Design and development of a piezo-resistive sensor based on PEDOT: PSS applied to Sisal's natural fiber for monitoring of 3D warp Interlock fabric

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Abstract. The aim of the current study is to develop and optimize a flexible sensor based on a natural sisal fiber. Based on this sensor yarn, different fibrous reinforcements of 3D warp interlock fabrics used in composite materials could be monitored. This natural yarn (Sisal) has been pre-coated with a layer of PVA solution which makes it possible to homogenize the surface of the yarn and serves as a substrate for the coating phase. A copper metal wire was used as a connecting device to the measurement acquisition system. Some Conductive Polymers Composite (CPC) based on PEDOT: PSS are compared and used as a coating layer in order to get the conductive appearance of the sensor yarn. A sensor calibration step was carried out which consists in testing the sensor yarn on a MTS traction bench while measuring their resistance variation simultaneously. The electromechanical behavior of the different types of sensors will be compared and discussed. Insertion of these developed sensors within 3D woven structures will be helpful for local monitoring and check the inner local elongation of the 3D warp interlock fabric during tensile tests.

Keywords: Mechanical properties; Piezo-resistive sensor; PEDOT: PSS; Sensor calibration; Weaving process; 3D warp Interlock fabric

1. Introduction

Today, metal and petrochemical materials have been decreased in the industrial sectors, especially those related to transportation applications. However, scientists begun looking for new materials, such as composite materials. This will allow concerned companies to reduce mainly the total cost and weight of their mobile structures parts. In addition, this will enable manufacturers to meet current climate and environmental challenges.

Nonetheless, there are some still barriers that restrict the use of these composite materials due to the ageing, fatigue behavior and their failure mode. This lack of knowledge can reduce and limit their use in some areas, where high performance is required. Then, to get out of these limits, composite materials must be more investigated with additional research works, specifically on the fibrous reinforcement. This type of fibrous reinforcement of such composites is often a woven structure. However, few studies have been conducted to investigate the influence of the weaving process (2D or 3D) for the final
architecture of textile composite. Since 2003, Kuo et al reported a study on failure behavior of 3D woven composites under transverse shear, by measuring the influence of the weaving process (3D) on the properties of the final fabric [1].

Recently, some researchers have highlighted defects caused by the degradation of yarns during the weaving process [2-4]. It was found that the warp yarns suffered a series of damage occurring at most stages of the 3D weaving process, due to the abrasion between the warp yarns and the dynamically moving parts of the loom machinery.

Furthermore, various researches have studied local and in-situ mechanical behavior of fibrous reinforcements used in composite materials [5]. However, few researches have used sensor yarns in textile structures as fibrous reinforcement of composite materials to understand and control their behavior during tensile stresses. The integration of these sensor yarns into the 3D woven structures have allowed also to measure the local and in-situ behavior of one of these yarns submitted to mechanical stresses [6]. This approach could make it possible to identify the various parameters influencing on the mechanical properties of the fibrous reinforcement during static and dynamic stresses.

Physical and mechanical characteristics of natural fibers are very important. Particularly, comparative studies have been done on composites based on natural and synthetic fibers [7-9]. In addition, some natural fibers such as sisal provide better features and performances than other natural fibers. Sisal yarns showed good adhesion and good physical and mechanical properties when is used in reinforcements for composite applications [10].

Therefore, the goal of this study is to develop sisal yarns collected from Morocco and to investigate how to use them both as a fibrous reinforcement of a textile structure and also as a sensor yarn within these structures, and consequently to control their behavior in-situ during mechanical and tensile stresses.

2. Material and Methods

2.1. Preparation of sisal yarn

The sisal yarns used have an average diameter of 2.6 mm, a linear density of 3200 Tex and twisted at 80 t/m. The natural yarn was put into the Soxhlet glassworks with petroleum ether as a solvent for 3 hours. This cleaning would lead to a better contact of the interface between the conductive layer particles and the sisal yarn.

