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Development of low-cost RFID sensors dedicated to air pollution monitoring for preventive conservation

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Abstract

Monitoring of atmospheric pollutants is a key point for preventive conservation since these species are known to impact the integrity of many artifacts including metals, papers, pigment and textiles. The problem can be amplified in closed environments, like in exhibition rooms and showcases, where temperature and relative humidity gradients and levels can introduce additional micro-climatic problems. One objective of the EU-SensMat project concerns the development of low cost and low visual nuisance sensors sensitive to these pollutants, making them affordable for all museums including small ones. For this purpose, the Radio-Frequency Identification (RFID) technology was considered to produce air-quality sensors based on reactive metallic dosimeter. Besides the low cost, they can be easily integrated to other existing RFID applications such as identification tasks since the interrogation is made with a commercial UHF-RFID reader. The description and the main features of these sensors are discussed in this paper. Moreover, as it will be shown, the subsequent chemical analysis of the sensitive part of the sensors provide useful information to determine the origin of the pollutants.

Keywords: Environmental corrosivity, Air quality, Autonomous sensors, RFID, XPS, H₂S

Introduction

Air pollution monitoring is of crucial interest for conservation and protection of historical artifacts in indoor environments. Indeed, gaseous pollutants in air are responsible of the degradation of many types of objects and materials. They are of diverse nature (organic acids, H₂S, NH₃, SO₂, NO₂ ...) and are produced either internally in the building where are localized artifacts or externally due to outdoor pollution [1]. Some devices dedicated to the monitoring of air quality exist and may be applied for cultural heritage conservation. These include the use of sampling techniques or gas sensors [2–6]. An alternative to these methods is to consider the

development of dosimeters based on the chemical reactivity of metallic surfaces with the mentioned pollutants. This cumulative monitoring method based on coupons has been applied in numerous cases and provides information on the thickness and nature of corrosion products after exposure. Sacchi and Muller [7] proposed a classification scheme dedicated to cultural heritage for silver and copper coupons. The ISO 11844-1 standard [8] gives the Indoor Corrosivity (IC) classes in indoor environment which range from IC1 (very low corrosivity) to IC5 (very high corrosivity). Steel, lead, zinc, copper and silver are sensitive to different pollutants and are therefore selected in this standard. The main drawback of this method is the lack of real-time information. For this purpose, several sensors have been developed in the past. The OnGuard™ sensor uses a quartz crystal microbalance to determine the mass gain of copper and silver during corrosion [9]. The AirCorr™ sensor was developed

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in a project supported by the European Commission (Protection of cultural heritage by real-time corrosion monitoring) and dedicated to the monitoring of corrosive atmospheres in the cultural heritage sector [10–12]. In this case, the sensitive part of the sensor is constituted by a thin copper or silver metal track. From the change in geometry of this metallic element (i.e., thinning) induced by corrosion, it is possible to calculate the loss of metal thickness and hence the IC corrosivity class. However, the method is costly and does not authorize the installation of tens or more sensors in a well-defined environment to investigate micro-climatic variations. In the European SensMat project (SensMat: Preventive solutions for Sensitive Materials of Cultural Heritage), the aim was to significantly reduce the cost of such sensors. Two solutions were considered: (i) reducing the cost of the electronic components of the ER (Electrical Resistance) sensors while maintaining a high sensitivity and (ii) developing new Radio-Frequency Identification (RFID) ultra-low-cost sensors. The present paper aims at presenting this second type of sensors.

