# An Impedance-Based Life-Monitoring Technique for a Graphene Water Filter

**Stefano Bellucci, Antonino Cataldo, Luigi Ferrigno, Samuele Giovannetti, and Antonio Maffucci**

**Abstract** The paper deals with the design and verification of a monitoring system for the analysis of the useful life of a water filter. The filter is made of pressed graphene nanoplatelets, obtained from commercial graphite with a low-cost fabrication procedure. The state of the filter is monitored by measuring the electrical impedance at the port of a suitable circuit embedding the graphene filter. It has demonstrated a good sensitivity of the impedance with respect to the saturation level of the pollutants into the filter. The technique is also shown to provide a high level of reproducibility and stability with environmental conditions.

## 1 Introduction

Due to its outstanding properties, the graphene has been recently proposed for environmental applications, such as water remediation [1] and water desalinization [2]. Delocalized π-electrons, surface area, and functionalization are the physico-chemical peculiarities of graphene that are most involved in the process of metal ion and organic compound pollutant adsorption [3]. In fact, one of the available mechanisms of adsorption of cations is the interaction with the π-electron system to form a complex. Moreover, the same interaction is involved between the π-electrons and organic aromatic compounds or organic compounds having high electronic density (like double or triple bonds).

The use of graphene filters for water and other liquids has been successfully demonstrated in the last years [1, 4, 5], with excellent results: the graphene in [1] has been demonstrated to filter water and other liquids about 10 times faster than the current commercial filters. Given these results on prototypes, the present stage

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of research is aimed at solving some crucial challenges in order to move to industrial applications, such as the scaling-up of membranes to large area. Recently, a solution to such a problem has been proposed, based on an industrially adaptable method of blade coating [6].

In view of the industrial use of graphene as water filter, it is important to assess suitable procedures to measure its state, eventually addressing the paradigms of the predictive maintenance. In this paper, we propose a monitoring technique based on the measurement of the electrical impedance of the graphene filter. Section 2 presents the fabrication of a low-cost graphene filter, whereas Sect. 3 is devoted to the proposed experimental setup. The results of the analysis are discussed in Sect. 4: specifically, we monitored the adsorption of acetonitrile, a water-soluble organic compound having a triple bond, widely adopted in industrial processes.

## 2 The Graphene Filter

The water filters analyzed in this paper have been realized by means of low-cost graphene produced in the form of graphene nanoplatelets (GNPs), by using the simple and industrially scalable procedure developed at the INFN nanotechnology laboratory in Frascati [7–9]. The starting material is a commercial expandable graphite, i.e., a type of graphite intercalated with chemical substances, sulfates, and nitrates, inserted between the graphene planes of the graphite. The graphite is irradiated in a microwave oven for a few seconds and is expanded by heat shock, due to the sublimation of the intercalating molecules. The result is a worm-like graphene that is then placed in isopropyl alcohol and is subjected to sonication, which disperses nanoparticles in a solution consisting of a few layers. Next, a vacuum filtration is carried out, with a filter of adequate porosity, so that most of the isopropyl alcohol is aspirated and a compact and rather thick layer of GNPs is created on the filter. The obtained compound is placed in the oven for some hours, in order to guarantee the complete evaporation of alcohol. Finally, the compound is pressed to obtain the final filter, showed in Fig. 1, with a diameter of about 3.5 cm and thickness of 0.5 mm. A SEM image is also shown in Fig. 2 to put into evidence the surface detail of the filter.

## 3 The Monitoring System: Principles, Design, and Realization

The monitoring system proposed here is intended to check the status of the filter during its normal operation, namely, without interfering with the filtering activity. The basic idea is that of exploiting the high sensitivity of the electrical properties of the graphene to the presence of external elements adsorbed in its lattice. Indeed,

