A Carbon-based NTC Resistive Temperature Sensor for Edible Electronics

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***Abstract* — A negative temperature coefficient (NTC) resistive sensor implemented using only food-grade materials, namely activated carbon, gelatin candy, gold, ethyl cellulose, and beeswax, was implemented. We envision this sensor might find future applications in measuring temperature within the gastrointestinal (GI) tract. Differently from other edible implementations typically based on ionic conductivity, the device herein presented leverages the electronic conductivity of activated carbon and therefore benefits from a simple readout circuit. The sensor features a sensitivity of -0.32% °C^{-1} and high linearity within the range 5 – 50 °C. Comparative analysis with a commercially available component reveals analogous functional output. The inherently safe-to-eat feature of the component shows a feasible pathway to miniaturized digestible platforms for GI tract monitoring, as well as sustainable solutions for agrifood and environment monitoring purposes.**

Keywords* — *Edible electronics, green electronics, green sensors, edible sensors, conductive coatings

I. INTRODUCTION

Temperature is one of the most frequently measured physical variables as it can influence a wide range of phenomena, from biological processes to physical states [1]. The choice of a specific temperature sensor is application-dependent and is based on the temperature range, accuracy, speed of response, and cost required by the application [2]. Thus, several temperature sensor implementations have been developed and integrated in multiple formats, such as thermocouples, thermistors, and integrated circuit-based sensors [1, 3, 4].

In healthcare, body temperature is a crucial parameter of human health, and its real-time monitoring is an immediate diagnostically relevant information [5]. Recently, significant attention has been directed towards the temperature of the gastrointestinal (GI) tract [6, 7]. GI temperature monitoring (normal range 36.3 °C [8] and 38.5 °C [9]) provides insights into the physiological state of the digestive system and can identify inflammation, infection, or other pathological conditions, assisting in uncovering such conditions early and enabling prompt treatment and better patient outcomes [7]. The GI temperature information can also be used in combination with other ingestible sensors, such as pH, as a reference condition and to assess the position of the ingestible device [10]. Ingestible systems capable of measuring several physiological parameters, including temperature, have been approved by the Food and Drug Administration (FDA) and are commercially available [6, 7, 10 - 12]. Among the different types of ingestible temperature sensors, thermistors are a suitable solution for applications that require high sensitivity, cost efficiency, and miniaturization [8, 13]. Despite the growing interest in ingestible systems, these devices still suffer from certain drawbacks [14]. In particular, the material selection remains one of the main limitations of ingestible devices [14 - 16]. For implantable medical devices, the FDA typically does not regulate materials; instead, it assesses the entire architecture. As such, potentially toxic materials can be employed in ingestible systems when properly encapsulated. However, in cases of device malfunction, toxic materials might leak from the encapsulant and represent a health hazard. Even in the absence of toxic materials, in case of retention of non-degradable devices or components, surgical extraction might be necessary.

Hence, edible electronics emerged as a research field aiming at increasing the safety of devices for GI monitoring by employing food-grade materials in place of potentially toxic ones [17]. The European Food Safety Agency (EFSA) has done extensive work in identifying safe-to-eat materials and their respective "no observed adverse effect level" (NOAEL). Among those, several materials exhibit interesting electronic properties and have been characterized for potential use in edible electronics [17, 18]. Proof-of-concept edible electronic components have been demonstrated, such as resistors [19], capacitors [20], transistors [21], resonators [22] and power sources [23 - 25]. Edible sensors are also progressively emerging [26 - 30], including temperature sensors [31 - 36]. Gels, like carrageenan, polyvinyl alcohol (PVA), and apple pomace, have been used to develop edible sensors for oral cavity temperature monitoring and tested on human patients [31]. Edible glutinous rice has been engineered into an ionically conductive gel e-skin that can monitor different body parameters, including temperature [32], while gelatin has been used in both a capacitance [33] and an electrochemical-based temperature sensor [34]. All such sensors exploit the ionic conductivity of the gel and the changes in ion mobility associated with temperature variations. Gelatin has been exploited also in a ferroelectric sensor, which allowed the development of a biodegradable e-skin capable of monitoring temperature and pressure [35]. A reoccurring limitation of edible temperature sensors developed so far is that they are not directly compatible with edible energy sources currently available, which can provide only a small constant voltage output (0.65 V [23]) and cannot support complex readout circuits. To overcome this limitation, a self-powered edible temperature sensor based on an ionochromic cell was demonstrated [36], although providing only a qualitative indication of a temperature range.

