



# Temperature Sensors Manufactured from Edible Materials Intended for Oral Cavity Operation

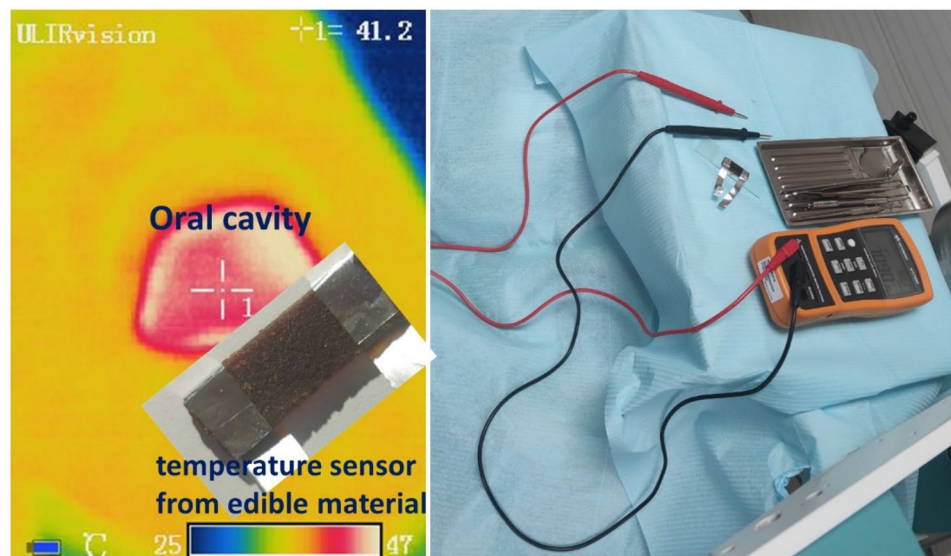
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## Abstract

Following the circular economy concept, all areas of science, including electronics, require individual approach. The green electronics approach implies processing natural based materials for development of electronic components even for single use, so that waste does not exist. This paper describes the production of thermistors from 3 different types of edible materials, their testing in laboratory conditions, as well as the application for measuring the temperature in the human oral cavity. To make the thermistor, three types of natural materials were used (carrageenan, polyvinyl alcohol and apple pomace-based). After fabrication, materials were poured into molds. Aluminium foil contacts were placed so that the electrical characterization of the materials could be performed. Several parameters were measured: resistance ( $R$ ), impedance ( $Z$ ) and the phase angle ( $\theta$ ) as a function of frequency. In order to determine whether the thermistors have a positive or negative temperature coefficient and to determine their sensitivity, measurements were conducted at three different temperatures: 25 °C (room temperature), 37 °C (human body temperature), and 50 °C. The thermistors demonstrated negative temperature coefficient behaviour. Additionally, it was demonstrated that the apple pomace-based thermistor can successfully measure healthy volunteer's temperature.

## Graphical Abstract



**Keywords** Edible electronics · Temperature sensor · Oral cavity · Impedance spectroscopy · Saliva

Extended author information available on the last page of the article

## 1 Introduction

Conventional electronic components inside the body have attracted widespread interest in biomedicine, especially in monitoring some vital parameters of our bodies—from oral cavity to gastrointestinal tract. Although these components are usually the size of pills, there is a possibility of being lodged and causing some health issues or inconveniences for the patients [1]. Because of that, scientists have started to develop edible electronic components [2] and food-based devices [3] that are safe to be swallowed. The possibility of being lodged is minimal or the same as during the eating process. These devices are biodegradable, meaning they can be taken regularly as we take our breakfast or lunch, and are environmentally friendly or belong to the green electronics field. It was necessary to develop new food-based materials to provide faster growth of this scientific field and potential theranostics application [4]. Considerable attention is focused on developing environmentally friendly approaches for the materials and manufacturing processes used in wearable sensors [5]. The process of human digestion starts in our oral cavity. Because of that, it is essential to develop sensors that can detect the mouth's main parameters, such as temperature, pH, conductivity, bacteria presence, etc. [6, 7]. However, manufacturing these sensors from edible materials is a serious challenge. Further exploring advanced nanomaterials and inclusion of edible materials, is needed to increase sensor performance, and to expand their potential applications [8].

