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Performance Characterization of a Biodegradable Deformation Sensor based on Bacterial Cellulose

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Abstract *– The diffusion of electronics in everyday life requires for the development of sensing systems, based on new materials and technologies. Deformation sensors are of interest in many application fields, ranging from assisted rehabilitation, virtual reality and augmented reality, and gaming applications, juts to mention a few. The diffusion of low-cost sensing systems outlines the need of environmentally friendly and low cost sensors. Cellulose is one of the most abundant material on the earth. Eventually, it is green, cheap and flexible. In the paper the deformation sensing properties of a three-layer structure based Bacterial Cellulose compound based on Bacterial Cellulose, impregnated by ionic liquids, and covered by Conducting Polymers are investigated. More specifically, it is shown that this novel composite is capable of sensing flexural deformations and producing a corresponding open-circuit voltage. The obtained results pave the road to the possibility of realizing a new class of deformation sensors that fits the requirements of more sustainable sensor technologies.*

Keywords— deformation measurements, paper-based sensor, bacterial cellulose, ionic liquids, greener transducers.

I. INTRODUCTION

The diffusion of electronics in everyday life imposes the needing of developing devices based on new materials and technologies. Moreover, we are in need of realizing environmentally friendly systems, as an answer to the request of sustainable development [1]. Also, low-cost and disposable technologies are required for facing the mass production of systems with sensing and actuating capabilities [2].

Deflection sensors will cover a significant part of the market. Such sensors will be the answer of relevant changes in Western society, where the aging phenomenon imposes the need of medical devices for rehabilitation or assistive living [3,4]. Further possibilities for mechanical sensor market will come from the recreational market, where sensors are employed for augmented reality or motion gaming [5].

Ionic ElectroActive Polymers (IEAPs) have been the object of big interest [6,7] since they allow for realizing scalable, light-weight, and flexible motion sensors [6,7]. Nevertheless, challenges exist for this class of materials and greener or better performing IEAPs are needed [8-15].

Cellulose has raised the researcher's interest as a possible bio-polymer to be used in the realization of greener electronics. The chemical structure is reported in Fig.1. It is among the most abundant and cheap biological composite, is flexible and light. Because of such properties it can represent a suitable candidate to the realization of flexible motion sensors. Eventually, it is ecofriendly and biodegradable. It is a biocompatible material which presents interesting mechanical and electrical characteristics. For this reason, this material have increased the interest of the scientific community since years, in several application fields including industrial biomedical and robotic applications [16]. Notwithstanding such interesting properties, the production of cellulose both requires significant energy and water consumption and implies the production of potential pollutants.

Recently Bacterial Cellulose (BC) has been proposed as a suitable substitute of plant derived cellulose [17,18]. It is cellulose produced by some kind of bacteria. BC and can be obtained in lab conditions by a greener process, and in very pure form, than conventional cellulose [19,20].

The electromechanical properties of BC have been already reported in the literature. More specifically, the transduction capability of BC, when used in a three layer structure, impregnated with Ionic Liquids (ILs), and covered with conducting electrodes, has been described [19-21]. Few contributions exist reporting on the mechano-electric properties BC based compounds. In [22], BC has been proposed for the realization of a piezoresistive sensor. Through a generating piezoelectric sensor has been proposed in [23], it uses cellulose filled with ZnO as active material. In [19], an engineered BC was proposed as the bulk of a piezoelectric sensor, and some preliminary results are given when the composite is subjected to compressive load.

The authors are currently involved in a research activity aimed at realizing green generating sensors, based on BC. More specifically, three layers compounds consisting of BC, impregnated with ILs and covered with Conducting Polymers (CPs) have been investigated as mechano-electric transducers.

In [23] authors propose an inertial sensor, realized by using plant derived cellulose, further filled with zinc oxide nanowires. This solution is complex approach and less green as respect the sensing method proposed in [24]. In fact, in this latter work, authors described some preliminary results on the possibility of using BC-ILs compounds for the realization of a green accelerometer.

In this paper, with the aim of realizing flexible and greener generating sensors, the possibility of using BC based compounds for realizing deformation sensors is investigated. The main aim is obtaining systems having the prerogatives of being: eco-friendly, bio-compatible, recyclable, environmentally safe, and biodegradable.

Results reported here extend preliminary investigations reported in [25]. More specifically, here that investigation is further carried on and new results are given on both the chemo-mechanical and transduction characterization of the sensor. Moreover, experimental evidence of a suitable mechanism that causes the mechanoelectrical transduction and the improvement of the performance in presence of ILs is given. The sensor operation is based on the mechanoelectrical transduction properties of this class of compounds. In fact, a voltage signal is produced at the polymeric electrodes, where it is collected in open circuit conditions, as consequence of ion motion.

To the best of the authors' knowledge, this is the first demonstration of this class of compounds as generating deformation sensor. Compared to other classes of IEAPs the proposed composite represents a step towards greener sensing devices. TABLE I summarizes various solutions, already proposed in literature, and a comparison in terms of characteristics.

