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# SEM ANALYSIS AND DEGRADATION BEHAVIOR OF CONVENTIONAL AND BIO-BASED PLASTICS DURING COMPOSTING

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## Abstract

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Recently, various materials have begun to be marketed that claim to be biodegradable or compostable. Terms such as “degradable”, “oxo-biodegradable”, “biological”, “compostable” and “green” are often used to describe and promote different plastics. Commercial bioplastics and a petrochemical plastic (claim to be degradable) were used for this study. The research was carried out in real conditions in the Central Composting Plant in Brno, Czech Republic. SEM analysis of the samples was done in order to analyze microstructure and morphology of specimens, validating dispersion results. It can be concluded that samples certified as compostable have degrade in real composting conditions. Samples (4–7) showed significant erosion on surface when subjected to the SEM analysis. Samples labeled (by their producers) as 100 % degradable (Samples 1–3) did not show any visual signs of degradation.

Keywords: bioplastics, biodegradation, real composting environment, SEM analysis

## INTRODUCTION

Problems associated with an ever-increasing amount of waste from packaging and disposable products made from conventional durable plastics have led to the search for new biodegradable packaging and disposable products (Sikorska *et al.*, 2015). Biodegradable polymers represent a promising way to reduce the amount of plastic waste disposed in landfills, with composting the preferred alternative for their disposal (Adamcová *et al.*, 2017). Many biodegradable polymers have been developed in the last two decades with the desired performance properties (Castro-Aguirre *et al.*, 2017). Thus, along with the development of these novel materials, evaluation and understanding of their biodegradation performance and their environmental impacts have become essential.

The European Waste Framework Directive recommends composting as a method of recycling post-consumer biodegradable waste. At present, a challenge is to develop composting as a disposal technology and the redirection from landfills to composting facilities. In the Europe packaging and packaging waste management, including a test scheme and a procedure to assess the compostability, is regulated by the European Norm BSI. 2000, which defines the criteria for material to be recognized as “compostable” (Sikorska *et al.*, 2015; BSI., 2000). Biodegradation of plastics depends on both the environment in which they are placed and the chemical nature of the polymer. Biodegradation is an enzymatic reaction; hence it is very specific to the chemical structures and bonds of the polymer (Vaverková *et al.*, 2014).

There are different mechanisms of polymer biodegradation. The biodegradation of polymers consists of three important steps: (1) Biodeterioration, which is the modification of mechanical, chemical, and physical properties of the polymer due to the growth of microorganisms on or inside the surface of the polymers. (2) Biofragmentation, which is the conversion of polymers to oligomers and monomers by the action of microorganisms and (3) Assimilation where microorganisms are supplied by necessary carbon, energy and nutrient sources from the fragmentation of polymers and convert carbon of plastic to $CO_2$, water and biomass (Emadian *et al.*, 2017).

Recently, various materials have begun to be marketed that claim to be biodegradable or compostable. Terms such as "degradable", "oxo-biodegradable", "biological", "compostable" and "green" are often used to describe and promote different plastics. These materials include conventional plastics amended with additives meant to enhance biodegradability as well as bio-based plastics and natural fiber composites (Gómez and Miche, 2013).

Our previous study was conducted to investigate whether the plastics marked "biodegradable", 100 % – degradable or certified as compostable are really biodegradable in real composting conditions. Original research was carried out in 2011 and 2012. The major goal of this study was to verify information obtained by repeated research and repeatedly examine the texture of the original samples and samples that underwent composting process. The rate of biodegradation has been analyzed by investigating the morphological properties using scanning electron microscopy (SEM).

## MATERIALS AND METHODS

Commercial bioplastics and a petrochemical plastic (claim to be degradable) were used for this study. The research of biodegradability was carried out in real conditions in the Central Composting Plant in Brno, Czech Republic. The company operates a regionally important (South Moravia) facility processing biological wastes. The composting plant is used for the conversion of biologically degradable waste (bio-waste) from the city of Brno and its surroundings (Vaverková *et al.*, 2014). The compost (three-month-old mature compost, which was provided by a full-scale aerobic composting) was the following physicochemical properties: moisture 30 ÷ 65 (%), combustibles min. 20 (%), total nitrogen min. 0.6 (% DM), pH 6.0 ÷ 8.5, undecomposable ingredients max. 2.0 (%), C : N max. 30, cadmium 2.0 (mg/kg), lead 100 (mg/kg), mercury 1.0 (mg/kg), arsenic 20 (mg/kg), chromium 100 (mg/kg), molybdenum 20 (mg/kg), nickel 50 (mg/kg), copper 150 (mg/kg), zinc 600 (mg/kg).