From above-mentioned parameters, the detection and monitoring of intra-oral temperature is one of the most important in dentistry and medicine (gas temperature in continuous monitoring of a mechanically ventilated newborn [9], for example). By measuring intra-oral temperature, we can detect periodontal inflammation, dental caries complications, peri-implantitis, reaction to some medications, etc. A commercial temperature sensor (MAX30208) was used in mouthguard-like monitoring device for measuring *in vitro* and *in vivo* temperatures, and for studying errors in temperature readings [10]. The average intra-oral temperature (33.9 °C during the day and 35.9 °C during the night) and pH value were measured by commercial pH meter with thermocouple connected with ordinary wires [11]. The temperature sensor fabricated on flexible polyimide foil based on Pt resistor and Au interconnected lines, which can be rolled around the dental implant, was reported in [12]. The chip thermal sensors arrangement was proposed in [13], in order to identify the tongue tumour. There have been some trials aiming to develop and test biopolymer film-based sensors that can be attached to a human tooth, and to establish function between changing the resonant frequency and temperature, or glucose concentrations [14].

The edible sensors represent a new class of biocompatible devices, which can be used for the detection of various biofluids important for determination of our health status [15]. For example, chewed gum can be used for developing a variety of sensors for healthcare monitoring [16]. Conductivity and mechanical properties of hydrogels based on edible biopolymers gellan gum and gelatine, with almost 97% water content, were studied in [17]. Cyclic voltammograms were recorded in [18] for edible structures in hollow tubular shape (e.g., penne, green beans, candy) filled with active charcoal, used for detection of salivary uric acid, gastric ascorbic acid, as well as dopamine in intestinal fluid. The first trial for fabrication of partly edible component that can measure saliva conductivity was reported in [19], with an aim of detecting luteinizing hormone (Ovulation cycle monitoring), as well as chronic kidney disease. Authors developed molded soft candy, but they used non-edible aluminium tubes as terminals (in order to have through hole electronic component). The resistive temperature sensor for out-of-body operation was reported in [20], where edible Mg film was used as an active layer between two dielectric layers and ecoflex layer for substrate/encapsulation. The application was demonstrated on measuring temperature of fish skin to track its temperature during shipping and storage. However, the area of edible electronics is still in its infancy. Processing new food-based materials and their application for measuring important parameters of our body are very important for further development of this field, as well as renewable and environmentally friendly energy sources to power [21].

Here, we studied three edible materials: carrageenan, polyvinyl alcohol and apple pomace, as well as their application in thermistor manufacturing for measuring the temperature in the oral cavity.

Carrageenan is a natural compound (sulphated polysaccharide) obtained from red seaweed (red algae). Carrageenan-based formulations can be applied to add benefits to scaffold in tissue engineering [22], drug delivery nanocomposites or wound healing [23, 24]. Carrageenan is water soluble, has low cytotoxicity, and has antimicrobial and antioxidant properties. Carrageenan and gel were used as sponges for hemostatic dental applications, taking into account their high blood holding capacity [25]. Poly-(vinyl alcohol) (PVA) is an easily available polymer with a wide range of applications that consists of many hydroxyl groups. It is biodegradable, has a very good chemical resistance, and it is nontoxic [26]. The conductive PVA/silver nanoparticles hydrogel was applied for sensing finger and wrist bending, swallowing, and nodding [27]. Apple pomace demonstrated antioxidant properties, with a polyphenol composition that include hydrocinnamic acids, dihydrochalcones and ascorbic acid [28] and may be treated as a material with very good health-promoting properties [29]. Temperature affected the electrical parameters of apple pomace, e.g., dielectric loss

[30], and authors stated that moisture content had high influence on temperature behaviour of apple pomace.

In this paper, a proof of principle of manufacturing temperature sensor from natural materials was presented, contributing to the further growth of the emerging field of green electronics. The complete characterization of the electrical, thermal, and structural properties of the edible thermistors was performed. SEM, XRD, FTIR, and wettability analysis were performed. Using impedance spectroscopy, resistance, impedance, phase angle at room temperature, as well as two higher temperatures were measured. The possibility of applying the apple pomace-based negative temperature coefficient thermistor for measuring temperature in oral cavity was also demonstrated.

