



Lead dodecanoate coatings for the protection of lead and lead–tin alloy artifacts: Two examples



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ABSTRACT

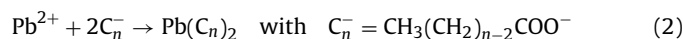
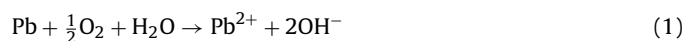
In order to understand the corrosive and morphological characteristics of lead dodecanoate protective coatings on real samples, three pipe organ samples were studied using optical microscopy (OM), scanning electron microscopy (SEM) coupled with energy dispersive spectroscopy (EDS), X-ray diffraction (XRD), and electrochemical impedance spectroscopy (EIS). The corrosion products and elemental composition of the top layer of the different pipe organ samples were investigated. The results indicate that the three pipe organ samples are made of an alloy composed mainly of lead and tin. After immersion and the deposition of the protective coating, only lead and no tin is detected, which indicates the formation of a thick coating containing lead dodecanoate complexes.

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1. Introduction

From the 19th century on, 15th and 17th century organs in churches all over Europe began to lose their specific sound due to corrosion caused by the increase of humidity, temperature, and the presence of organic acids in the nearby atmosphere [1]. Research performed during the COLLAPSE (corrosion of lead and lead–tin alloys of organ pipes in Europe), TrueSound (A challenge for materials science: bringing true baroque and medieval sound back to life in historic and new pipe organs) and SENSORGAN (sensor system for detection of harmful environments for pipe organs) projects, as a part of the European Community (EC) 5th and 6th Framework Programme, demonstrated that formic acid and acetic acid, which are released from the wooden wind chest, are primarily responsible for irreversible damage to lead-based European organ pipes [2,3]. In fact, ancient lead and lead alloy-based artifacts exhibited or stored in wooden display cases used in museums worldwide suffer the same type of severe corrosion [4,5]. The concentrations of these organic acids, especially of acetic acid, in wooden display cases and in wind chests can range from 100 to 6000 $\mu\text{g L}^{-1}$ depending on the type of wood [6].

Efforts to protect lead-based artifacts against corrosion can take two approaches: (1) reduce the corrosive substances in the nearby environment and/or (2) apply a surface protection treatment by developing a coating using corrosion inhibitors. These inhibitors should be stable, reversible, inexpensive, and aesthetically suitable. In this article, we focus on the application of protective coatings. This approach uses surfactant inhibitors, which are composed of a polar hydrophilic group, the head, and a non-polar hydrophobic group, the tail. Surfactant inhibitors have many advantages compared to other organic compounds, including high inhibition efficiency, low price, low toxicity, and easy production [7–9]. An interesting candidate in this family is the group of non-toxic saturated linear monocarboxylates of the type $\text{CH}_3(\text{CH}_2)_{n-2}\text{COONa}$, hereafter called NaC_n , with $n = 7–11$. An initial study by Rocca et al. [9,10] showed that the immersion of lead metal objects in NaC_n solutions caused the growth of a protective coating consisting of a crystalline lead monocarboxylate complex, $(\text{CH}_3(\text{CH}_2)_{n-2}\text{COO})_2\text{Pb}$, hereafter called $\text{Pb}(\text{C}_n)_2$. The production of the coating relies on an unassisted formation of lead ions (1) followed by a passivation mechanism (2) according to the following reactions in a neutral solution:



In previous work, we have used two different methods to deposit lead dodecanoate ($\text{Pb}(\text{C}_{12})_2$) coatings on a polished lead metal

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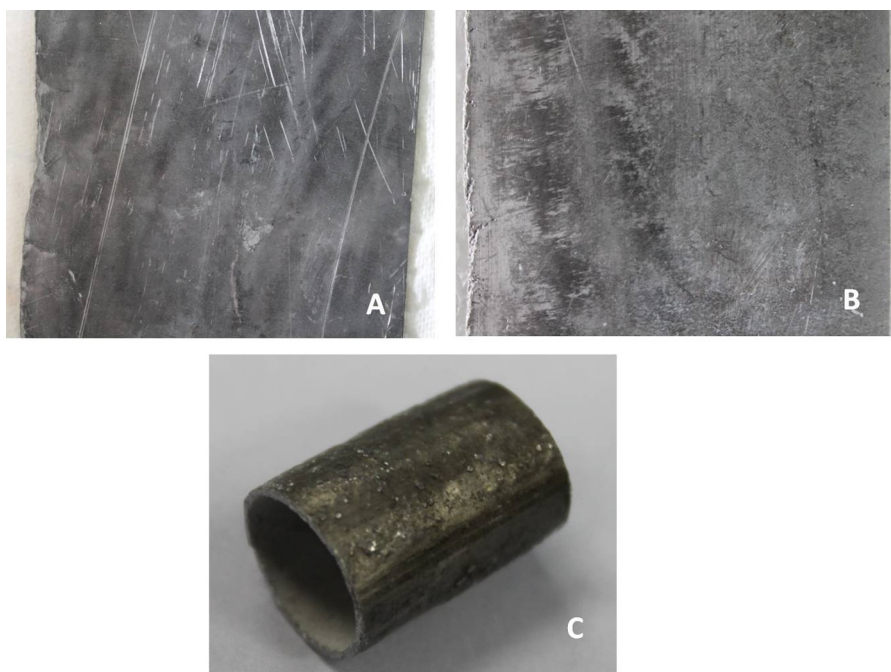