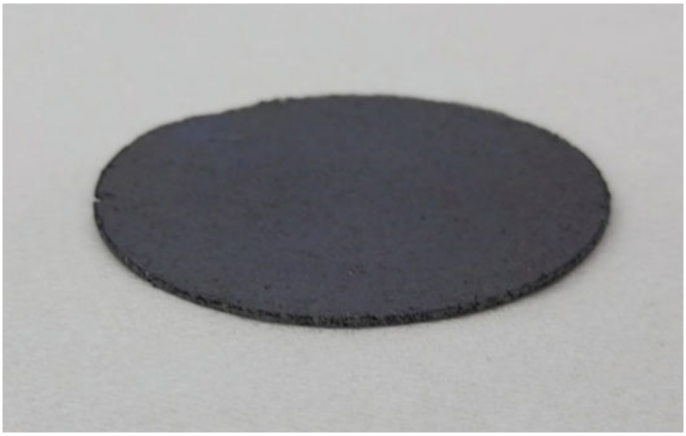

**Fig. 1** The final GNP filter, obtained after the steps of sonication, alcohol filtering, and pressing

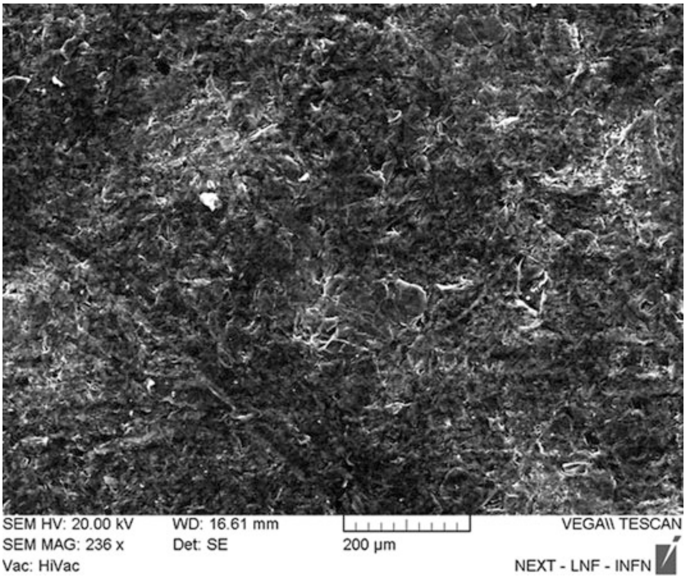

**Fig. 2** SEM detail of the filter surface

intentional doping is a widely diffused technique aimed at tailoring the electrical properties of graphene, such as the carrier mobility [10], the electrical conductivity [11], and the electrical permittivity and magnetic permeability [12]. Therefore, the adsorption of pollutant molecules into the graphene filter must change its electrical impedance, so that its variation can be assumed as an index of the quantity of pollutant adsorbed by the filter.

The design of the proposed system is reported in Fig. 3: the solution to be depurated flows in a hollow plastic cylinder, passing through the graphene filter. Two metal rings are put directly in contact with the filter, anchored at the ends of the two tubes. The overall system is geometrically fixed and sealed to prevent any loss of load: the realized setup is shown in Fig. 4.

The electrical impedance is measured between the two rings shown in Fig. 3. The two wires for the impedance measurement are connected to each metal ring (these wires are visible in Fig. 4). The impedance measurements were performed by using a commercial chip (SENSIPLUS® chip from Sensichips [13]) and a PC for data processing. This chip allows performing the analysis at different frequency values, in the range of 20–75 kHz. Before each measurement, a proper calibration procedure is executed, able to compensate the effects of contacts, wire impedances, and parasitic circuits. This procedure allows to improve the reproducibility of the hand-made realized setup.

The measurement system has been verified in terms of reproducibility of the results and of stability with environmental parameters. The reproducibility has been verified by measuring the value of the imaginary part of the impedance with five different filters, without pollutants, at the three operating frequencies of the adopted chip. The results reported in Table 1 clearly show that using the higher available frequency (75 kHz) leads to the best performance. Next, the stability of the measurement results against the variation of the temperature has been checked, by measuring the impedance values for one of the filters, in the temperature range of 24–45 °C, at five different frequency values. The results are reported in Fig. 5: once again, the best performance is obtained when choosing a frequency of 75 kHz, which provides the smaller variation of reactance with respect to the temperature variation, compared to other frequencies. Therefore, in the following, such a frequency value is chosen to perform the analysis of the filter status.

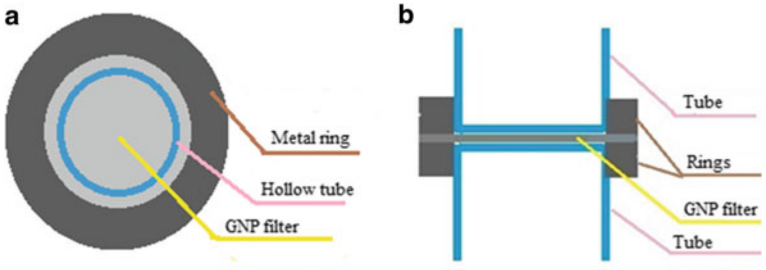

**Fig. 3** The proposed setup for the impedance measurement of the graphene filter: (**a**) section view, (**b**) side view