During the last decades, the RFID technology has become more and more popular for a variety of applications including the cultural heritage sector [13, 14]. It is indeed an efficient way to transfer data and track items. This attractiveness is explained by the ability of the RFID readers to interrogate numerous flexible tags at a very low price and with a low visual nuisance. A recent progress concerns the integration of sensing devices within this technology. Some RFID sensors were proposed for corrosion monitoring [15–27]. For this purpose, several RFID architectures were selected with their advantages and drawbacks. In particular, radiofrequency sensitive resonators were produced to be integrated in chipless RFID technology. In this case, due to the absence of any electronics in the sensor, the robustness of the sensor is ensured. However, this technology suffers from the absence of any commercial reader and thus a low TRL (Technology Readiness Level). Within the European project SensMat, an Ultra High Frequency (UHF) RFID corrosion sensor was developed to assess the air quality and IC classes in museums [26, 27]. The chosen architecture was then the UHF-RFID one since it employs commercially available readers which can also be used in museums for identification tasks. Moreover the reading distance, i.e. the distance between the sensor and the reader is significantly increasing with respect to Low-Frequency (LF) RFID method leading to reading distance of few meters. The present study aims at demonstrating the interest of such sensors. For this purpose, the air quality was monitored in buildings specialized in conservation-restoration of archeological and historical woods. Two case studies were selected: a storage room with a possible

internal emission of H₂S pollution by treated archeological woods, and the monitoring of a room dedicated to the restoration of polychromic woods in urban environment, both located in the Atelier de Recherche et de Conservation (ARC-Nucléart), institutions belonging to the Commissariat à l'Énergie Atomique et aux Énergies Alternatives (CEA) of Grenoble. The second part of this study focuses on the determination of the origin of the observed pollution by X-Ray Photoelectron Spectroscopy (XPS) applied to the sensitive metallic silver films integrated in the RFID sensors.

Methods

Principle and selections of RFID sensors

The RFID sensor is presented in the inset of Fig. 1a. Its dimensions are $11.3 \times 6.5 \text{ cm}^2$. The principle of the sensor was described in detail elsewhere [26, 27]. As shown, it is based on the electromagnetic coupling between a metallic sensitive thin film which acts as a coupon exposed to a corrosive environment and the antenna of an RFID tag. This latter is integrated inside the flexible sensor and cannot be seen in the figure. Corrosion of the metallic layer induces an increase of its electrical resistance and hence, a change of the property of the coupled antenna. The signal strength emitted from the tag to the reader is then modified when corrosion occurs. This variation can be monitored by measuring the RSSI (Received Signal Strength Indication) by a commercial UHF-RFID reader.

Before corrosion, with the presence of the metallic conductive thin film, the properties of the antenna of the tag are degraded. Therefore, it does not respond and no RSSI value is collected by the reader. When corrosion of the active layer occurs, as seen in Ref. [26], the antenna progressively returns to its “free state” i.e. without any surrounding conductive layer. A radio-frequency signal and the associated RSSI level are then received by the RFID reader. After corrosion of the entire metallic layer, the RSSI of the sensitive tag is identical to that of an RFID tag without any metallic sheet. As the consequence, comparing the RSSI values of a reference tag without any metallic layer and the sensitive one is indicative of the corrosion level of the metallic sheet. The sensor is thus constituted of these two RFID tags. The reference tag is depicted in Fig. 1a by a blue line. As the other one, it is integrated in the plastic sheet and cannot therefore be seen.

A calibration procedure was performed in Ref. [27] to correlate the variation of RSSI measured by the RFID reader and the decrease of thickness of silver thin films. It was shown that by keeping a RSSI value of -45 dBm for the reference tag, the sensitive one is responding with a RSSI value of -55 dBm when a thickness of 7.5 nm of silver remains. This value of -55 dBm moreover corresponds to the minimal value for which the tag can be