## 2 Materials and Methods

### 2.1 Samples Preparation

Three types of edible materials were processed: Sample 1—carrageenan based; Sample 2—polyvinyl alcohol based; and Sample 3: apple pomace based. The procedure of their preparation was as follows:

- 3 g of iota-carrageenan (Sigma-Aldrich- USA) was dissolved in 43 g of a technical grade mixture of water/glycerol (glycerol content undisclosed) and the obtained suspension was homogenized (magnetic stirrer) during 8 days between 15 and 30 °C. A clear gel was obtained and used without additional treatment.
- 10 g of polyvinyl alcohol (POVAL PVA-205 bought from Kuraray, Japan) of low viscosity (4.6–5.4 mPa.s) and partial hydrolysis (86.5–89 mol%) was homogenized (magnetic stirrer) in 50 g of a technical grade mixture of water/glycerol (glycerol content undisclosed) during 48 h between 100 and 115 °C. A clear gel was obtained and used without additional treatment.
- 16 g of apple pomace and 27 g of sucrose (both bought from a local supermarket in Terrassa, Barcelona, Spain) were manually crushed and then carefully heated up to 110 °C under manual mixing. The obtained molten paste

was cooled at room temperature and used without additional treatment.

Dimensions of molds used for samples preparation for characterization of electronic parameters were 25 mm × 10 mm, while the thickness was 1.5 mm. After edible materials were heated and melted, they were poured into molds. Contacts made of aluminum foil were placed, providing two-terminal electronic components, leading to the instrument for measuring electrical characteristics. The electronic components created from three different edible materials are presented in Fig. 1.

### 2.2 Instruments

All edible samples were studied under Scanning Electron Microscopy (SEM) (Hitachi TM3030, Japan) to visualize internal structures of the materials that have influence on their electrical performances. The X-ray diffraction analysis of the investigated films was performed on a Rigaku, Mini-Flex 600 instrument, using Ni-filtered CuK $\alpha$  radiation. The ALPHA (Bruker, Germany) instrument was used for Fourier Transform Infrared (FTIR) analysis. The drop shape analysis (DSA) was performed using Drop Shape Analyzer DSA25B (Kruss GmbH, Germany). The chemical impedance analyser HIOKI IM3590 was used to measure the electrical characteristics of the prepared samples. In order to determine temperature behaviour of the proposed edible materials samples, hot air source was used. An infrared camera ULIRVision TI160 was used to monitor the exact temperature. Handheld LCR meter (Keysight model U1733C, Colorado Springs, USA) was used during in vivo experiments measuring resistance at five different frequency points when sensor was in the subject's mouth.