The investigated materials were single-use plastic bags available in various networks of shops on the European market, advertised as 100 % – degradable (Sample 1–3) or certified as compostable (Sample 4–7). The thickness of each sample was 0.2 mm. The material composition of the samples is presented in Tab. I.

The samples were made of high density polyethylene (HDPE) with the Totally Degradable Plastic Additives (TDPA) (Sample 2) and made of polyethylene (PE) with an addition of pro-oxidants (d2w) (Sample 1 and 3). The eighth, control sample was cellulose paper (CP) (with dimensions 0.3 mm thickness) (Sample 8). CP was to check the potential of biological decomposition in the tested environment (positive control). Samples (1–8) were placed into frames. The frames were designed and made by the authors themselves of wooden slats as follows: width = 280 mm, length = 340 mm and height = 50 mm. A 1 × 1 mm polyethylene mesh was fixed onto the frames. The frames were designed so that they would facilitate the placement and identification of the samples in the compost pile. The frames with the samples were properly marked and photographed to document future visual comparison (Vaverková *et al.*, 2014). All 8 samples were inserted into one clamp within the compost pile (Fig. 1). The samples were installed at a height of 1m from the upper side of the compost pile and at 1.5 m from the lower side of the pile. In these conditions, the experimental period was estimated to be 12 weeks. The samples were checked visually at regular intervals of about 14 days. After the end of the experiment, the samples were lifted from

I: *Material composition of the samples.*

| Sample | Type | Description |
|---|---|---|
| **1** | N/A | BIO-D Plast |
| **2** | HDPE + TDPA | 100 % degradabel |
| **3** | N/A | 100 % degradabel |
| **4** | Starch | Compostable 7P0147 |
| **5** | Starch and Plycaprolactone | OK Kompost AIB VINCOTTE |
| **6** | N/A | Compostable 7P0202 |
| **7** | Natural material | Compostable 7P0073 |
| **8** | Cellulose (blank) | – |

the compost pile and all samples were subsequently photographed and assessed (Vaverková *et al.*, 2014). In addition, all original samples as well as samples obtained after the end of the experiment were submitted for SEM.

## RESULTS

### Visual assessment of the samples

Upon the end of the experiment in composting plant the samples were taken to laboratories of the Department of Applied and Landscape Ecology at Mendel University in Brno where they were subjected to detailed evaluation. In all samples, a visual comparison was made of their initial and final states. Samples 1, 2 and 3 did not show any significant visual changes or signs of decomposition, the test material remained completely intact. No breakthrough in disintegration was observed after 12 weeks of composting. The surface was smooth, and there were no pinholes observed on the surface after the test. However, Sample 3 exhibited some changes in pigmentation.

The biodegradation of the certified compostable plastic bags (Samples 4–7) proceeded very well. In terms of visual assessment, Samples 4 and 5 exhibited the highest degree and rate of decomposition (70%). Samples 6 and 7 were decomposed to about 80% of their initial condition. The CP (Sample 8) completely degrade implying that the conditions required for biodegradation to occur in sampling environment were present (Vaverková *et al.*, 2014).