2.2. Mechanical measurement of sisal yarn

After the cleaning of sisal yarns with the soxhlet extractor, the mechanical properties of sisal yarns were investigated using uniaxial tensile tests with five sisal coupon yarns. This investigation of the mechanical behavior of the sisal yarns was important as a substrate of our sensor. It was performed by using a servo-electric MTS 2/M [11-12] corresponding to the standard: ISO 2062:2009. The results of the load–strain curve are shown in Table 1 and Figure 1. The standard test parameters were:

- initial distance between clamps (Li): 200 mm
- preload: 2 N
- speed of the tensile test: 200 mm/min
- number of cycle: 1

| Yarn   | Load at maximum (N) | Strain at break (%) |
|--------|---------------------|---------------------|
| Sisal 1 | 645.312             | 9.148               |
| Sisal 2 | 551.838             | 7.739               |
| Sisal 3 | 700.245             | 9.239               |
| Sisal 4 | 702.419             | 8.753               |
| Sisal 5 | 564.209             | 7.721               |
AVERAGE 632.805 8.52

Figure 1. Tensile strength curves of sisal yarns.

2.3. Design and Production of sensor yarns
The main elements of the developed sensor yarns are:
- Sisal yarn
- Piezo-resistive coating.
- Connecting wires

Figure 2 shows a general structure of the sensor wire based on sisal yarn (1). This later was pre-coated with a PVA solution (2) to protect the sensor from abrasion. Then, it was ligated by copper connector wires (3), which was used to measure the electrical resistance of the sensor during its elongation on the tensile bench. The distance between the connecting wires has been chosen to guarantee accurate and precise sensibility of measurement. At the end, layers of piezo-resistive coatings (4) have been deposited on the surface of the sensor yarns to increase its conductivity.

Figure 2. Schematic of the sensor yarn.

3. Results and discussions
3.1. Characterizations of sensor yarns
3.1.1. Mechanical test of sensor yarns. Mechanical behavior of sisal yarns (five yarns in an average) was carried out on the MTS bench, as it was reported in section 2.2, in order to measure their mechanical properties and to show their elastic behavior. The sisal yarn tends to behave as a quasi-elastic material. This mechanical behavior was needed to ease the sensor characterization. Yarns sensor were prepared (refer to section 2.3) and tested on their tensile strength at breaking referring the same test procedure as it was reported in section 2.2. It is based on determine the mechanical behavior of three sensors using sisal yarns. The first was designed without PVA solution (0% PVA). The other two sensors were coated with 3% and 6% PVA layers respectively.
The obtain results from these tests show that the strain at break of the sensor yarns was increased to 10.531%, 10.450% and 10.455% respectively for sensor without PVA, 3% PVA and 6% PVA. The elongation at break was significantly higher on the coating sensors, compared to the sisal yarns (8.520% at max) (see Table 2). Therefore, all the prepared sensor yarns have a solid elastic increased behavior, thanks to the increases rates of PVA. In terms of load, all the sensor yarns has an average value between 753.020 to 836.366 N, whereas the sisal yarns have an average load of 632.805 N (see Table 3). Therefore, the PVA solution could enhance the mechanical properties of the coating. These properties were expected as they had been previously cited in the work of Chen et al. [13].

### Table 2. Strain at break (%) values for sensor yarns.

|               | Strain at break (%) | Standard deviation (%) |
|---------------|---------------------|------------------------|
| Sisal         | 8.520               | 0.743                  |
| Sensor yarns without PVA | 10.531             | 0.772                  |
| Sensor yarns with 3% PVA   | 10.450             | 0.638                  |
| Sensor yarns with 6% PVA   | 10.455             | 0.521                  |

### Table 3. Maximum load (N) values for sensor yarns.

|               | Load at maximum (N) | Standard deviation (N) |
|---------------|---------------------|------------------------|
| Sisal         | 632.805             | 72.131                 |
| Sensor yarns without PVA | 753.020             | 53.538                 |
| Sensor yarns with 3% PVA   | 836.366             | 127.038                |
| Sensor yarns with 6% PVA   | 779.631             | 65.908                 |

#### 3.1.2. Calibration of sensor yarns.

The sensor calibration consists in measuring the electrical response by the sensors simultaneously with the tensile bench measurements. In this case, the MTS bench was linked together with a data acquisition system (National Instruments USB-6003) and DAQExpress Processing Software (see Figure 3).