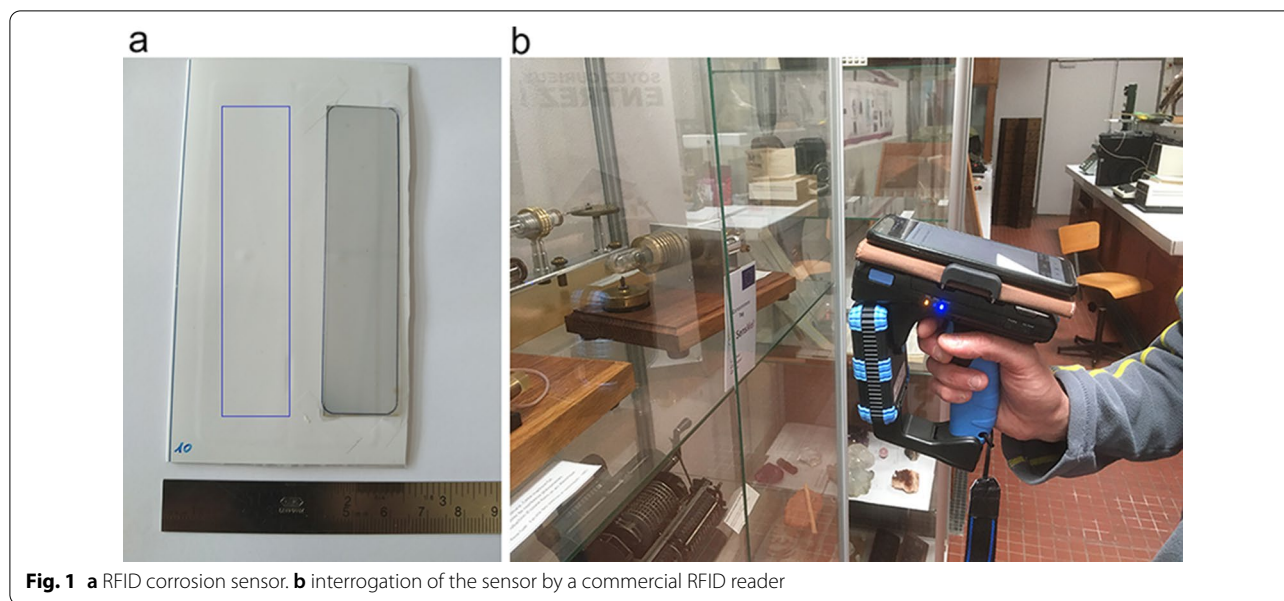


Fig. 1 a RFID corrosion sensor. b interrogation of the sensor by a commercial RFID reader

detected by the selected RFID reader. Accordingly, the interrogation of the sensor is made by selecting a value of -45 dBm for the reference tag and by detecting or not the sensitive tag. This indicates if the remaining thickness is higher or lower than 7.5 nm. When the sensitive tag is detected, an averaged corrosion rate over the exposure time can be thus be calculated.

Figure 1b displays the UHF-RFID reader used for the interrogation of the sensor; this latter being located in a showroom. The portable CS108 model from Convergence Systems Limited is chosen but other readers fit also very well with the application. As shown, a mobile phone is connected by Bluetooth to the reader. The RSSI value and identification number associated with one or two tags present in the sensor appears then on the mobile. To provide a user friendly interrogation tool, the development of a specific application dedicated to museums is currently in progress. It includes additional functionalities such as the locations of sensors in museums or storage rooms. Note that this example of interrogation does not correspond to the studied cases in the building under monitoring but aims at highlighting the possible application of the method in showrooms in museums. Additional tests of interrogations were also made successfully in showrooms containing more metallic parts.

In the supplementary data, a video shows the interrogation process in the case of Fig. 1b. When interrogating the sensor localized inside the showroom, the identification of the reference tag appears on the mobile phone. As seen, at few meters from the showroom, it is lower than -45 dBm. To perform correctly the interrogation, the user has therefore to move towards the sensors

at a distance of about 50 cm leading to a RSSI value of -45 dBm for the reference. A correct interrogation is therefore made.

To analyze the air quality in the two situations described below, silver sensors were produced due to the sensitivity of this metal to sulfur species independently of the relative humidity value. The initial thickness of the film was 20 nm, so that the detection of the sensitive tag corresponds to a loss of metal of 12.5 nm.

