In 10 samples out of 20 the spectra of degradation products were distinguishable by the rising intensity of antisymmetric carboxylate stretching vibration band at 1570 cm^{-1} (Figure 9), corresponding with alkali metal monocarboxylate [13,14]. Since these molecules are much less spatially demanding than e.g. di or tricarboxylates, it is possible to expect their higher mobility in the system which can lead to crystalisation at the surface of the cylinder. To support this hypothesis, it is planned to examine the degradation products by GC-MS and elementar analysis, to get the exact information about carboxylate and metal ion.

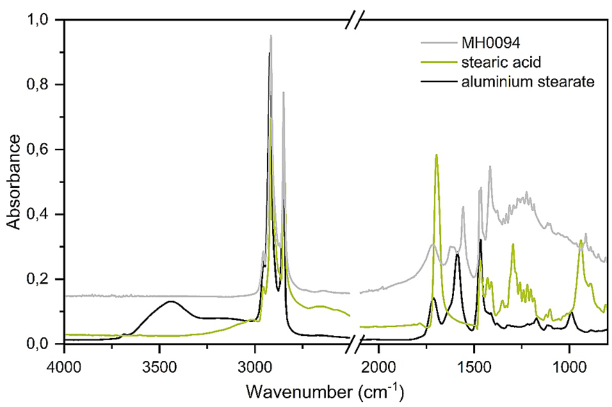

Fig. 6. Matrix of MH0094 correlated with stearic acid and aluminium stearate

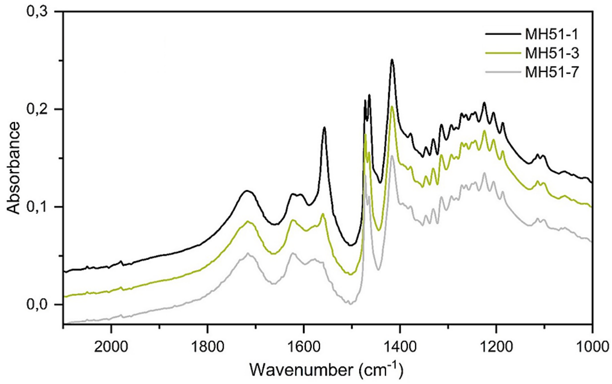

Fig. 7. Matrix samples of MH0051, showing the differences in spectral response caused by a change in the mixture and inhomogeneity within one cylinder. The changes are visible mainly in the interval of 1700-1500 cm^{-1}, corresponding mainly with the degree and type of saponification

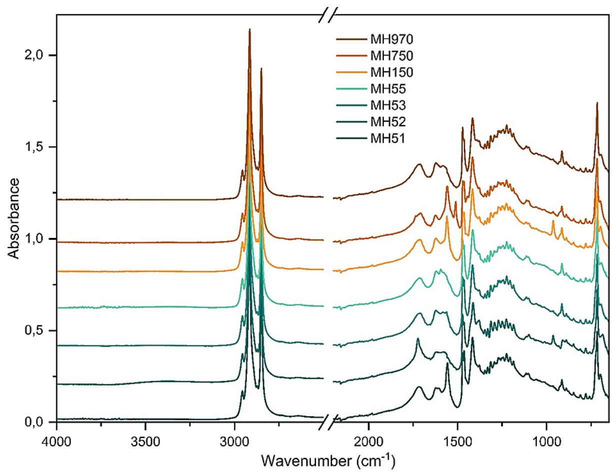

Fig. 8. The main differences in FTIR spectra of selected phonographic cylinders

In other samples, the differences between matrix and degradation product weren't visible. The spectra probably reflected the matrix below the thin layer of degradation product.

The results did not show any trend corresponding with the degradation pattern or colour shade.

The spectral results of degradation products did not correspond with the results from our previous measurements [15] or studies of other research groups. This suggests that the degradation proceeds via many pathways and is affected by the matrix material composition, manufacturing and homogenisation process and the storage conditions [16].

CONCLUSION

The degradation of brown wax phonographic cylinders is a complex problematic. From the microscopic examination, several types of degradation are observable, some of which bear similarities with the beeswax efflorescence caused by linear monounsaturated alkenes containing 31 and 33 carbon atoms [9]. The degradation patterns did not correspond with the colour shade of phonographic cylinders. The FTIR analysis showed changes in the matrix fatty acid to metallic soap ratio. However, these changes as well did not correspond with the degradation patterns. To unravel the composition of degradation products, separation methods are needed and planned to use as well as elemental analysis, giving us detailed insight into the matrix and degradation products composition.

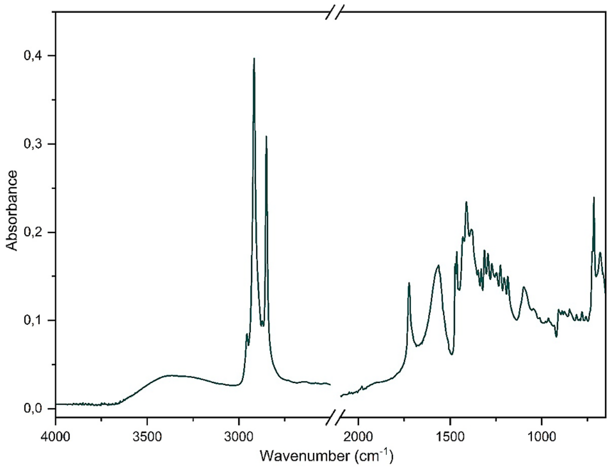

Fig. 9. Degradation product distinguishable from matrix – sample MH0231. The same spectra of degradation products were collected with samples MH0053, MH0089, MH0095, MH0150, MH0459, MH0571, MH0710, MH0711 and MH0727. The band at 1724 could correspond with acid carbonyl stretch of acid-carboxylate pairing [14]. The band of approx. 1570 corresponds with the frequency shift of antisymmetric carboxylate stretching vibration of alkali metal monocarboxylate

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