Here we present an edible resistive temperature sensor, i.e. a thermistor, compatible with currently available energy sources. The sensor uses a previously developed edible composite material based on activated carbon (AC) and gelatin-based Haribo gummy [19]. Effective encapsulation of the active material against competing humidity effects was achieved using only food-grade materials, namely edible gold (E 175), ethyl cellulose (E 462), and beeswax (E 901). The resulting thermistor exhibits a negative temperature coefficient (NTC) behavior, with a sensitivity of -0.32% °C^{-1}

in the 5 – 50 °C range. We demonstrate that the edible thermistor can be operated in the same way as commercial components, producing comparable output signals. Such a result demonstrates the feasibility of edible thermistors in the context of future edible electronic systems for safe-to-ingest devices and sustainable environmental sensors.

II. THE EDIBLE TEMPERATURE SENSOR

The temperature coefficient α describes the temperature behavior of a resistive material as:

$$R = R_{\mathrm{ref}} \cdot [1 + \alpha(T - T_{\mathrm{ref}})] \tag{1}$$

where R is the resistance at temperature T, and R_{ref} is the resistance at a reference temperature T_{ref}. AC is an electronic conductor and is known to be sensitive to temperature (with a negative temperature coefficient) [37, 38] and humidity [39]. In previous works, we have developed an edible conductive composite ink composed of AC, Haribo gummy, and ethanol-water dispersant suitable for spray-coating with a sheet resistance of ~50 Ω cm when used in a filler-binder ratio of 200% [19]. However, to enable the use of this AC composite as the functional material of a resistive temperature sensor, the competitive humidity effect must be suppressed. We envisioned suppressing humidity dependence by providing the device with a humidity-proof barrier. Therefore, the device was prepared as outlined in Fig 1a. A thin ethyl cellulose film was obtained by casting 40 ml of a solution of ethyl cellulose in ethanol (20 g L^{-1}) into a glass Petri dish (diameter 9.5 cm) and drying the film overnight at 60 °C. The film was peeled off and a 4 x 4 cm^2 edible gold foil was attached to it by lamination [23]. Edible gold was then selectively removed using a wet swab to create an exposed ethyl cellulose area of 1 x 1 cm^2 and two rectangular gold electrodes. 3 mL of ink with 200% AC was deposited onto the area by spray deposition as in [19]. The custom setup for spray deposition utilizes a commercial device with a 750 µm nozzle (Paasche VL Series) supplied with compressed air (2 bar). The ethyl cellulose film was then folded, thus sandwiching the AC-based coating and the gold electrodes, and hot pressed at 120 °C, 10 MPa, for 2 minutes (PI-KEM model YLJ-HP60-LD). The sample was then dip-coated into melted beeswax (80 °C) three times, encapsulating the device, except for the two gold contacts used to access the active material.

The edible thermistor shown in Fig 1b is constituted only of materials that are approved by EFSA as safe to eat. Activated carbon (AC, E 153, Fig 1c) is a food colourant with no reported safety concerns up to 10000 mg kg^{-1} [40]. Haribo gummy is a commercial food material. Edible gold leaf (E 175) is approved as food decoration [41]. Ethyl cellulose (EC, E 462) is a food emulsifier with a NOAEL of 660-900 mg per kg of body weight per day [42]. Beeswax is a glazing agent (E 901) with a NOAEL of 1.1 g per kg of body weight per day [43]. The thermistor has been developed without overcoming the NOAEL values of all the edible materials used (Table 1).