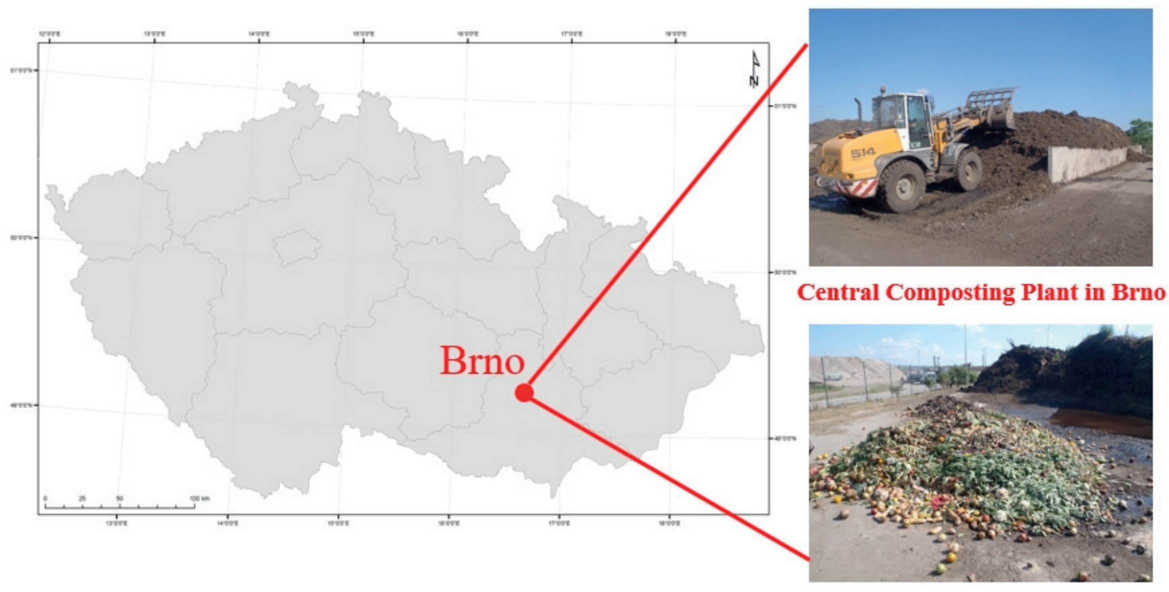
### The evaluation of the samples by means of Electron Microscopy

SEM analysis technique allows examining of changes in the morphology of materials at the micro scale. In order to perceive monitoring and the changes in the structure of the samples, images from the SEM were used. All samples were subsequently submitted for SEM. Surface morphology by scanning electron microscopy was determined on a FEG Quanta 200 (ESEM, USA) in Analytical Centre of Warsaw University of Life Sciences – SGGW.

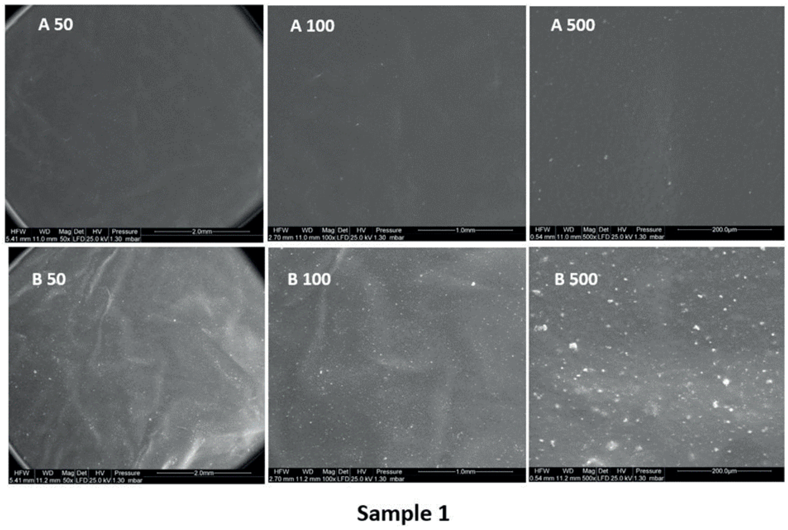
The experiment involved images of both the original samples and samples that underwent composting process. Each sample was depicted at 50 ×, 100 × and 500 × magnification. The illustrations are presented in Figs. 2–8. Via SEM images analysis, is was possible to verify the considerable quality of the samples.

The figures from SEM (Figs. 2–4) illustrate that the structure of surface of the original samples (defined as A) compared to the composted samples (defined as B) show the smallest difference in case of Sample 1–3. For the samples amended with additives that were supposed to enhance biodegradability, almost no biodegradation was observed after 12 weeks of composting. SEM images did not reveal qualitative changes in the appearance of Sample 1 and 3. Only slight changes can be observed at Sample 2 (Fig. 3 B). Sample 2 shows certain erosion of surface.