Locations and exposures

The RFID sensors were located in some areas of a building dedicated to conservation and restoration of woods at the end of 2020 and first semester of 2021. A temporary storage area for archaeological artefacts displayed in Fig. 2a was selected. Materials stored in this place were wood and vegetal fibers. As seen, a large plastic liner containing a treated archeological basket with sediments as well as a treated boat are located in this area. The interest of this area relies on the possible emission of H_2S by treated wood. Note that this storage room has no active climate control system. The second monitored place was the sculpture restoration workshop depicted in Fig. 2b. In this area, activities of conservation-restoration for historical polychromatic wood are performed. Medieval, Renaissance and Early modern polychrome religious sculptures are the main objects restored in the workshop. These polychromes are generally produced by metallic coatings (silver or gold) and colored pigments (metallic carbonates or acetates). Pollution by sulfur gas such as SO_2 which can be found in urban atmospheres is then a problem because

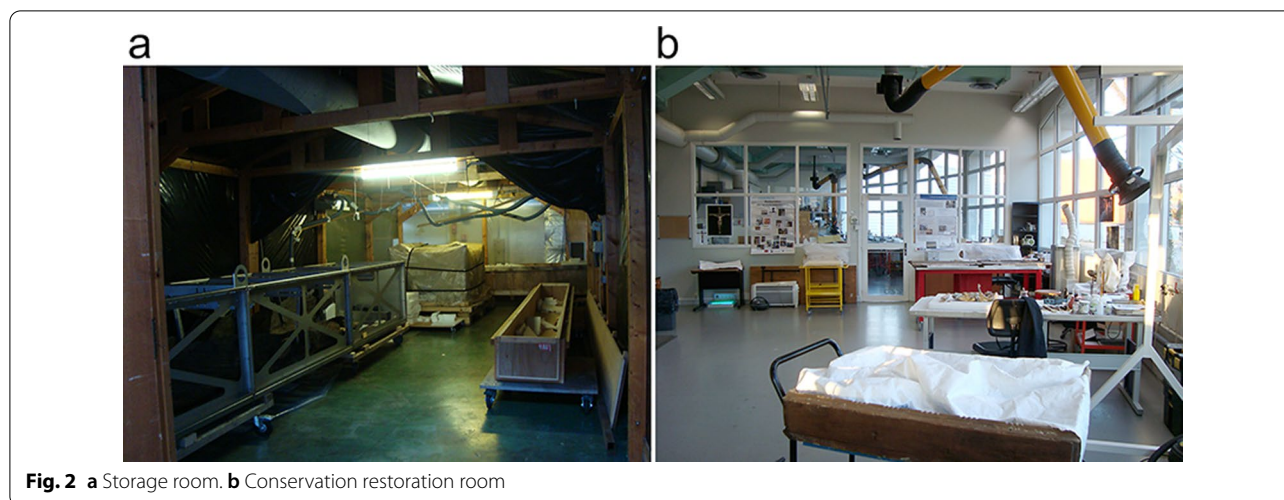


Fig. 2 a Storage room. b Conservation restoration room

it is known to react with pigments, causing darkening of the polychromy [28–31]. To limit such effects, the HVAC of the room keeps the temperature and relative humidity relatively constant and below 25 °C and 60% RH. There is no particulate or chemical filtration system. Checking the low corrosivity in this space by the proposed method is a key point of the present study.

RFID sensors located in these two selected areas were interrogated during two periods: from December 2020 to February 2021 and from March to July 2021. Location of sensors are detailed in Additional file 1: Figure S1. They are placed vertically to avoid dust deposition. The interrogation by the RFID reader was made each week due to the very slow corrosion process. During the first test, one silver sensor was placed in the storage room and another one in conservation room (sensors 1 and 2). For the second test, two silver sensors were located in the storage room to investigate the reproducibility of the method (sensors 3 and 4). Moreover, to test the sensitivity of both copper and silver materials to sulfide based gases, a copper sensor was also tested in the storage area (sensor 5). Finally, one silver sensor (sensor 6) was also considered during the second test in

the conservation room to investigate an eventual variation of corrosivity during the two testing periods.

Analytical methods: XPS

To determine the origin of the pollution, sensitive layers can after their exposure being analyzed by analytical methods. Among them, the XPS method is suitable to investigate the chemical products on the surface. The apparatus consists of an Al-K α X-ray source (Thermo VG) and a cylindrical mirror analyzer from RIBER. The C (1 s) line at a binding energy of 284.8 eV was used to normalize the absolute binding energies.