III. RESULTS AND DISCUSSION

a. Pristine AC composite

The resistance of the pristine AC composite in different temperature and humidity conditions was first analyzed. Three identical rectangular samples (2.5 x 0.5 cm^2) were obtained by spray depositing the AC composite (0.6 mL cm^{-2}) onto plasma-treated (1 min at 20 W) microscope glass slides. The material was introduced into an environmental chamber (Memmert HPP110eco) and its resistance was measured using an external precision source meter (Keithley 2612B, with a 100 mV bias) in a temperature range of 5 – 70 °C and a humidity range of 15 – 90%. The reference temperature was measured using the temperature sensor embedded in the environmental chamber. The electrical connection between the source meter and the samples was obtained using standard wires passing through a cable window embedded into the environmental chamber. First, resistance measurements at different temperatures (5 – 70 °C) and at a constant relative humidity (RH = 15%) were carried out and reported in Fig 2a.

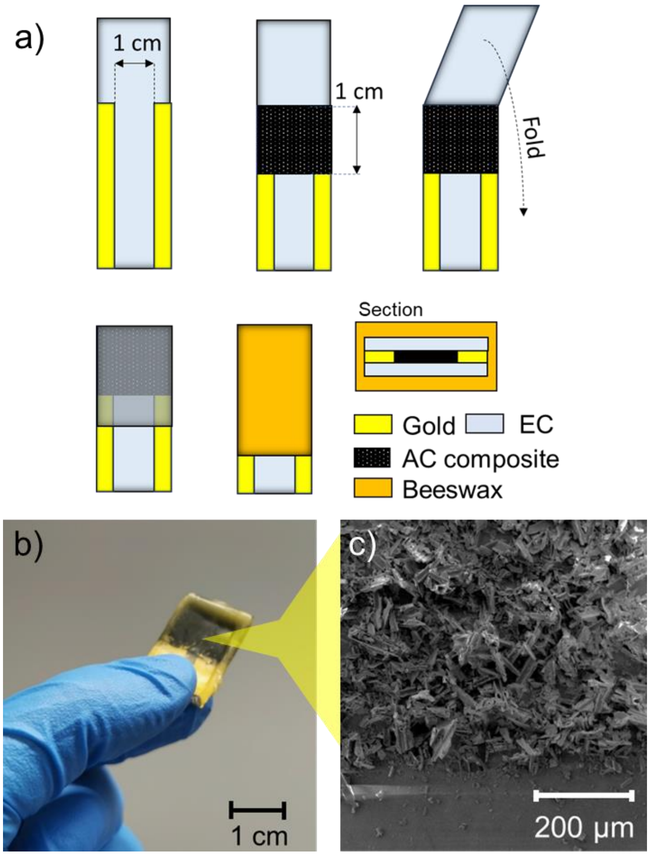

Fig 1. (a) Illustration of the manufacturing process. (b) Demonstrative image of the edible thermistor. (c) Scanning Electron Microscopy (SEM) image of the functional layer of AC composite deposited onto ethyl cellulose.

TABLE I. MATERIALS OF THE EDIBLE TEMPERATURE SENSOR

Material	E number	Amount per sensor (mg)	NOAEL*	Ref.
Activated carbon	E 153	60	~ 1000	[40]
Haribo gummy	n.d. (food)	30	N/A (food)	n.d.
Gold leaf	E 175	2	Not available	[41]
Ethyl cellulose	E 462	50	660-900	[42]
Beeswax	E 901	564	~ 1100	[43]

* Expressed in mg per kg of body weight per day.