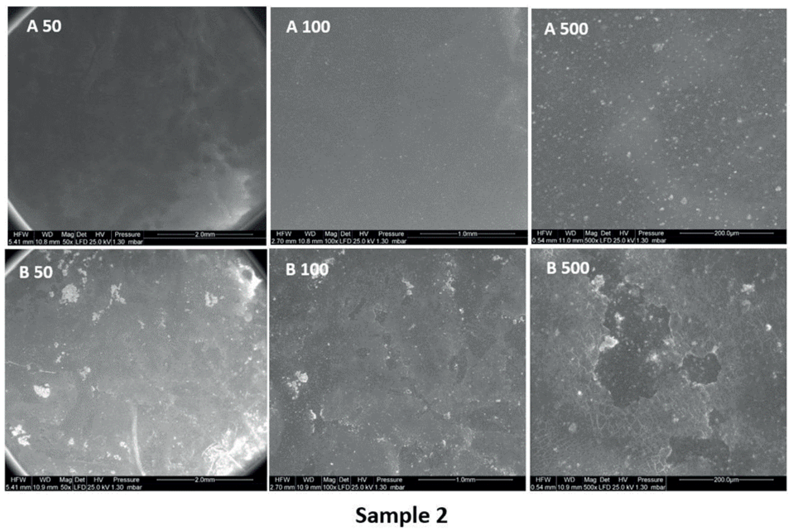
SEM analysis exhibited the microbial activity of degradation on the bioplastic Samples 4–7 (Fig. 5–8). The surface structure of the material had lost its smoothness, and cracks were evident. The samples showed a significant change in the structure. SEM images confirmed the biodegradation process



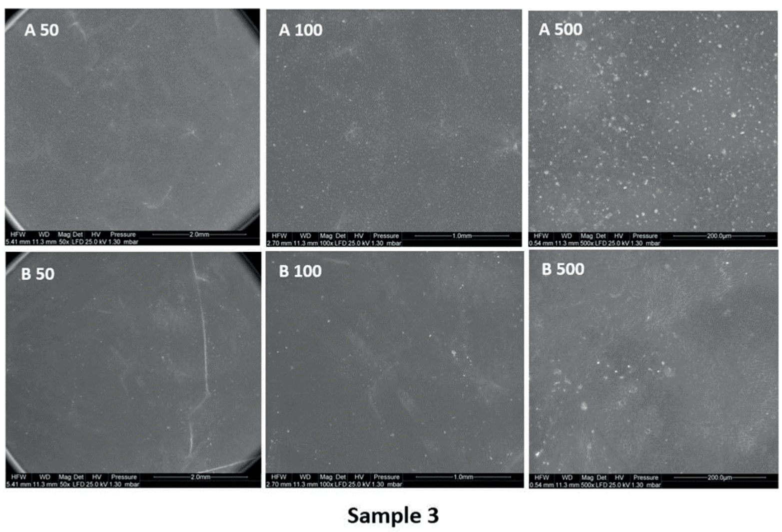

1: *Location of the Central Composting Plant in Brno*



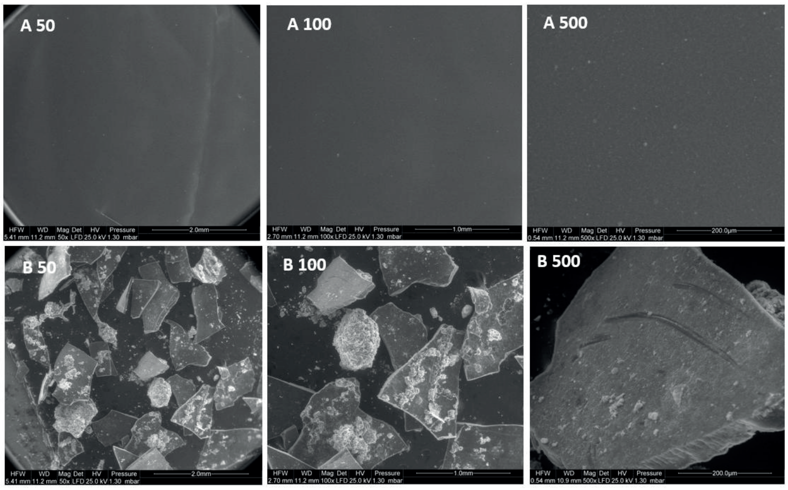

2: *Sample 1 - Surface of the original sample (A) compared to the composted sample (B)*