Though biotoxicity issues have been raised for ILs, studies have been reported on the possibility of realizing biocompatible and green ILs [1,20]. This paves the road to the possibility of realizing even greener paper based sensors based on BC and ILs. It is worth to mention that future specific applications of this novel approach, thanks to the main advantages of the BC, such as flexibility, low cost, green, it finds several applications like disseminated measurement systems for environmental monitoring including structural monitoring, cultural heritage, distributed sensing systems in smart cities.

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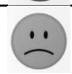
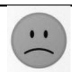
The paper is organized as follows: Section II will describe materials, the realized prototype and the methods used to characterize the composite and its transducing properties. Section III reports and discuss the obtained results, while concluding remarks are given in Section IV.

TABLE I. COMPARISON OF TECNOLOGIES FOR THE REALIZATION OF MOTION TRANSDUCERS

Motion transducer	***Nature of the sensor***	***Output***	***Output level***	***Flexibility***	***Environmental impact (green solution)***	***Reference***
Lead zirconate titanate (PZT)	*generating*	*voltage*	*high (tens of V)*	*low*		*[26]*
Silicon-based (MEMS)	*generating*	*voltage/resistive*	*medium/low (tens of mV)*	*medium*		*[27]*
Lead-free PZT	*generating*	*voltage*	*high (tens of V)*	*low*		*[28]*
Ionic polymer metal composite IPMC	*generating*	*voltage/current*	*low (up to tens of mV)*	*high*		*[29]*
Piezoresistive	*modificating*	*resistive*	*depends on the powering*	*high*		*[30]*
Ionic polymer-polymer composite IP^2C	*generating*	*voltage/current*	*low (up to tens of mV)*	*high*		*[14]*
BC	*generating*	*voltage*	*low (up to tens of mV)*	*high*		*This work*

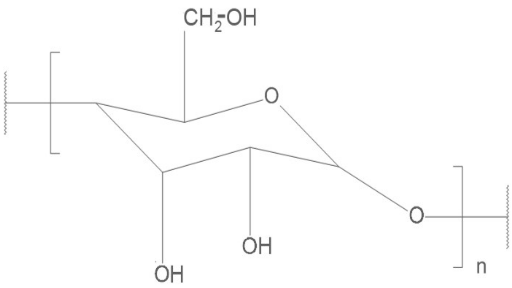


Fig.1. *BC chemical structure.*

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II. MATERIALS, PROTOTYPE AND METHODS

A. Materials

CBP-GS010 film of bacterial cellulose (BC) (wet average thickness = 395 μm) was purchased from BioFaber (Italy). Ionic liquid (IL) 1-Ethyl-3-Methylimidazolium tetrafluoroborate (EMIM-BF_4) was purchased from Alfa Aesar. Poly-(3,4-ethylene-dioxythiophene)-polystyrene-sulfonic acid (PEDOT) was purchased from H.C. Starck(1.3 wt% dispersion in water, Baytron P AG).

B. Prototype

The prototype has been conceived considering the transduction properties of an all organic composite, realized by using BC, CPs, and ILs. All organic composites were produced in two steps. As the first step BC pieces (5 cm x 5 cm) were cut and dried in an oven overnight to eliminate water (about 4%). The membranes were then dipped in EMIM-BF_4 for 24 h and were dried in a vacuum oven for 24 h at 65 °C. As the second step, BC/EMIMBF_4/PEDOT devices were fabricated by depositing four layers of a PEDOT-PSS water dispersion on both sides of the cellulose membranes via a film spreader (24 μm). After every deposition the membranes were dried in an oven for 5 min. This last procedure was used also to prepare BC/PEDOT samples also. The membranes where trimmed to eliminate short circuits between the electrodes and to obtain devices of the needed geometry. Fig.2 shows a produced sensor, which has the shape of a beam having a length of ~34.4 mm a width of about ~10.6 mm and a thickness of ~0.16 mm.

C. Characterization methods

The IL absorption percentage of BC was determined by using an analytical balance. Thermogravimetric analyses (TGA) have been carried out by a Shimadzu model DTG-60 instrument. TGA curves have been recorded at a heating rate of 10 °C min^{-1}, under static air atmosphere, from 35 °C to 700 °C. Analyzed sample mass varied between 8.0 mg and 11.0 mg. X-ray photoelectron spectroscopy (XPS) measurements were carried out on a VG Instrument electron spectrometer using a Mg $K_{\alpha1,2}$ X-ray source (1253.6 eV). The X-ray source in the standard conditions had been working at 200 W, 10 kV, and 20 mA. The base pressure of the instrument was $5x10^{-10}$ Torr and an operating pressure of $2x10^{-8}$ Torr was adopted. A pass energy of 100 eV and 50 eV was used for widescans and narrowscans, respectively. For acquiring the spectra a take-off angle of 80 ° was used. Binding energies were referred to the C-H level at 285 eV. A 2000 TA

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DMA produced by Triton Technology Ltd. was used to study the viscoelastic properties of the devices. The temperature dependence of the membrane modulus has been evaluated, in tension mode, by applying a sinusoidal force to a rectangular sample, in the range 25-200 °C at a 2 °C/min rate and working frequency of 1 Hz. Measurements of storage modulus (E'), loss modulus (E") and tan delta (tan δ = E"/E') were obtained.