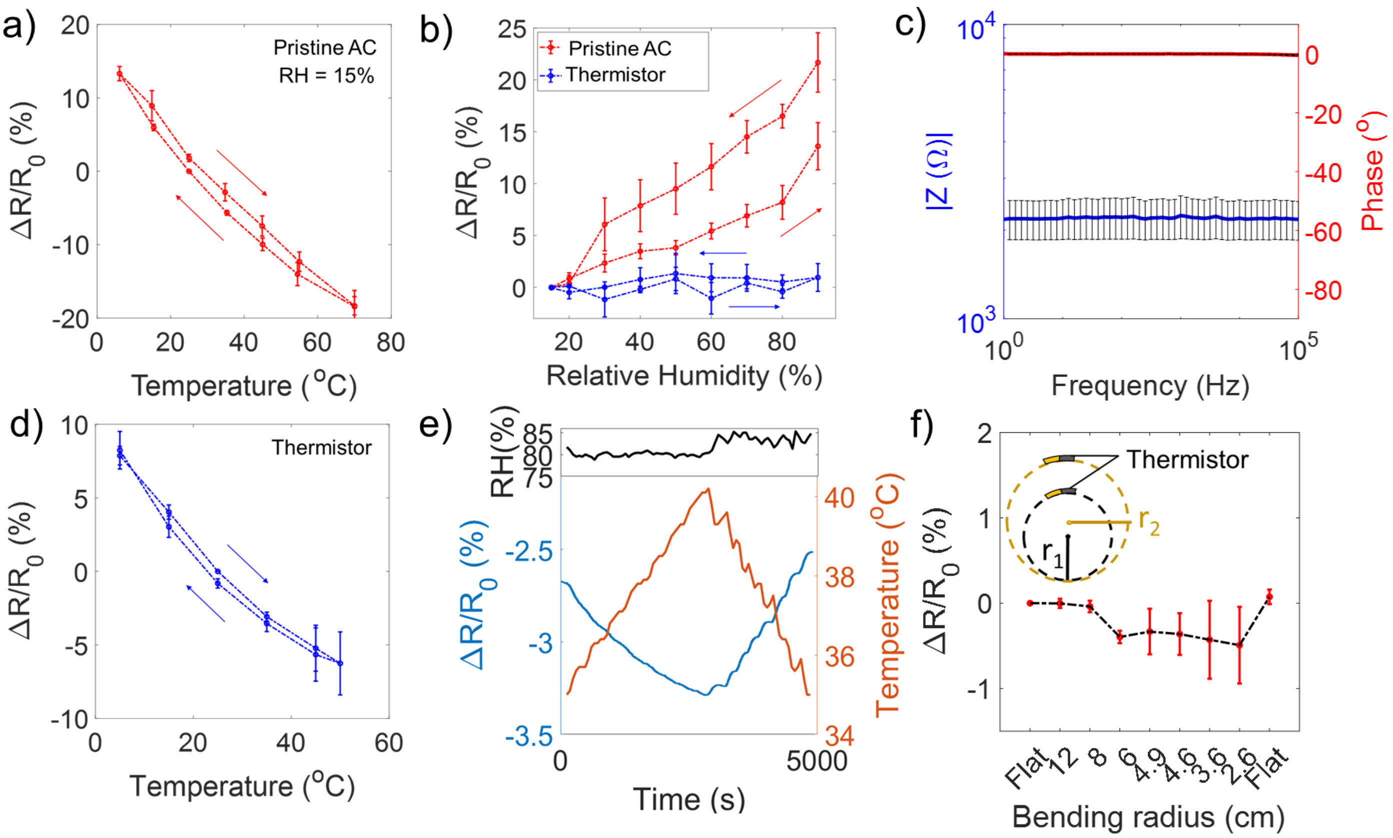

Fig 2. (a) Relative resistance variation vs temperature at a constant RH = 15% of the pristine material deposited on glass. Average and standard deviation over three identical samples. (b) Relative resistance variation vs RH at a constant temperature (25 °C) of the pristine material (red) and thermistor (blue). Average and standard deviation over three identical samples. (c) Average of the impedance modulus (blue) and phase (red) of the sensor over three identical samples at room temperature (~19 °C), demonstrate the electronic conduction nature of the component. (d) Relative resistance variation vs temperature of the edible thermistor. Average and standard deviations over three identical samples. (e) Resistance variations of a single thermistor (blue) operating in a simulated gastric environment with a temperature between 35 and 40°C (red) and relative humidity between 80 and 85% (black). Temperature is increased/decreased with 0.7°C steps. (f) Relative resistance variation vs bending radius at constant temperature and humidity. Inset: measurement setup. Average and standard deviation over three identical samples.

R values were normalized to the reference temperature R_{ref} measured at the reference temperature (T_{ref} = 25°C) according to the following equation:

$$\Delta R/R_0\ (\%) = 100 \cdot (R - R_{ref})/R_{ref} \qquad (2)$$

Fig 2a shows that the resistance of the pristine composite decreases when increasing temperature, i.e. NTC behavior was observed. The temperature coefficient was quantified to be $\alpha = -4.7 \cdot 10^{-3}$ °C^{-1} calculated as per eq. (1).