Results and discussion

Table 1 reports the exposure time needed to recover the signal from the sensitive tag as detailed in the “methods” section.

In the storage room, for the first test, the exposure time needed to corrode 12.5 nm of silver was 57 days. During the second test, as seen in the table, this time was increased to 80 days. Both sensors responded at the same time indicating a very good reproducibility of results. In contrast to silver, the copper sensor did not react at this location. This result is explained by the low

Table 1 Results achieved by RFID sensors in the two areas under monitoring

		Metal	Detection time	Corrosion rate	IC
Test 1	Storage room: sensor 1	silver	57 days	0.22 nm/day	3
	Conservation room: sensor 2	silver	Not detected		1
Test 2	Storage room: sensor 3	silver	80 days	0.16 nm/day	3/2
	Storage room: sensor 4	silver	80 days	0.16 nm/day	3/2
	Storage room: sensor 5	copper	Not detected		
	Conservation room: sensor 6	silver	95 days	0.13 nm/day	2

relative humidity value (about 45% RH) in the storage room which significantly reduce the corrosion of copper. The principle of the sensors being based on an increase of the electrical resistance of the coupons, this parameter was checked after 57 or 80 days of exposure time. It was indeed equal to about 100 Ω indicating the insulating nature of corrosion products and the reduction of metallic thickness. On the other hand, no variation of the electrical resistance of the copper thin film was observed in agreement with the non-detection of the copper sensitive RFID tag. In the present case study, the emission of H₂S by archeological woods is expected to explain the level of atmospheric corrosion. The decrease of the average corrosion rate between the two tests is therefore most probably explained by a reduction of number of objects present in the storage room.

For the restoration workshop, the situation was somewhat different. Indeed, during the first period, the silver sensor did not react and no variation of the electrical conductivity was observed leading to the conclusion of a very low corrosivity. For the second test, the sensitive silver tag was detected after an exposure time of 95 days. In this case, the only difference between the two tests was the human activities in the room and the variation of the outdoor pollution.

The sensors may be used to extract the corrosivity classes from the ISO 11844-1 standard. In this latter, IC1 (ultra-low)/IC2 (low)/IC3 (medium)/IC4 (high)/IC5 (very high) classes are associated with a loss of 16/62/285/630/1570 nm of silver during a period of 1 year of exposure. Due to the principle of the sensors, a strict ISO classification cannot be provided since the exposure time is not fixed to this particular period of 1 year.