Fig. 1. Photograph of the investigated organ samples: (a) sample 1, (b) sample 2 and (c) sample 3.

sample [11,12]. We tested the immersion method (IMM) using a reduction pre-treatment of the lead surface (-1.5 V for 600 s) and the cyclic voltammetry (CV) method. Both deposition methods seem to produce a coating protecting the lead against anodic corrosion, which was proven using polarization and impedance measurements. The advantage of using electrochemical techniques is the improvement of the reproducibility of the coatings. The aim of this study is to protect three different lead–tin alloy organ samples using the formation of lead dodecanoate coatings. In this report, two deposition methods are tested and compared: the uncontrolled IMM method with an initial reduction step also at -1.5 V , and the controlled CV method. The corrosion protective properties of the treated samples are determined using non-destructive EIS measurements in a 5 mg L^{-1} acetic acid solution.

2. Description of the organ samples

All organ samples studied in this paper are shown in Fig. 1. The first (sample 1) and second (sample 2) samples are two sheets (with dimensions $20\text{ cm} \times 10\text{ cm}$) of organ metal, which consist of a mixture of lead and tin. The two metal sheet samples were pre-treated by hammering and painting. To produce the organ pipe, the pipe maker simply needs to cut the sheet to the right size, roll it around a cylinder to shape it, and solder the seam closed. The third sample (sample 3) is a piece of a real organ pipe (measuring $2 \times 1.5\text{ cm} \times 1.5\text{ cm}$) originating from the principal part of the organ in St. Mary the Virgin Parish Church (Edith Weston, Rutland, UK), which was built by Samuel Green of London in 1787 (English Baroque) for an unknown residence in the capital (Fig. 2). The organ was sold in 1842 to the Rev. C.H. Lucas, who was vicar of the church, and transported by rail to Edith Weston where it was placed in Edith Weston Hall. The brass plate on the organ bears the inscription: “This organ is presented to the inhabitants of Edith Weston by Richard Lucas, 1867”. It is presumed that Porrit of Leicester installed the present swell front in 1873. An electric blower was installed in 1971, and the organ was restored in 1981 by Goetze and Gwynne [13]. Using X-ray fluorescence, the research group of Storme (Artesis, Antwerp, Belgium), observed that the organ’s corpus contains

a large amount of tin, and that the organ’s foot consists mainly of lead [14].

3. Experimental methods

3.1. Materials

A 0.05 mol L^{-1} inhibitor solution was prepared by dispersing 1.252 g of dodecanoic acid (Sigma Aldrich, 98%) in 250 mL water and by subsequently neutralizing the obtained suspension with a standardized 0.25 M NaOH solution. When adding NaOH to the dodecanoic acid, it is important to stop before the calculated equivalence point (at 98% moles of NaOH compared to those of dodecanoic acid). This method avoids the formation of hydroxide ions, which can easily form lead hydroxides and lead oxides on the lead surface when oxidation begins. The slow addition of NaOH helps the gradual dissolution of the dodecanoic acid (HC_{12}), which has minimal solubility in water. The initially turbid solution was stirred overnight to neutralize all the acid and to obtain a clear and transparent solution. To accelerate the dissolution process, the sodium dodecanoate solution was slightly heated to $25\text{--}30\text{ }^\circ\text{C}$ and stirred for 1 h. The pH of the solution should be around 9. The corrosion resistance of the sodium dodecanoate coating was tested in a corrosive solution containing an acetic acid concentration of 5 mg L^{-1} .

3.2. Deposition methods

Electrochemical deposition experiments were performed using a three-electrode system in a glass cell. The setup consists of a saturated calomel reference electrode (SCE) containing two compartments (Radiometer Analytical, France), a carbon counter electrode, and the organ sample as a working electrode. The working electrodes (the organ samples) were not pre-treated, and were used as received. Two deposition procedures were used to apply a lead dodecanoate coating on the sample surface. Both were carried out in a neutralized 0.05 mol L^{-1} sodium dodecanoate solution. The CV method, as described by our research group

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