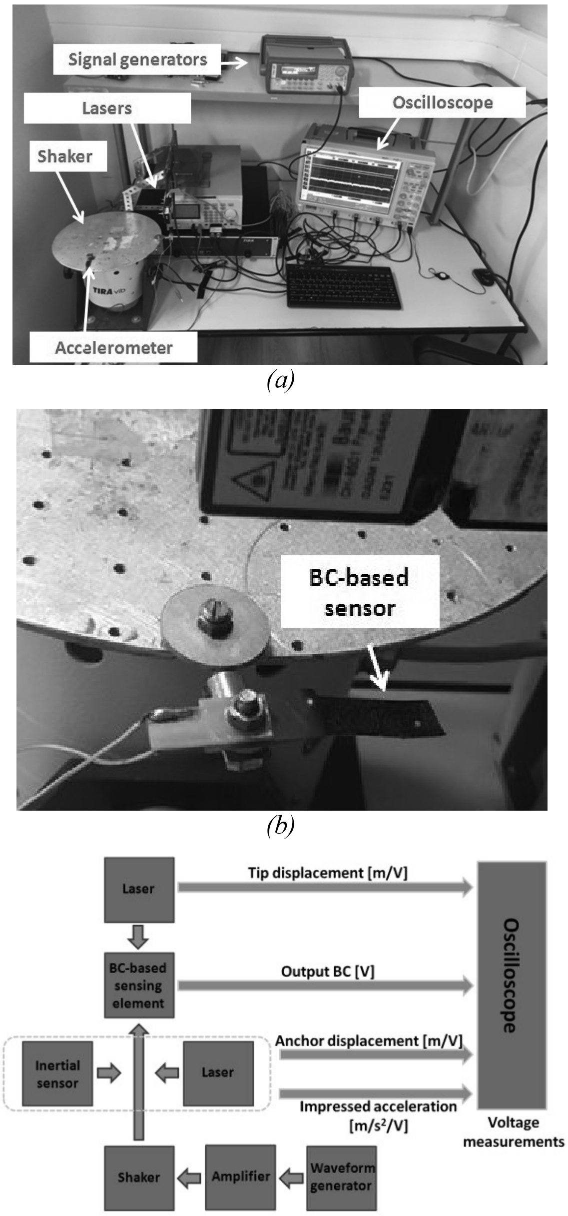
Fig.2. *Realized paper based sensor.*

Scanning Electron Microscopy (SEM) micrographs have been obtained using a Cambridge 90 instrument equipped with an energy dispersive X-ray microanalysis (EDX) facility.

In order to characterize the sensor for deformation measurements and to study the device in the voltage and frequency domain, a suitable experimental setup has been purposefully realized.

Fig.3 shows the architecture that is composed of:

- a shaker (TIRA TV 50009), to excite the structure.
- A signal generators HP33120A, to impress a suitable waveform to the shaker.
- A digital Teledyne LeCroyWaveRunner 6050, to acquire the signals.
- A single-axis accelerometer, used to monitor the imposed vibrations.
- Two laser sensors, used for the measurement of the sensor deformation.



(c)

Fig.3. *(a) Experimental setup, (b) zoom of the sensor and of the two lasers used to measure the deformation (anchor and tip displacement, respectively), (c) block diagram of the measurement setup.*

III. RESULTS AND DISCUSSION

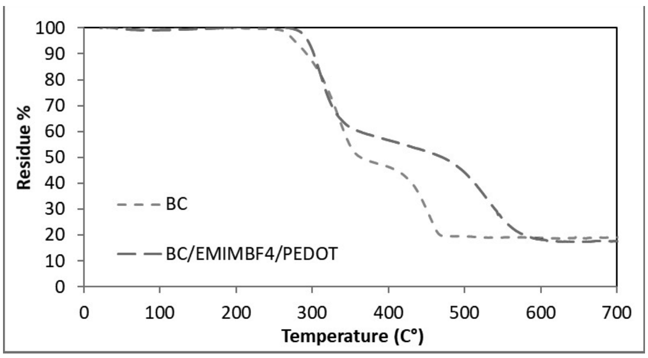
A. Performance characterization

This section is devoted to study the composite characteristics and the transduction performance of the BC based sensors. The amount of EMIM-BF_4 absorbed by BC during the first preparation step was about 34%. BC thermodegradation curve and its derivative (Fig.4) shows three different raw processes of thermal degradation, which are associated with maximum degradation rates at 284 °C, 336 °C and 446 °C, respectively. As reported in literature [31], these temperatures of maximum weight loss rate are related, respectively, to phenomena of

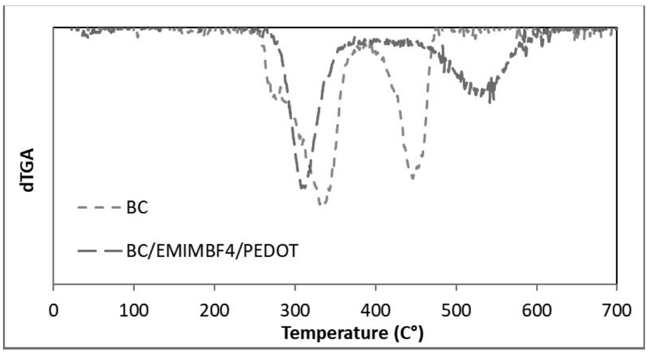
1) dehydration and fragmentation of polymer chains;

2) formation of anhydromonosaccharide and conversion into low molecular weight polysaccharides;
3) formation of charred degradation products.