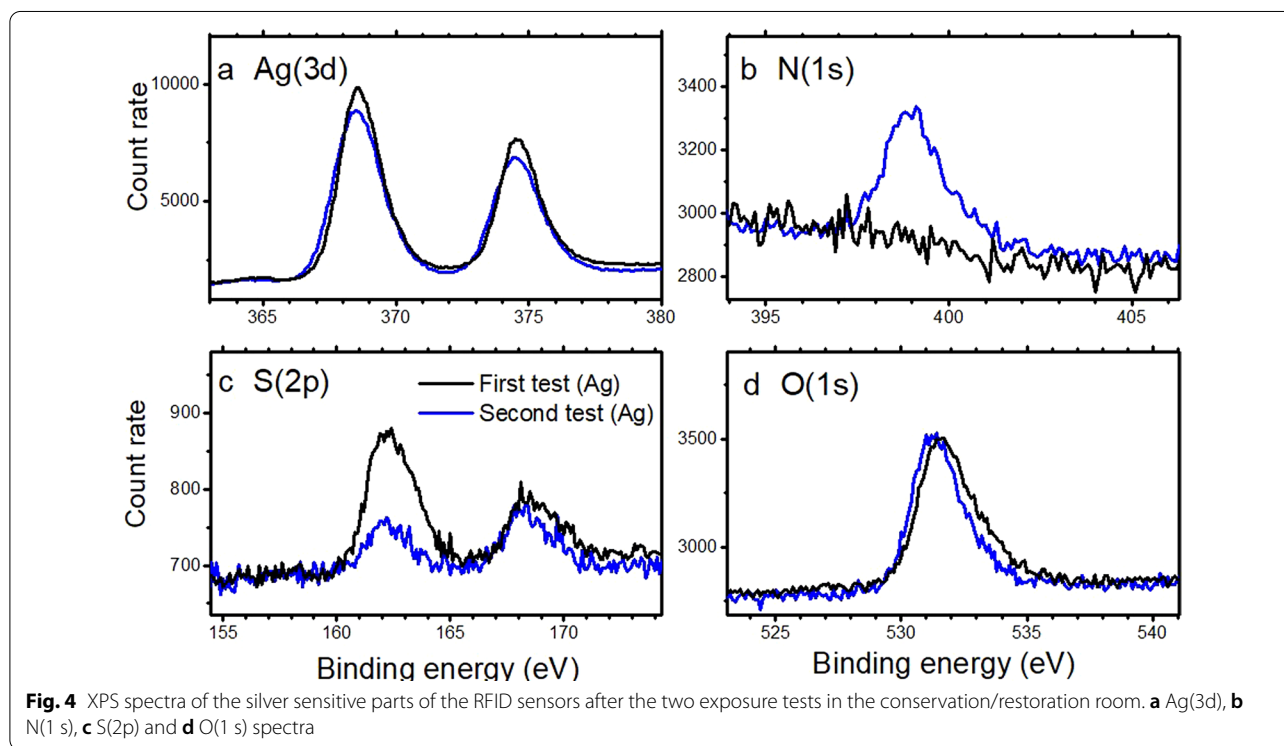
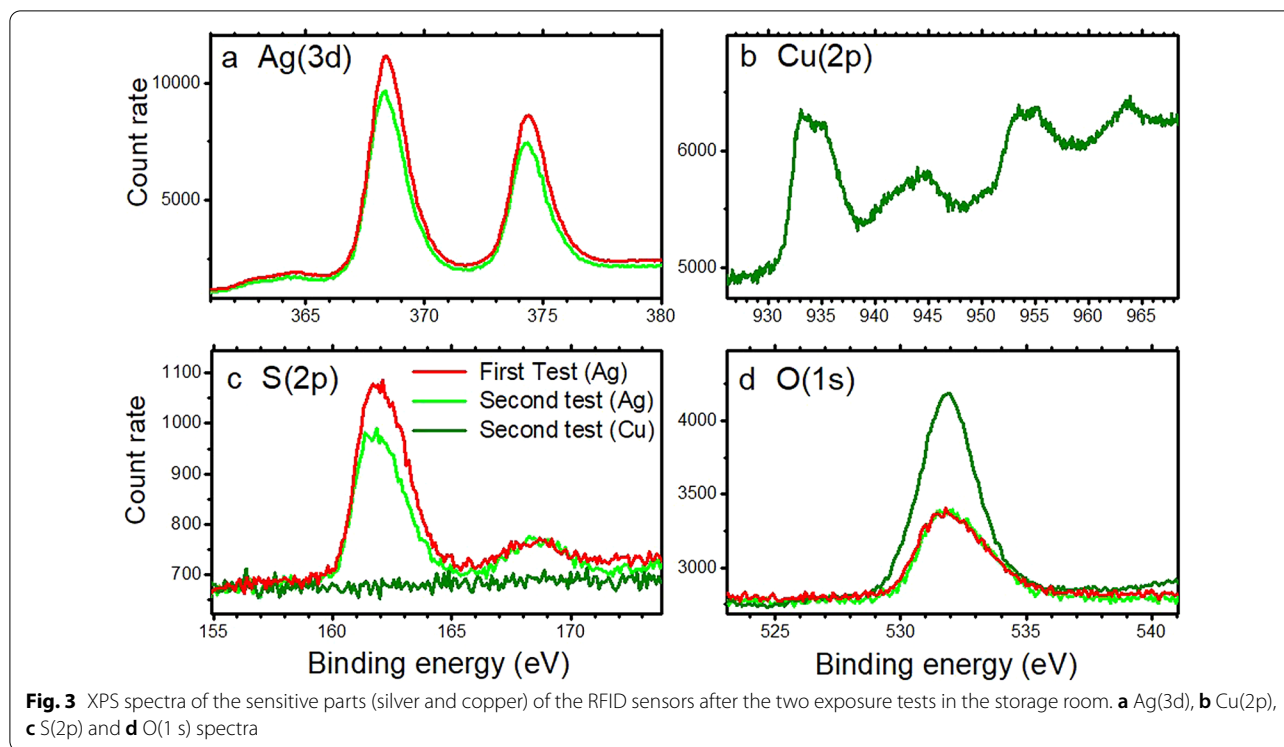
However, after some considerations, an evaluation of the IC class and corrosion risk can be provided from the present measurements. The first point concerns the mathematical law which governs the loss of metal as function of the exposure time. For silver, a linear variation is observed in the environmental conditions considered here [7, 10]. For copper, the situation is not so favorable since a parabolic law of the metallic loss is expected with the exposure time. As the consequence, an extrapolation of the results achieved with silver sensor during a period of less than 1 year may be valid for IC determination. However, this requires the assumption of a constant concentration of pollutants during 1 year which is debatable and strongly dependent of the selected area under monitoring. With these hypotheses taken into consideration, evaluation of IC classes by the present method is given in Table 1. As seen, in the storage room, for the first test, the exposure time needed to corrode 12.5 nm of silver was 57 days leading to the IC3 class (medium corrosivity).