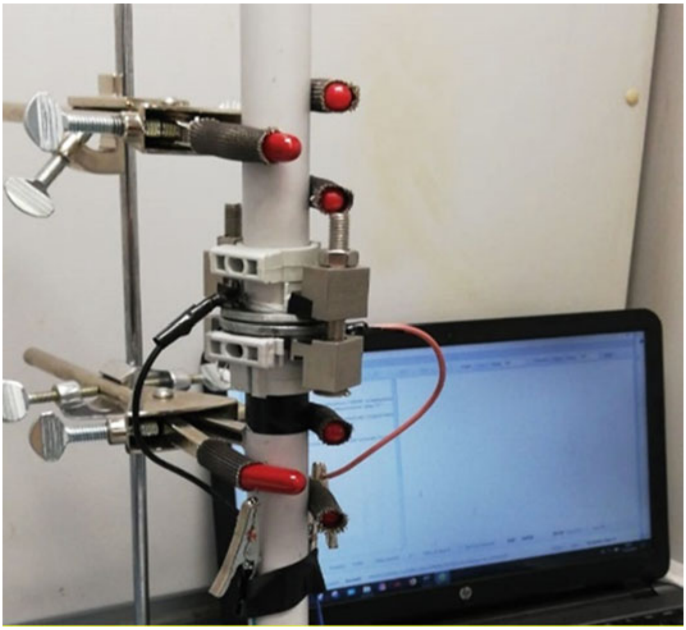

**Fig. 4** The realized setup for the impedance measurement of the graphene filter

**Table 1** Reproducibility test: the obtained standard deviation values for the imaginary part of the impedance

| Frequency (kHz) | Standard deviation |
|---|---|
| 25 | 6.11 |
| 50 | 3.99 |
| 75 | 1.47 |

## 4 Results and Discussion

The system in Sect. 3 has been employed to detect the state of the graphene filter, when it is used to remove acetonitrile, a toxic solvent of organic nature of wide industrial use.

Three different case studies have been analyzed: in the first two cases, the filter is used to adsorb the pure pollutant, whereas in the third case, the filter is used to clean a solution of water and pollutant. In all cases, even if the experimental results have shown appreciable sensitivities both in the real and imaginary part of the impedance, due to the adopted setup, the variation of imaginary part seems to show a better repeatability. Hence, in the following, we will refer to the measured reactance. Table 2 reports the initial values of the measured reactance, $X_0$, for the considered case studies, confirming the good reproducibility observed in Sect. 3.

The impedance measurement on the graphene filters has been then carried out, by varying the pollutant concentration until the saturation of the filter is reached.

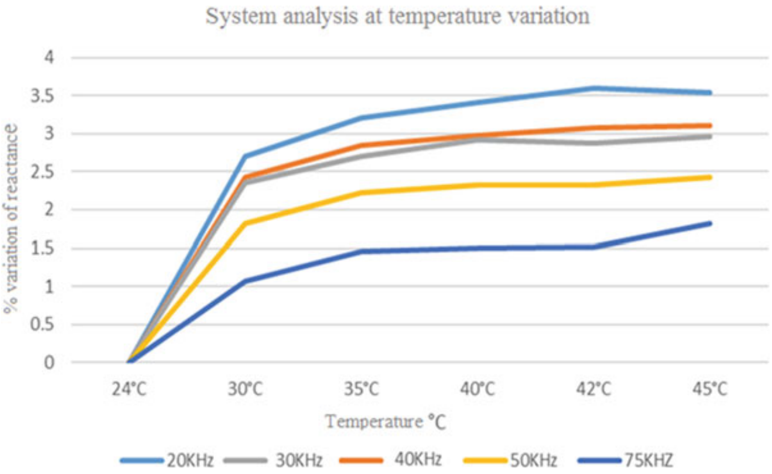

**Fig. 5** Variation of the measured reactance vs. temperature, for five different frequency values

**Table 2** Measured initial value of the reactance (at 75 kHz) of the filters used for the analyzed case studies (0% pollutant concentration)

| Case study | Initial reactance $X_0(\Omega)$ |
|---|---|
| 1. Filter with pollutant only | 16.36 |
| 2. Filter with pollutant only | 16.37 |
| 3. Filter with polluted water | 16.62 |

Specifically, the saturation is detected when the reactance is found to reach a stabilized value.