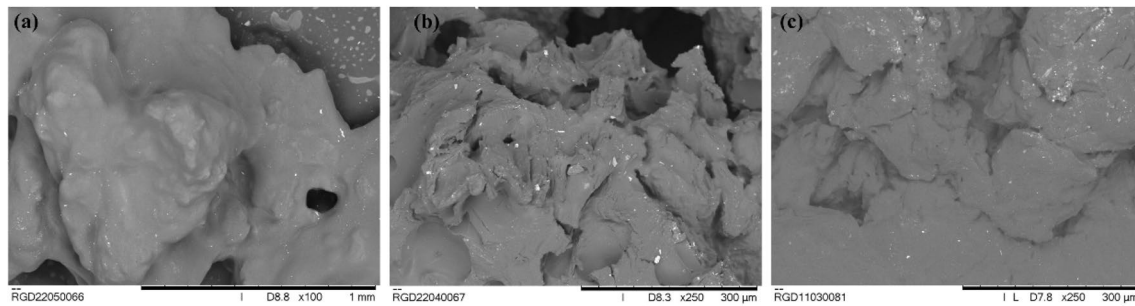
## 3 Results and Discussion

### 3.1 Materials Characterization

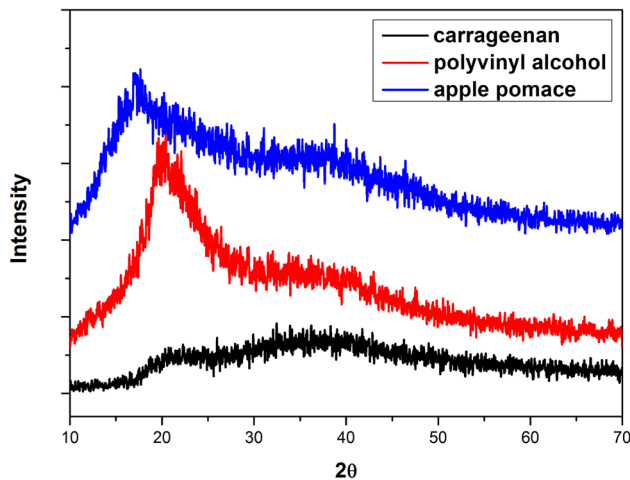
Scanning electron microscopy (SEM) micrographs of studied materials samples are shown in Fig. 2. It can be



**Fig. 1** Edible thermistors with aluminium contacts, made of **a** carrageenan, **b** polyvinyl alcohol and **c** apple pomace



**Fig. 2** SEM micrographs of **a** carrageenan, **b** polyvinyl alcohol and **c** apple pomace based edible samples



**Fig. 3** XRD patterns of analysed edible samples

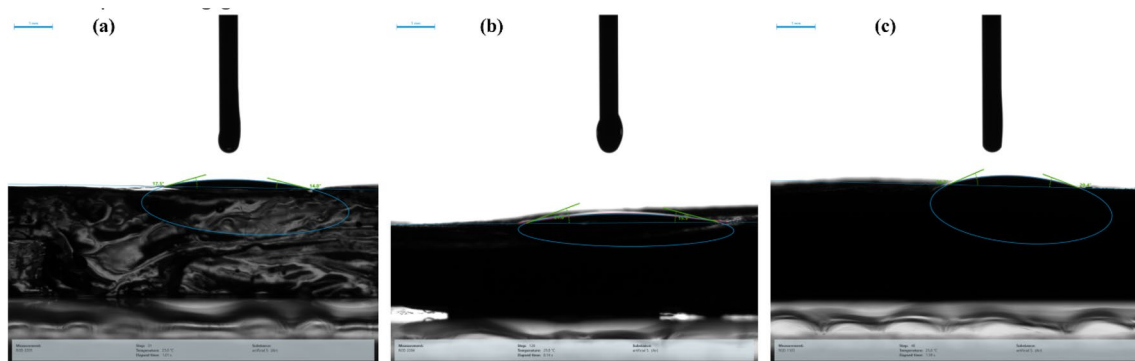
concluded that apple pomace structure had dense structure (this will have later consequence in the largest contact angle of this sample in drop shape analysis), whereas the carrageenan based sample had porous structure.

XRD patterns of analysed carrageenan, polyvinyl alcohol and apple pomace based edible samples are presented in Fig. 3. Amorphous nature of carrageenan is evident due to

the presence of two broad peaks near 23 and 35° [31]. On the other hand, PVA and apple pomace samples showed very small degree of crystallinity due the sharper peak near 21°, indicating the co-existence of a small nanocrystalline phase with the bulk amorphous phase [32] and [33]. FTIR spectra are provided in supplementary materials (Figure S1).

### 3.2 Wettability Analysis

Drop shape analysis (DSA) was performed to determine the contact angle from the contra-lit image of a sessile drop. The drops of artificial saliva were applied to the surface of the prepared samples. Since samples had high roughness, the DSA measurements could only be considered as an explanation of liquid-surface interaction. All three presented materials demonstrated very high absorbance. The measurements were recorded, and for the measurement point, the picture after 2 s was selected for all samples. When apple pomace-based material was treated with artificial saliva, the measured contact angle was 26.2° on one side of the droplet and 20.4° on the other (Fig. 4). During treatment of polyvinyl alcohol sample with artificial saliva, contact angles of 21.0° and 15.5° were measured, about 5° less than the apple pomace sample. In sample 1, the measured contact angle was around 14.0°. The apple pomace sample had



**Fig. 4** Drop shape analysis of **a** carrageenan, **b** polyvinyl alcohol and **c** apple pomace based edible samples



the largest contact angles and absorbed the artificial saliva the slowest. In contrast, carrageenan and polyvinyl alcohol samples had smaller contact angles and absorbed artificial saliva faster and more efficiently. Extremely high wettability of all samples qualified them as promising green/edible electronic materials.