By linear fitting of the curve, a sensitivity of -0.49% °C^{-1} and a linearity of $R^2 = 0.956$ were determined. Subsequently, the humidity-dependent resistance variations of the pristine AC composite were evaluated. Data in Fig 2b indicates that the resistance of the exposed coating increases by increasing the relative humidity, with relative resistance variations up to 20% with 15 - 90% RH at a constant temperature of 25 °C. Hysteresis, quantified to be 1.8% at 25 °C, was also observed, indicating a possible difference in the rates of adsorption and desorption of moisture in the material. Such evidence confirms the need for a humidity-proof barrier to obtain reliable temperature sensing through the edible AC composite.

b. *Activated Carbon Thermistor*

The encapsulated device depicted in Fig 1a, defining the proposed edible thermistor, was then characterized using an identical setup. Data reported in Fig 2b shows that the encapsulation technique is successful in preventing humidity effects, as shown by the absence of an obvious trend with humidity, and by a maximum observed resistance variation of +1.3% in the 15 - 90% RH range. Impedance spectroscopy demonstrates that upon encapsulation the edible composite preserves its electrically conductive behaviour and that the average and standard deviation of the sensor resistance calculated over three identical samples is 2175.7 ± 325.9 Ω at room temperature (Fig 2c).

The temperature test range of the thermistor was adjusted according to the theoretical melting point of beeswax (~64 °C). The calibration curve of the thermistor was obtained in the range 5 – 50 °C with no humidity control - see Fig 2d. The thermistor shows a NTC of $\alpha = -2.9 \cdot 10^{-3}$ °C^{-1}, hysteresis of -0.82% at 25 °C, sensitivity of -0.32% °C^{-1}, and linearity with $R^2 = 0.919$. As proof of operation inside the GI tract, the thermistor was dynamically measured in a simulated GI environment, namely within a temperature between 35 and 40°C and high humidity (80 – 85%).

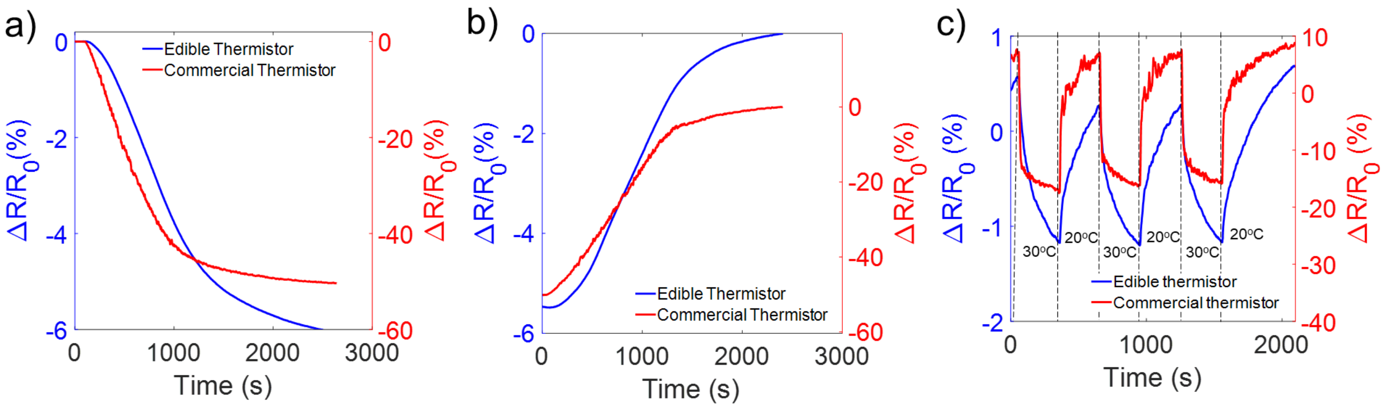

Fig 3. Output curves from a commercial NTC thermistor (red) and one edible thermistor (blue) (a) during a heating cycle where the temperature is gradually increased from 25 to 50 °C; (b) during a cooling cycle where the temperature is gradually decreased from 50 to 25 °C; (c) during a cyclic test where sensors were exposed to two different environments at 20 and 30 °C (step response).