The results for the first two case studies are given in Fig. 6, which shows the time evolution of the relative reactance variation, defined as:

$$x_r(t) = \frac{X(t) - X_0}{X_0} \tag{1}$$

where $X(t)$ is the reactance measured at the time instant $t$. The filters 1 and 2 exhibit a good sensitivity to the presence of pollutant, since the relative variations of its reactance are of the order of some percent. In addition, a maximum relative variation of about 4.6% is reached when the two filters saturate. By weighing the filters before and after the experiments, it has been evaluated that they saturate after adsorbing about 0.18 ml of pollutant.

The third case study refers to the use of the same kind of filter to clean a solution of water and pollutant. The time evolution of the measured relative reactance value is given in Fig. 7. The relative variation exhibits the same qualitative behavior as for Case Studies 2 and 3, with a slightly higher sensitivity level, since now its value at saturation is about 6.5%.

These results confirm that the proposed monitoring system retains enough sensitivity to sense the presence of the pollutant in the filter and is also able to detect its saturation. In addition, the qualitative and quantitative behavior is very

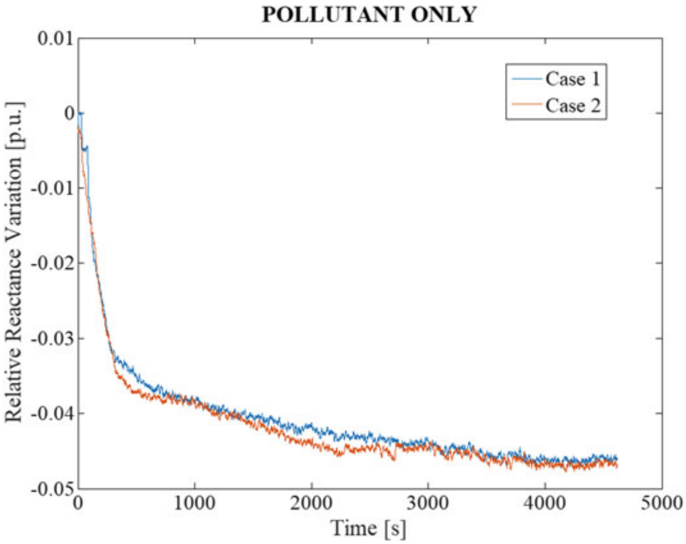

**Fig. 6** Time evolution of the measured relative reactance variation (Eq. 1) for Case Studies 1 and 2 (filters with pollutant only). The reference values are given in Table 2

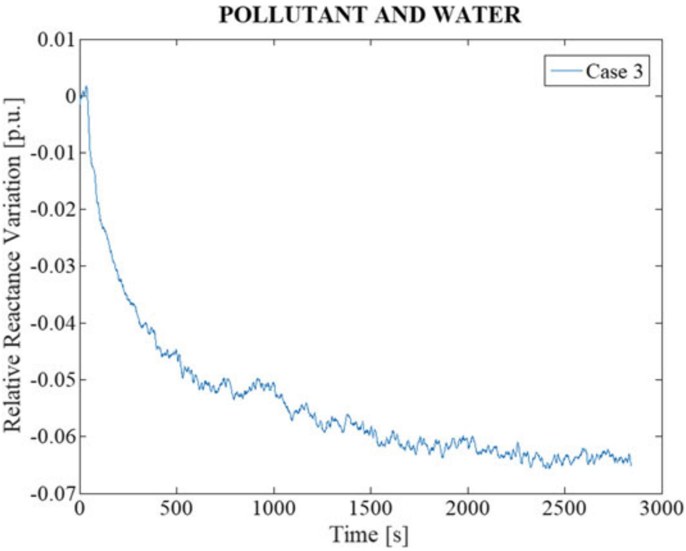

**Fig. 7** Time evolution of the measured relative reactance variation (Eq. 1) for Case Study 3 (filter with polluted water). The reference value is given in Table 2

stable from sample to sample, so that the good reproducibility shown in Sect. 3 is here confirmed.

## 5 Conclusions

An experimental setup has been proposed and validated, to monitor the state of a graphene water filter, based on the measurement of the electrical impedance as a function of the pollutant concentration.

The graphene filter has been fabricated from a low-cost material (intercalated graphite), by means of a simple and industrially scalable technique based on heating, sonication, and pressing.

The proposed monitoring system has been shown to be sensitive to the pollutant presence, with a relative variation of the reactance of the order of some percent (up to about 5%). In addition, the system can detect the state of saturation. Experiments carried out on different filters have also demonstrated a good degree of reproducibility of the results as well as their stability with environmental conditions.

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