3: *Sample 2 - Surface of the original sample (A) compared to the composted sample (B)*

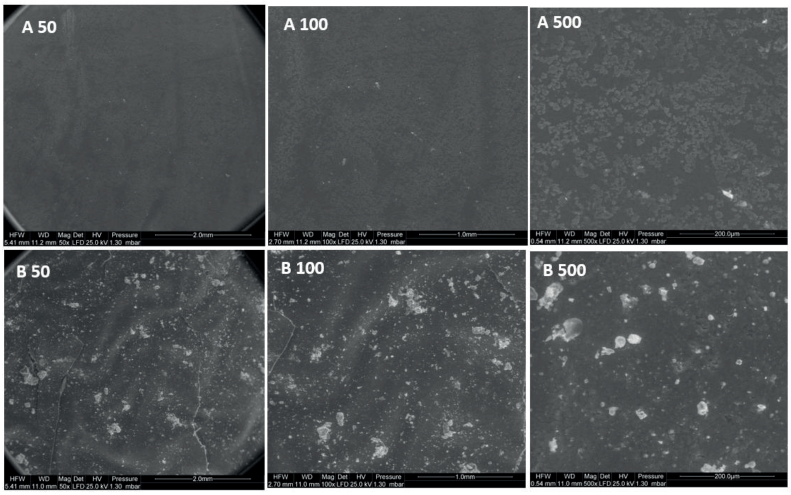



4: *Sample 3 - Surface of the original sample (A) compared to the composted sample (B)*



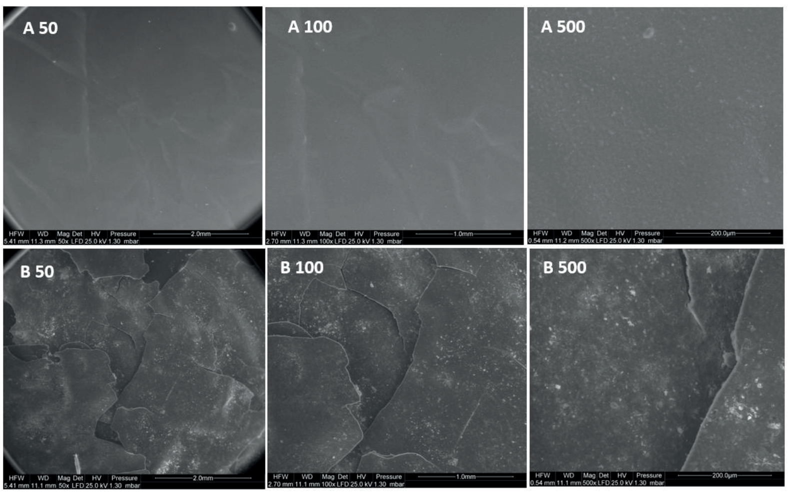

**Sample 4**

5: *Sample 4 - Surface of the original sample (A) compared to the composted sample*



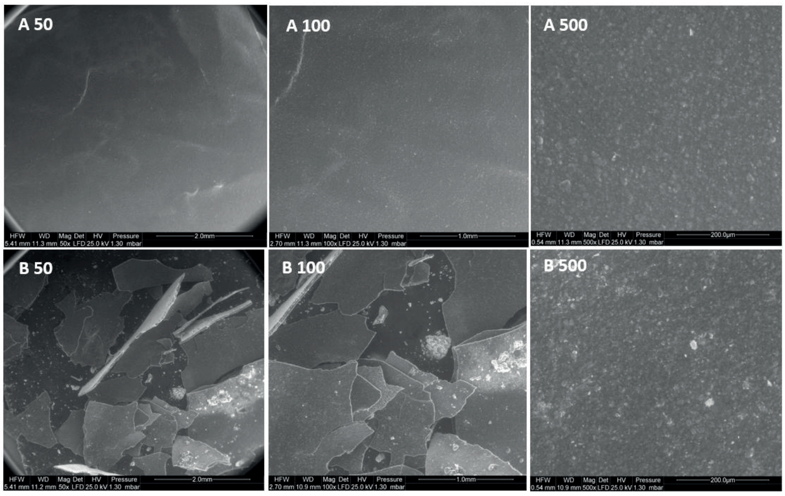

**Sample 5**

6: *Sample 5 - Surface of the original sample (A) compared to the composted sample (B)*