The BC treatment with IL, followed by PEDOT/PSS electrode deposition, modifies the thermodegradation producing an apparent merging of 1 and 2 processes into a single stage with a maximum weight loss rate at 315 °C; while the maximum weight loss rate of the third process shifts to 531 °C.



(a)



(b)

Fig.4. *TGA (a) and dTGA (b) of BC, and BC/EMIMBF$_4$/PEDOT.*

XPS analysis (performed as described in Section II) of BC showed the presence on the surface of Carbon, Oxygen, Silicon and Sodium (TABLE II). C_{1s} and O_{1s} regions were in accord with the cellulose structure. The sodium presence is due to NaOH used by the manufacturer for whitening cellulose, while silicon is a contaminant deriving from sample container. The BC/EMIMBF_4 sample shows on the surface, beside to signals due to cellulose, fluorine and nitrogen due to IL. On the surface of the all organic composite (BC/EMIMBF_4/PEDOT) (Fig.5), beside to sulphur, present in the structure of PEDOT/PSS, there are fluorine and nitrogen thus showing that some IL migrates into the PEDOT outer layer.

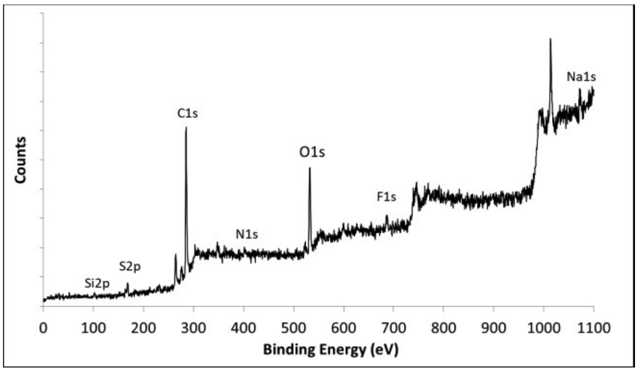


Fig.5. *XPS widescan of sample BC/EMIMBF$_4$/PEDOT.*

TABLE II. SURFACE ATOMIC ABUNDANCE AS OBTAINED BY XPS

Sample	C	O	Si	Na	F	N	S
BC	64	31	3	2	-	-	-
BC/EMIMBF$_4$	74	18	2	2	3	1	-
BC/PEDOT	68	20	3	2	-	-	6
BC/EMIM BF$_4$/PEDOT	73	19	2	1	2	2	5

Sample surface morphologies were examined using SEM. BC SEM micrograph (Fig.6a) showed an irregular arrangement of cellulose agglomerates. Sample BC/EMIMBF_4 showed that IL swelled cellulose and produced fiberlike structure (Fig.6b). The deposition of the PEDOT layer produced a regular surface because of PEDOT absorption and filling of the empty spaces among BC agglomerates (Fig.6c). The micrograph of the surface of the all organic composite (BC/EMIMBF_4/PEDOT) showed a smooth morphology testifying an homogeneous deposition of the conducting layer with island features presumably rich of IL as revealed by XPS(Fig.6d).

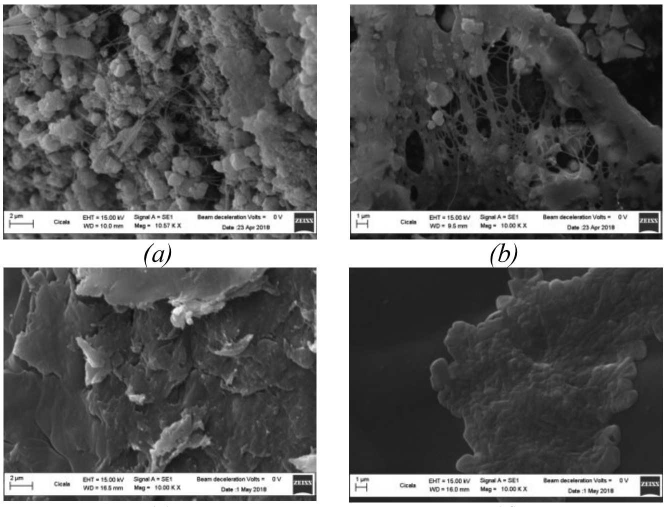


Fig.6. *SEM micrographs of (a) BC, (b) BC/EMIMBF$_4$, (c) BC/PEDOT and (d) BC/EMIMBF$_4$/PEDOT samples.*

The results of DMA studies carried out on BC, BC/EMIMBF$_4$, BC/PEDOT and BC/EMIMBF$_4$/PEDOT samples are reported in Fig.7 and Table III. The storage modulus of BC shows a negligible decrease as the temperature increases and tan δ remains almost constant to a low value (~0.12) that testifies of the quasi elastic character of the sample. The soaking of BC with EMIM BF$_4$, because of the swelling/degradation of crystalline domains of BC, determines a dramatic drop of storage modulus with respect to BC one, and the higher value of tan δ at 30 °C confirms the higher liquid-like behaviour of the sample. The PEDOT electrode deposition doesn't significantly modify the storage modulus at 30 °C with respect to the one of BC ($4.0x\ 10^9$ Pa with respect to $5.5x10^9$ Pa), but dramatically increases the storage modulus at 30 °C with respect to the one of BC/EMIMBF$_4$sample ($5.4x10^7$ Pa).