![Figure 3. Calibration test of sensor wires on traction bench.](image)

On the other hand, the electrical resistance of the yarns sensors has also been recorded during tensile breaking tests. The electrical measurements were carried out by means of a divider bridge type electric assembly to determine the voltage at the sensor wire terminals which is considered as resistance $R_2$ according to the following relationship (see Figure 4).
Figure 4. Electrical scheme for measuring the resistance of sensor yarns.

\[ R_2 = R_1 \times \frac{V_s}{V_e - V_s} \quad (1) \]

Where the resistance \( R_1 \) is a decade box serves to balance the divider bridge to choose a value close to \( R_2 \) when the wire is not stressed. An input voltage \( V_e \) has been applied to this arrangement. Subsequently, the determination of sensor’s resistance \( R_2 \), from the output voltage \( V_s \) delivered by the acquisition system, is performed by applying the Ohm's law across the resistance \( R_2 \) using the equation [Equation 1].

The evaluation of the sensor’s sensibility can be determined by the gauge factor (k). This factor corresponds to the ratio of the relative variation of resistance compared to the initial resistance \( R_0 \) divided by the measured elongation (\( \epsilon \)) using the [Equation 2]:

\[ k = \frac{\Delta R}{R_0 \epsilon} \quad (2) \]

Where:

\[ \frac{\Delta R}{R_0} = \frac{R_1 - R_0}{R_0} \quad \text{and} \quad \epsilon = \frac{l_i - l_0}{l_0} \quad (3) \]

\( R_0 \) and \( R_i \) respectively, represent the initial resistance of the sensor yarn and the electrical resistance value measured at the instance of measurement. \( l_0 \) is the initial length of the sensor yarns and \( l_i \) is the length value measured of the sensor yarns at the instance of measurement [Equation 3].

The relative difference of resistance measurements was synchronized with the tensile force measurements applied to the three sensor yarns (without PVA, 3% and 6% PVA), carried out by a MTS tensile bench at fixed speed (200 mm/min).

The gauge factor increases and changes greatly with the elongation of the sensitive coating. However, for our application, strain variation does not exceed 8%. For these series of prepared and tested sensor yarns, the gauge factor (k) was defined and determined. It is observed that a sensor yarn with 6% PVA has a better factor gauge \( k \approx 1.31 \) compared to the sensor yarns without PVA and with 3% PVA, respectively 0.65 and 0.79. In addition, this factor increased with the ratio of 6% PVA solution. The mixture of the PVA solution and the Pedot:Pss could give a better uniformity of the thickness, and then could provide a better compromise between elongation detection sensitivity and electromagnetic noise resistance. This behavior could indicate that resistivity of the coating was changed under tough strain and not only due to a change of geometry.

4. Conclusion
This work consists in the development of a new type of mechanical sensor based on natural fiber. These sensor yarns will be able to monitor and record in situ strains and stresses on sisal yarns, commonly used as natural fibrous for composite application. The work has furthered previous studies undertaken at our institute and has been combined with new technologies to meet the specific needs. A new design to prepare these sensor yarns has been achieved. As a first step, the sisal yarns were cleaned with a
Soxhlet extractor and characterized. Thus, the sisal yarns were pre-coated with a PVA solution used as protection against abrasion. Then, sisal yarns were ligatured by copper connector wires, in order to connect the electrical measurement devices and piezo-resistive coating of the sensor yarns. After that, sisal yarns, pre-coated with a PVA and ligatured by copper wires, were also coated by an aqueous solution of Pedot:Pss. In the second step, three sensors yarns were prepared; the first one without PVA and the two others respectively with 3% and 6% of PVA. These series of sensor yarns have been characterized on a MTS bench linked together with a data acquisition system (National Instruments USB-6003) and DAQExpress Processing Software. The mechanical and electrical behavior were investigated. The sensor yarns with 6% PVA revealed a good factor gauges k greater than 1. In summary, a successful sensor formulation has been established and now it could be ready for forthcoming in-situ measurements during the weaving process.

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