### 3.3 Electroimpedance Spectroscopy Analysis

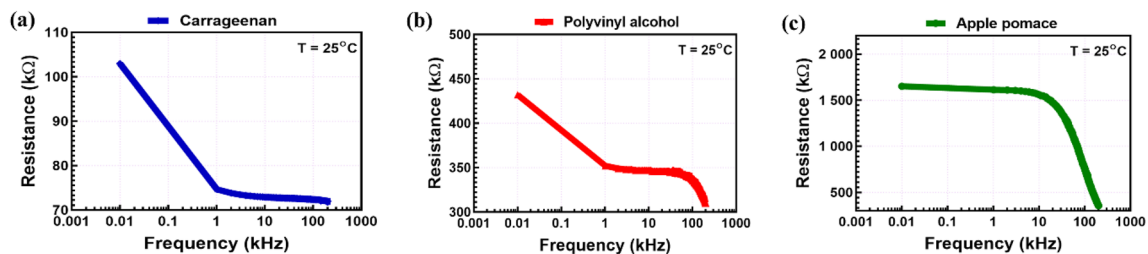
After samples were prepared, the characterization of edible thermistors' electrical properties was performed. First, electrical resistance ( $R$ ) of three samples was measured as a function of frequency at room temperature (25 °C). Measured results are shown in Fig. 5. Phase angle was also measured as a function of frequency. Phase angle was around 0 (Supplementary material Figure S2) for nearly the whole frequency range, confirming the resistive behaviour of the studied components. The same trend and similar resistance range (from  $10^2$  to  $10^6$ ), as presented in Fig. 5b, was obtained for PVA/polyvinylpyrrolidone (PVP) blend [34].

Afterwards, the measurement of electrical parameters was repeated at human body temperature (37 °C), at 50 °C, and at 80 °C, to examine the sensitivity of developed thermistors and determine whether they are positive or negative temperature coefficient thermistors. However, carrageenan sample was melted at 80 °C. In order to be able to compare three studied materials, this paper presents the resistance as a function of temperature up to 50 °C (Fig. 6), while the measurement results for polyvinyl alcohol and apple pomace based edible samples are shown in (Supplementary material

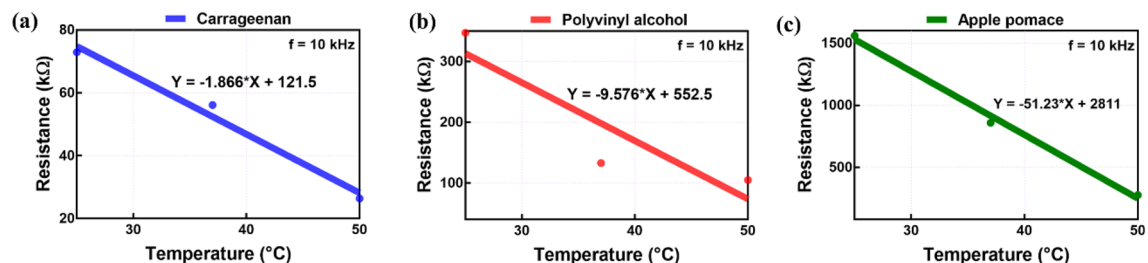
Figure S3). The frequency point of 10 kHz was selected to present temperature dependence of the analysed samples for two reasons: (1) because resistance has plateau for all samples around that frequency (Fig. 5), and (2) this frequency point is usually used for designing readout electronics.

Figure 6 shows that negative temperature coefficient (NTC) thermistor was manufactured and successfully tested. It can be concluded that its resistance decreased as temperature increased. The energy of atoms increased with elevating temperature, and consequently, the vibratory motion was more active, providing them with enough energy to become free and participate in the conduction mechanism. As a result, electrical resistance decreased. Negative temperature coefficient of PVA was also reported in [35]. The similar temperature range (20– 50 °C) was analysed in [14]. In addition, this temperature range is in connection with real-scenario application of this temperature sensor in oral cavity. Moreover, from practical point of view, it is more desirable to have NTC thermistor for application in oral cavity, and not PTC. Table 1 presents the temperature coefficient of resistance ( $TCR = (R_2 - R_1) / (R_1 (T_2 - T_1))$ ), as well as linearity coefficient calculated from Fig. 6.