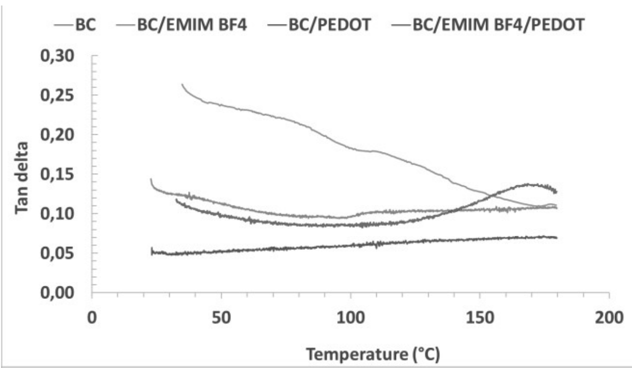


Fig.7. *Tan δ obtained through DMA measurements of BC, BC/EMIMBF$_4$, BC/PEDOT and BC/EMIMBF$_4$/PEDOT samples respectively.*

TABLE III. STORAGE MODULUS AND TAN δ OF SAMPLES DETERMINED BY DMA

Sample	Storage modulus (Pa)		Tan δ	
	30°C	170°C	30°C	170°C
BC	5.5 x 10^9	5.3 x 10^9	0.12	0.11
BC/$EMIMBF_4$	5.4 x 10^7	3.9 x 10^7	0.29	0.11
BC/PEDOT	4.0 x 10^9	3.0 x 10^9	0.049	0.071
BC/$EMIMBF_4$/PEDOT	3.3 x 10^9	2.7 x 10^9	0.12	0.14

In order to investigate the output signal, produced by the paper-based sensor for deformation measurements, several mechanical excitations have been applied and various analyses have been performed. In particular, Fig.8 shows signals used for estimating the deformation of the beam. Two laser sensors have been used for measuring the anchor and tip displacements, respectively. The beam deformation has been estimated as the difference between the tip and the anchor position. A step waveform has been used as the electrical input signal for driving the shaker, by using a frequency of 2 Hz. It should be noted that, as consequence of each step, the sensor output is linked to the its natural frequency response, which corresponds to about 21 Hz. Fig.9 presents the output as a function of the displacement at the anchor in presence of a mechanical step. As it can be noted for an increment of the step amplitude, a higher output voltage can be observed.

Fig.10 shows the frequency response of the device, obtained through a sinusoidal excitation having a frequency in the range 13-29 Hz. For each value, the output voltage has been measured by using the digital Teledyne oscilloscope (see Section II). A maximum peak appears in correspondence of the mechanical resonant frequency of the device, which is very close to the natural frequency [32,33]. The characterization of the sensor has been performed at the resonant frequency. The amplitude of the input sinusoidal signal has been varied and the BC-based sensor deformation has been estimated, according to the laser sensors outputs.

Fig.11 shows the output voltage of the device for several values of the sensor deformation. The diagram includes the mean value and the corresponding uncertainty. A sensitivity of about $4.3*10^{-5}$ V/mm has been estimated with a resolution of about 0.04 mm.

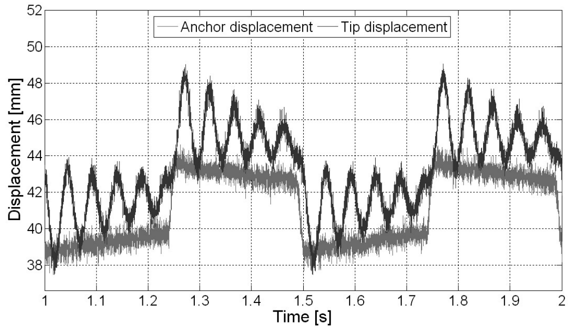


Fig.8. *Displacements at the anchor and at the tip of the sensor measured through the two lasers.*

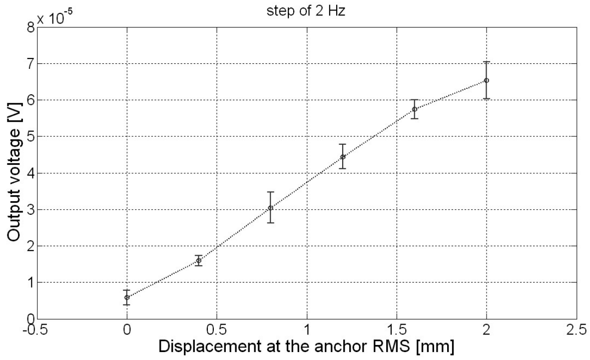


Fig.9. *Output voltage of the sensor as function of the displacement at the anchor in presence of a step signal of 2 Hz.*