**Sample 6**

7: *Sample 6 - Surface of the original sample (A) compared to the composted sample (B)*




**Sample 7**

8: *Sample 7 - Surface of the original sample (A) compared to the composted sample (B)*

that happened over the bioplastic film with the presence of cracks and loss of filmy nature. After the termination of the testing process in real conditions, the bioplastic sample showed visual modifications, and broke into pieces when touched upon. Sample 6 and 7 significantly degraded as apparent by visual detection. This fact was additionally confirmed by the figures from the SEM analysis (Sample 6 B and Sample 7 B see Fig. 7 and 8). The reduction on mechanical properties is one of the main consequences of the degradation process occurring during composting, this process also favored the microorganism's action. This effect could be explained since the loss in mechanical properties after 12 composting weeks produced a brittle material consisting in broken pieces with a high defects density, such as cracks and porous structure, permitting the easy access of microorganisms to the polymer bulk. The optical inspection of Samples 4–7 (Fig. 5–8) revealed these defects. SEM images of bioplastics before and after composting showed substantial changes in the surface of the material.

## DISCUSSION

Composting seems to be the most promising for waste management options for biodegradable plastics because the composting process is designed to degrade wastes. There are, however, obstacles that make many communities reluctant to accept plastic bags for composting. Various studies have shown that new biodegradable polymers do biodegrade under controlled composting conditions (Gómez and Miche, 2013; Unmar and Mohee, 2008; Leejarkpai *et al.*, 2011; Vaverková *et al.*, 2012; Bahramian *et al.*, 2016; Castellani *et al.*, 2016). However, the biodegradability of plastics is a complex process and is influenced by the nature of each plastic (Selke *et al.*, 2015).

The results of Gómez and Miche (2013) study indicate that conventional plastics containing additives do not biodegrade any faster than non-additive containing plastics. Manufacturers of these additives claim that if at least 1–5 % (by weight) of their additive is added to plastics products, these will fully biodegrade when disposed of in microbe-rich environments. These claims are not supported by the findings of study by Gómez and Miche (2013). Moreover, for conventional plastics with additive no significant conversion was observed over the entire period of study.

An experimental investigation was conducted by Leejarkpai *et al.* (2011). It was observed that the swelling at the starch granules occurs throughout the surface of the PE/starch due to water absorption by the granules. However, almost all surface of the PE/starch remained relatively unchanged suggesting that only a small degree of swelling had occurred (Leejarkpai *et al.*, 2011). Additionally, in this study SEM examination, confirmed the biodegradation of biodegradable plastics material. The results also prove that both PE and PE/starch are non-biodegradable plastics whereas Polylactic acid is a biodegradable plastic.

In a different study, Selke *et al.* (2015) examined the effect of biodegradation-promoting additives on the biodegradation of PE and polyethylene terephthalate (PET). Biodegradation was evaluated in compost, anaerobic digestion, and soil burial environments. None of the additives tested significantly increased biodegradation in any of these environments. Thus, no evidence was found that these additives promote and/or enhance biodegradation of PE or PET polymers. The finding provides evidence that anaerobic and aerobic biodegradation are not recommended as feasible disposal routes for plastics containing any of the biodegradation-promoting additives (Selke *et al.*, 2015).

## CONCLUSION

The experimental samples were placed in the compost pile operated by the Central Composting Plant in Brno, and were checked and visually assessed during the experiment which lasted 12 weeks. The goal of the experiment was to test the biodegradation of the above-described samples in real composting conditions. After the expiration of the experimental period it was found out that the samples with the additive (Samples 1–3) had not been decomposed, their color had not changed and that no degradation neither physical changes had occurred. SEM analysis of the samples was done in order to analyze microstructure and morphology of specimens, validating dispersion results. SEM images showed the biodegradation indicators such as fractures, breaches, cavities, and holes on the surface (Samples 4–7). It can be concluded that samples certified as compostable have degrade in real composting conditions. Selected Samples (4–7) showed significant erosion on surface when subjected to the SEM analysis. Samples labeled (by their producers) as 100% degradable (Samples 1–3) did not show any visual signs of degradation.

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