During the molding process, these materials expressed different properties. Sample 1 had a gel-like structure, was hard to mold and lost shape easily. While Sample 2 was crumbling easily and had a slippery surface, Sample 3 was better for molding, sticky enough to attach electrodes to and had the most homogenous internal structure (which can be also seen in SEM micrograph in Fig. 2(c)). Additionally, Sample 3 had the best taste. Apple pomace-based sample



**Fig. 5** Electrical resistance as a function of frequency for **a** carrageenan, **b** polyvinyl alcohol and **c** apple pomace based edible samples



**Fig. 6** Electrical resistance as a function of temperature for **a** carrageenan, **b** polyvinyl alcohol and **c** apple pomace based edible samples

**Table 1** Characteristics of edible thermistors

Edible material	TCR (1/°C)	Goodness of fit ( $R^2$ )
Sample 1—carrageenan based	−0.0255	0.982
Sample 2—polyvinyl alcohol based	−0.0279	0.818
Sample 3—apple pomace based	−0.0329	0.994

demonstrated the best linearity (Table 1), and bearing in mind its proven highest stability, this sample was used in further analysis for in vivo study.

### 3.4 Apple Pomace Thermistor In Vivo Study

The apple-pomace temperature sensor was tested on a healthy volunteer (male, age 47). The volunteer signed informed consent before the test. The experiment was observed by the supervising researcher. Three real cases were simulated: (a) cold conditions in oral cavity—this was simulated in that way that subject ate ice, and using infrared camera, temperature measured in the mouth was around  $26 \pm 1$  °C; (b) normal temperature in the mouth ( $36 \pm 1$  °C); and (c) subject drank a cup of hot tea, and measured temperature in the month was  $41 \pm 0.5$  °C. The experimental set-up for performing this part of the study can be seen in Fig. 7. Short video on how sensor was positioned in the month is provided in Supplementary material (video V1).

The dimensions of the sensor were similar to the size of an average bite during eating, or big enough to cover the tongue. After the measurement, the subject could decide: (a) to swallow the sensor, because it was created from natural and edible materials, and in this case generated zero waste, or (b) to throw out the sample from mouth, in this case minimal waste is generated, and it is biodegradable or can be recycled and reused. In both mentioned cases, the main idea was to have a single used, cost-effective, natural material-based, eco-friendly electronic component.

Figure 8a presents measured resistance as a function of frequency on three different temperatures, whereas Fig. 8b shows resistance as a function of temperature at frequency point of 10 kHz.

Based on the measurement results, two thermistors models were also included in Fig. 8b. Namely, so-called  $\beta$  model and Steinhart-Hart (S-H) model [36]. Since values of measured resistance were in the range of k $\Omega$ , the S-H equation had to be modified with the corresponding reference resistance. In its original form, the value of the reference resistance was presumed to be 1  $\Omega$  [37]. The used formula is shown in Eq. 1.

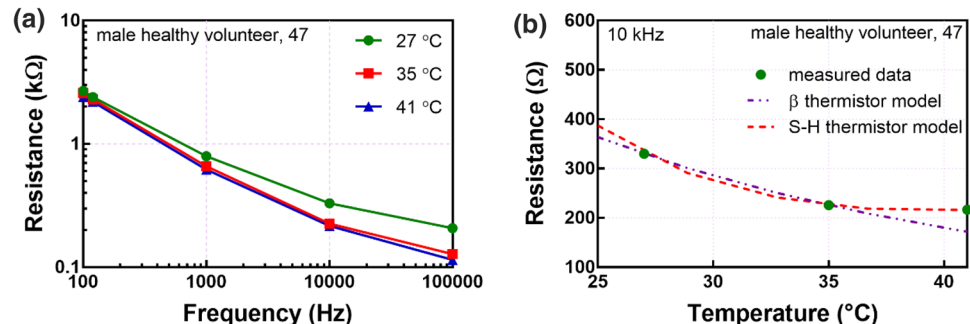
$$\frac{1}{T} = A + B \cdot \ln\left(\frac{R}{R_0}\right) + C \cdot \ln^3\left(\frac{R}{R_0}\right) \quad (1)$$