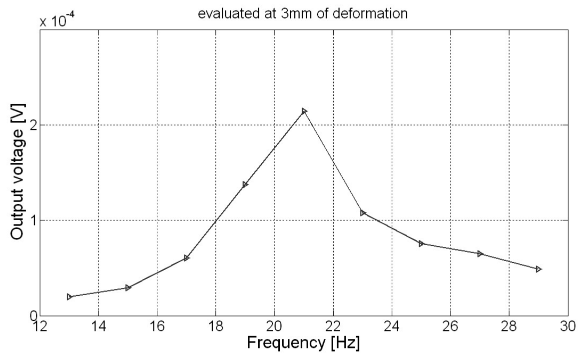


Fig.10. *Output voltage of the sensor as function of the frequency in presence of a sinusoidal mechanical excitation.*

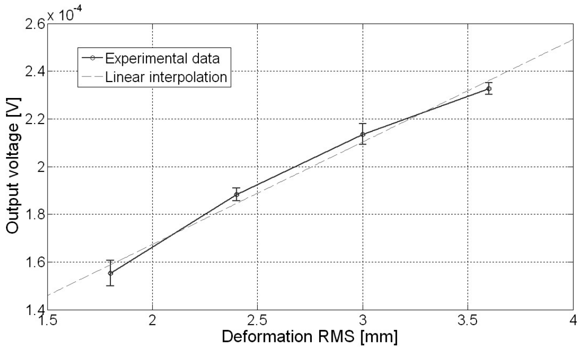


Fig.11. *Output voltage of the sensor evaluated in presence of a sinusoidal vibration at 21 Hz as function of the measurand.*

In order to further investigate on the transduction performance of the BC based device, including nonlinearities and generated voltage as function of the measurand, an investigation of the input-output signals at the resonance frequency was accomplished.

For this analysis, in order to filter the effect of the noise, a pre-filtering was performed on both signals (input and output waveforms). The filter used is a digital low-pass filter having a cut-off

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frequency able to remove the power-line contribution and higher frequency noisy contributions. Various signals having different amplitudes were analyzed. Fig.12 shows the input-output relationship for four different maximum values of the sensor deformation.

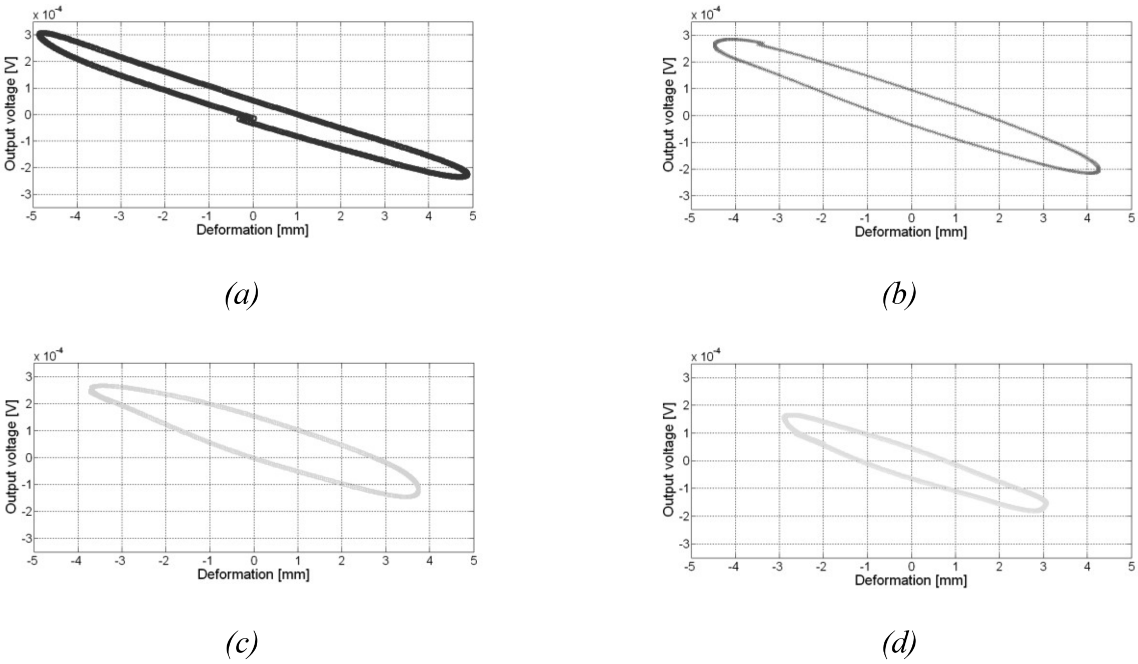

Fig.12. *Output voltage of the BC as function of several amplitudes of deformation.*

As it can be observed the increase of the input deformation does not produce a proportional change in the output voltage generated through the BC. This can be considered as indicative of a nonlinear behaviour of the compound. This result in accordance with Fig.11 where a linearization was accomplished in the considered range of the measurand. The jitter of the output waveform can be observed in Fig.13, where the input-output curve for a number of nominally identical periods is presented.