During the second test, this time was increased to 80 days. The IC class is then in between IC2 (low corrosivity) and IC3 (medium corrosivity). Both results are thus very close and display clearly a non-negligible effect of corrosion in this area. In the conservation room, the corrosivity class is lower and evaluated to be IC1 (very low) and IC2 (low). The slight observed variation of the environmental corrosivity between the two tests is most probably ascribed to a non-constant load of pollutants during the 7 months of exposure. Therefore, the evaluated IC classes provided by RFID sensors cannot be considered as ISO values and are indicative. However, they clearly help to identify in which locations corrosion or degradation problems can occur. It is also noticeable that further developments of the method can be proposed by producing sensors of different thicknesses. For example, thicknesses of 16/62/285/630/1570 nm of silver could be selected to evaluate IC classes after 1 year of exposure. Other strategies may also be considered.

XPS analysis of the sensors

To further get some insights on the origin of pollution, Fig. 3 and Fig. 4 display the XPS spectra of the sensitive part of the sensors after exposure in the storage and conservation rooms, respectively. As seen in Fig. 3a,c,d, after the two tests in the storage room, the chemical composition of the two silver surfaces is very similar indicating the presence of the same type of pollutants in air during the two periods. Figure 3a and Fig. 3c present the Ag (3d) and S (2p) spectra of silver samples after these two tests. The peaks located at 368.3 eV in Fig. 3a and 161.6 eV in Fig. 3c are attributed to Ag₂S, in agreement with results obtained in the literature [32]. The presence of a slight contribution associated with silver sulfate species on the surface at 168.3 eV binding energy is also noted in Fig. 3c. On Fig. 3d, the O (1 s) spectra displays a peak located at 531.7 eV associated to hydroxycarbonate and sulfate species. Note that in a recent paper [33], the investigation of corrosion of silver under H₂S at 0.5 and 2.5 ppm was performed by XPS and others techniques. The spectra presented in Fig. 3 are nearly identical to those presented in this study. The emission of H₂S by the archeological objects [34] is therefore clearly the main source of contaminants in the storage room leading to the formation of only one uniform corrosion product: Ag₂S. The slight change of corrosion rate observed between the two tests is therefore explained by a decrease of emission of this pollutant. Indeed, some of the objects present in this room were removed in between the two tests.