For modelling purposes, the following inverse equation of Eq. (1) was used:



**Fig. 7** Experimental setup for measuring temperature in the oral cavity, **a** sensor and instruments, **b** positioning the sensor in the oral cavity, **c** measuring exact temperature in the oral cavity using an infrared camera

**Fig. 8** **a** Resistance as a function of frequency for three different temperatures in the oral cavity, **b** thermistor characteristics



$$R(T) = R_0 e^{\left(A_1 + B_1 \frac{1}{T} + C_1 \left(\frac{1}{T}\right)^3\right)} \quad (2)$$

Equation 2 can be viewed as a series expansion of equation Eq. 3 used in the  $\beta$  model.

$$R(T) = R_0 e^{\beta \left(\frac{1}{T} - \frac{1}{T_0}\right)} \quad (3)$$

Equations 2 and 3 were used in the modelling process.

During the modelling process, the following steps were executed:

1. The reference resistance and temperature were chosen, and all temperature values were changed from degrees Celsius to Kelvin.
2. The coefficients for each model were calculated:
  - a. For  $\beta$  model, two measured points were used and  $\beta$  coefficient was calculated directly;
  - b. For S–H model, all three measurements were taken into consideration and a system of three equations was formed, through which the coefficients  $A_1$ ,  $B_1$  and  $C_1$  were calculated.
3. With the calculated coefficients, the models were formed and a range of temperatures were fed, giving out the resistance values, as can be seen in Fig. 8b.

The S–H model demonstrated a better fit in comparison to the  $\beta$  model, which was expected as it used all three measured point. Overall, the use of the S–H model is widespread. Compared to the  $\beta$  model, it showed a lower error margin and greater fitting capabilities over a wider range of values [38].

## 4 Conclusions

This paper presents a proof-of concept of the realization of three completely edible thermistors with a negative temperature coefficient that are entirely biodegradable and non-toxic. After a detailed analysis of the electrical properties at three different temperatures, it can be concluded that the proposed edible thermistors can measure the temperature in the oral cavity or some other biomedical applications. A complete structural analysis of the proposed NTC edible thermistors was also performed, including XRD, FTIR, drop shape analysis and electro-impedance spectroscopy. The suggested NTC thermistor from edible material (apple pomace based) can be used as an eco-friendly alternative for existing methods of measuring temperature in the oral cavity. It was demonstrated that S–H model perfectly fitted the NTC thermistor measuring data. The next steps of our study will be directed towards providing wireless readings

from temperature sensors in the oral cavity and testing with more volunteers.

**Supplementary Information** The online version contains supplementary material available at <https://doi.org/10.1007/s40684-023-00535-2>.

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**Author Contributions** All authors contributed to the study conception and design. Materials preparation was done by RGD, sensors creation was done by GMS, MR and SK. Testing of materials was done by SK, TK and JV. Data collection and analysis were performed by SK, BP and GMS. Modelling was performed by LM and MS. The first draft of the manuscript was written by GMS and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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**Data Availability** The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

## Declarations

**Conflict of Interest** The authors have no relevant financial or non-financial interests to disclose.

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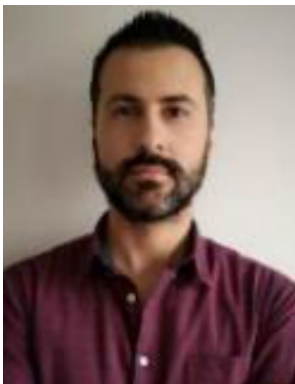


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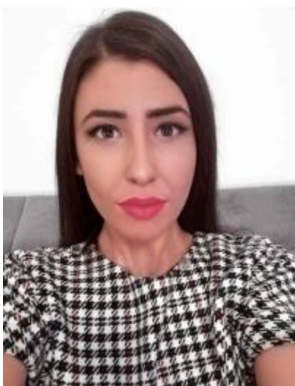
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