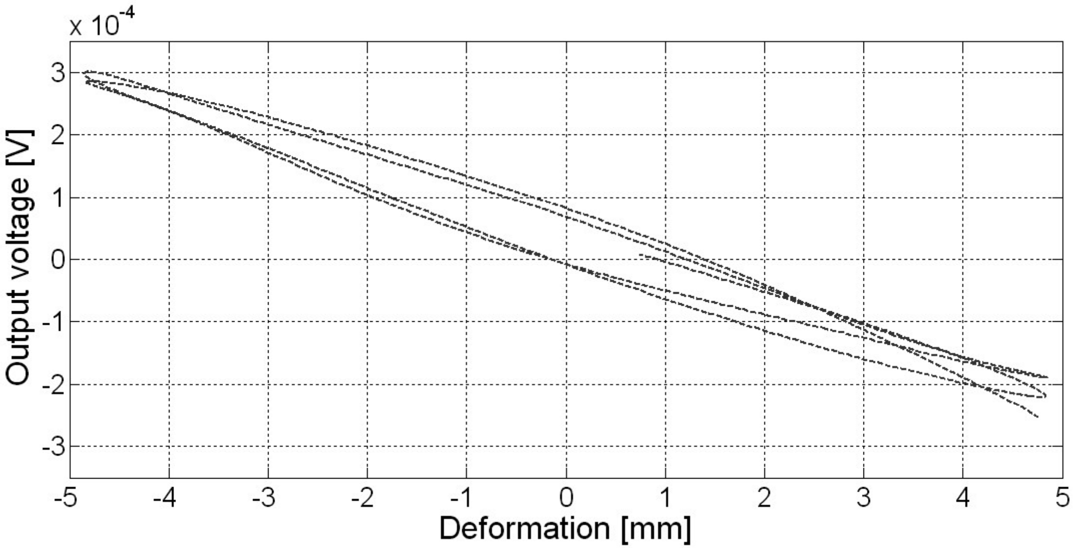

Fig.13. *Output voltage of the BC as function of a deformation having an amplitude peak-to-pek of about 9 mm. The graph includes more periods of the input source.*

B. Investigation of the transduction nature and effects of the ILs

This section is devoted to investigating the transduction nature the of the proposed BC based device and in particular the effect of the ILs in the transduction mechanism.

In fact, BC compound, impregnated with ILs, and electrically connected with electrodes have been reported to be capable of electro-mechanical and mechano-electrical transduction features, because of “piezo-ionic” properties correlated with the migration of charged species. For these reasons the proposed BC based composite should be classified as a ionic-electroactive polymer (IEAP) [23,34].

In order to perform experiments on the BC based compound (with and without ILs) both as a sensor and as an actuator a suitable setup has been implemented, as shown in Fig.14. It is composed of a shaker to study the compound as sensing element, two lasers in order to measure the distance at the anchor and at the end of the beam. In order to impress low frequency signals, a fork shaped hinge to force the movement of the composite has been also considered during the characterization as sensor.

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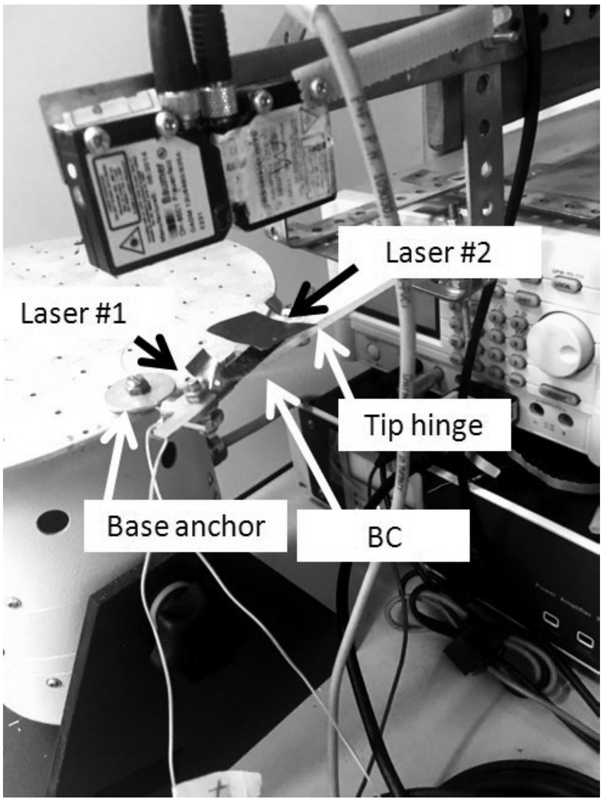
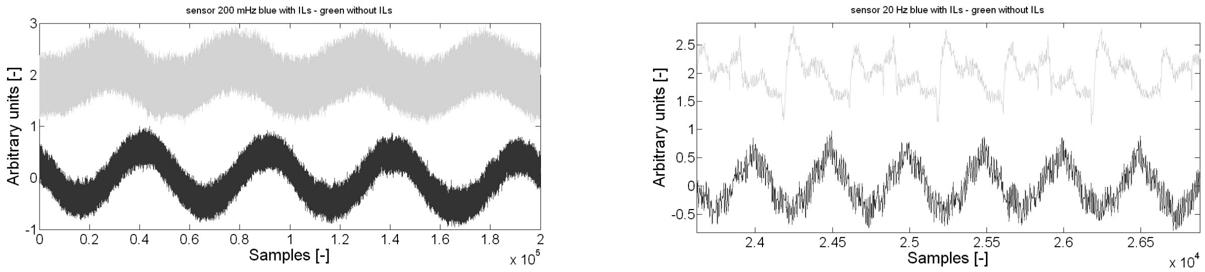


Fig.14. Setup used for the investigation on the mechanoelectrical transduction in presence and in absence of ILs.