To mimic a temperature change in the GI tract, the reference temperature was increased/decreased with 0.7 °C steps. Fig 2e shows that the device exhibits a detectable and reliable output which is highly compatible with the reference temperature. This simulated experiment represents a preliminary demonstration that the device can be used for GI temperature monitoring.

Despite the use of beeswax, the sensor retained a certain degree of bendability provided by the use of the thin ethyl cellulose substrate. To quantify the resistance variations produced by the mechanical deformation, three identical samples were attached to curved surfaces with progressively reducing radii, thus producing a controlled bending of the device (inset Fig 2f). The resistance of the samples were measured in each condition. Fig 2f illustrates that bending radii ≥ 8 cm produce virtually identical results. Further reduction of the bending radius creates an increased relative resistance variation reaching its maximum of -0.5% with a bending radius of 2.6 cm. However, a full recovery of the initial resistance was observed when samples were brought back to their initial position. We therefore assume that bending the device with bending radii ≥ 2.6 cm do not produce any obvious and permanent damage.

The output of the thermistor was validated against an external NTC commercial thermistor (MF52B, 10 kΩ at 25 °C) – see Fig 3. For this aim, the output signals from both the edible and commercial thermistors were collected simultaneously using two different channels of the same source meter during heating (25 °C → 50 °C) and cooling (50 °C → 25 °C). Both the sensors were operated in the same conditions with an applied 100 mV bias. Although a lower amplitude was observed, the output of the edible thermistor is highly comparable with the one produced by the commercial sensor in both heating (Fig 3a) and cooling (Fig 3b). The output of the edible thermistor remains stable and reliable also in cyclic conditions, applying repeated step temperature changes (Fig 3c). The sensors were alternatively exposed to two different environments with a controlled temperature of 20 and 30 °C to produce the step temperature changes. Although with a slower response time with respect to the commercial component, the thermistor provides a stable signal also in dynamic conditions, and its output remains highly compatible with the commercial component. We speculate that the slower response is related to the high thermal capacity of the device compared to the commercial thermistor, due to the use of the beeswax encapsulant and the large size of the device.

c. Discussion

The bendability of the component can be improved in future implementations by replacing beeswax with different edible coatings and film barriers, such as oleogels [44] or shellac-based composites [45]. Starting from our results, further efforts towards miniaturization of the component will allow its integration into edible capsules for GI temperature monitoring. Miniaturization will be favoured by the adoption of a different encapsulant (beeswax currently accounts for 80% of the device in weight) and will decrease the thermal capacity of the device, thus reducing its response time. In its current form, the component is also suitable for other application scenarios. In particular, the sensor is composed of edible materials only, so there is no contamination risk when in contact with food, thus enabling sensors-on-food applications. The sensor is also inherently compostable, therefore it can be disposed of as food waste after its use in agrifood applications. The sensor may be also considered for a broad range of environmental probing, including soil and water, with the further advantage of not representing a hazard for wildlife.

The edible thermistor can benefit from standard readout circuitry for resistive sensors. In fact, in our test the edible and the commercial thermistors were controlled using an identical setup and with a constant voltage bias. As a result, readout circuits commonly in use for thermistors, such as voltage dividers, differential amplifiers and filters, are also applicable to our implementation to further enhance the signal quality. The adoption of edible energy sources [23] and edible components [17, 18] is under study in order to deliver a fully edible system [46]. Future works will entail the analysis of the degradability of the components within the human body and in other relevant environmental conditions.

IV. CONCLUSION

A resistive temperature sensor was produced using only food-grade materials. The sensor exploits the electronic conduction of an AC-based composite as the functional layer and therefore complements, and overcomes some limitations of the use of hydrogels adopted in previous applications, including the compatibility with edible energy sources [23].

The sensor exhibits a negative temperature coefficient, with a sensitivity of -0.32% °C^{-1} and high linearity ($R^2 = 0.919$) in the range 5 – 50 °C. When compared to a commercial component, the edible sensor provides functionally equivalent output. We believe that the findings herein presented offer a promising path to a new class of edible thermistors that can be integrated into miniaturized digestible systems for point-of-care monitoring of the GI tract, as well as offering sustainable alternatives for environmental monitoring devices.

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