Figure 3b,c,d present also the XPS spectra of the copper surface after exposure in the storage room during the second test. As seen in Fig. 3c, the S (2p) spectrum display no component associated with sulfur species. The Cu



(2p) spectrum depicted in Fig. 3b is complex and shows the appearance of components associated with metallic copper and Cu (OH)₂ compounds [35]. The presence

of metallic species at a depth of about 3 nm probed by the XPS method supports the very low corrosion rate of copper in the present environmental condition and

the interest of considering high sensitive silver coupons instead of copper in sulfur contaminated air.

XPS spectra of silver samples exposed in the conservation room are depicted in Fig. 4. Some differences are clearly observed with respect to the previous location. Figure 4c displays the S (2p) spectra. As seen, the two contributions associated with Ag₂S and sulfate based products are present, this latter being observed at about 169 eV. However, a clear increase of the relative intensity of this sulfate peak is observed with respect to the tests performed in the storage room. After the second test, sulfate species are even the dominant species on the surface. A second difference appears on the chemical products present on the surface. Indeed, as seen in Fig. 4b, some nitrogen products appear on the surface during the second test in the storage room. The binding energy of the nitrogen component is associated with NH₃ and/or NH₄⁺ species.

These results highlight the complexity of pollutants in this second area under monitoring with respect to the first area. Several sulfur based pollutants such as hydrogen sulfide (H₂S), sulfur dioxide (SO₂), carbon disulfide (CS₂) and carbonyl sulfide (OCS) [36–38] may participate to corrosion of silver. The presence of nitrogen is rarely reported under atmospheric corrosion. It may be ascribed here to the presence on the surface of ammonium sulfate particulate matter. Such particles are indeed frequently observed and are composed of (NH₄)₂SO₄ due the interaction in air of sulfuric acid and gaseous ammonia. The presence of such nitrogen species on the surface is correlated to an increase of the IC class. However, the role of such particles in corrosion of silver is not clear. Only Lin et al. [39] and Sanders et al. [40] proposed the formation of silver sulfate during corrosion of silver with the dissolution of such particle on the surface.

Conclusions

As a conclusion, the present work reports the application of RFID sensors for the monitoring of air quality in buildings dedicated to conservation and restoration of woods. As shown, the RFID sensor has to be considered as a binary sensor, which detects a loss of well-defined thickness of metal. This value can be tuned experimentally to monitor several environments with different IC classes. Here, with a nominal thickness of 20 nm, it was possible with two tests of about 3 months to obtain some results on (1) the influence of the number of archeological objects present in the storage room on the corrosivity and (2) on the non-negligible corrosivity during some periods of the year in the conservation room. Such results were obtained with silver sensor due to the presence of sulfuric pollutants. However, the method can be applied to other sensitive materials for

monitoring organic acids, NO_x, or ozone in museums. As shown, being based on coupons, the sensitive part can also be analyzed after exposure to investigate the origin of the corrosion. The proposed method can be extended to many issues in museums or storage areas, and should, therefore, be considered as a promising technique for preventive conservation in cultural heritage. Beside the very low cost of the sensor, it presents many advantages. Among them, it is very user-friendly and simple to use so that many actors in small and medium museums staff (conservators-restorers, archaeologists, museologists, curators...) can interrogate the sensors and get after few months of exposure a clear idea on the air-quality in the museum. Evaluation of emission of pollutants and the efficiency of HVAC or absorbents become then affordable for all museums.

Abbreviations

RFID: Radio frequency identification; UHF: Ultra-High frequency; LF: Low frequency; XPS: X-Ray photoelectron spectroscopy; HVAC: Heating, ventilation and air-conditioning; RH: Relative humidity; ID: Identification; IC: Indoor corrosivity; TRL: Technology readiness level; ER: Electrical resistance.

Supplementary Information

The online version contains supplementary material available at <https://doi.org/10.1186/s40494-022-00755-w>.

Additional file 1: Figure S1. Locations of sensors in the area under monitoring.

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Author contributions

GAR made the measurements by the RFID reader. SR made the XPS and wrote the main manuscript. All authors read and approved the final manuscript.

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Availability of data and materials

The data measured by XPS are available from the corresponding author on request.

Declarations

Competing interests

The authors declare that they have no competing interests.

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