Fig.15 shows the investigation of the BC as sensor in presence and in absence of ILs. In particular, Fig.15a shows the output waveform at low frequency (200 mHz). As it can be observed in presence of ILs the output signal presents less noise as respect the BC without ILs. This is coherent with literature which evinces that the ILs improve the performance of IEAP [34]. At higher frequency (see Fig.15b), this effect decreases.



(a) *(b)*

Fig.15. Comparison of signals obtained when the device is used as a sensor (a) at 200 mHz (b) at 20 Hz. The blue graps are in presence of ILs, the green in absence of ILs.

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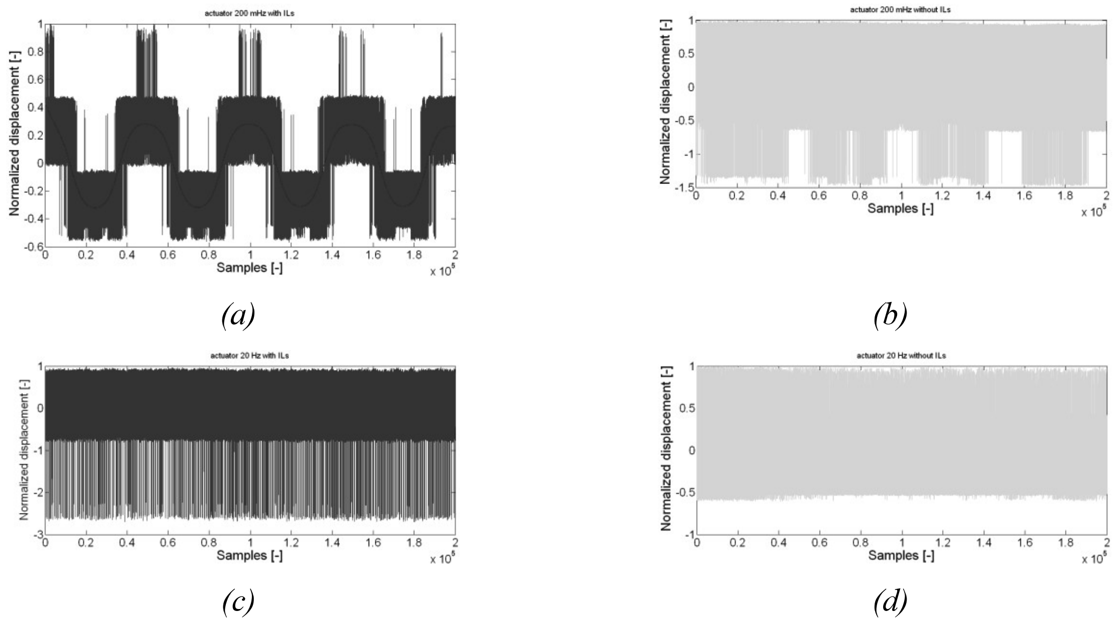

Fig.16. Comparison of signals obtained when the device is used as a actuator (a) at 200 mHz with ILs (b) at 200 mHz without ILs, and (c) at 20 Hz in presence of ILs, (d) at 20 Hz in absence of impregnated ILs.

The effect of the ILs has been also studied focusing the attention on the actuation features of the compound. Fig.16 shows a comparison of the signal applying a sinusoidal waveform across the BC at 200 mHz and 20 Hz. The output has been measured through the lasers (see Fig.14). Though signals are affected by the resolution limits, imposes by the used laser sensors. Fig.16a shows that at low frequency (200 mHz) the ILs improve the actuation performance of the transducer with respect to the device without ILs (see Fig.16b). Furthermore at higher frequency the effect of the ILs is very weak as confirmed through Fig.16c and Fig.16d.

IV. CONCLUSIONS

In this paper the implementation and characterization of a paper based sensor for deformation measurements have been pursued. The conceived sensor is based on a nanocomposite consisting of BC, impregnated with ILs and covered with a CP. The structure is mounted in cantilever configuration and its flexural behaviour is studied. The beam deforms as a function of the external mechanical source and it is able to generate a voltage level, dependent on the measurand. Both the frequency behaviour and the step response of the system have been studied. It is worth noting that the proposed solution is the first demonstrations of the generating sensing properties of this class of compounds, which can be used for the realization of sensors having the prerogative of being flexible, ecofriendly, and low-cost. A sensitivity of about $4.3*10^{-5}$ V/mm at the frequency of 21 Hz has been estimated with a resolution of about 0.04 mm. Evidence of the possible contribution of the ILs in the performance of the transducer is, also, given.

Further work is needed both at a "material" and a "sensor" level to improve the performance of the sensor. Reported results show that the considered compound can contribute to the realization of the next generation of sensors, suitable to the implementation of smart sensing systems.

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