

## Electric Elegance: Enhancing Cotton Fabric with Conducting Polypyrrole for Gas Sensing Applications

Vinod S. More\*, B.K. Sakhare†, R.P. Tandel‡, G.G. Padhye♦ and T.N. Ghorude\*\*

### Abstract

Nitrogen dioxide ( $\text{NO}_2$ ) is part of the nitrogen oxides ( $\text{NO}_x$ ) group, which comprises air pollutants generated by combustion activities. The primary sources of  $\text{NO}_2$  are traffic, unvented heaters, and gas stoves among others. This compound poses significant risks to human health and the environment. Moreover, it plays a crucial role in the creation of Acid Rain. The current work is carried out to study the indigenously developed conducting polymer-based gas sensor for  $\text{NO}_2$  detection. The gas sensor fabricated using conducting polymer such as Polypyrrole (PPy) as the active layer on the cotton fabric surface. The effects of washing on the composition and structure of samples were investigated through the application of XRD and FTIR spectroscopy. XRD analysis revealed notable changes in crystal structure. FTIR analysis provided insights into the molecular bonds present, highlighting variations in the functional groups before and after washing. We evaluated the electrical conductivities of

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\* Department of Physics, N. B. Mehta Science College, Bordi, Dahanu, Maharashtra, India; [vinod13570@gmail.com](mailto:vinod13570@gmail.com)

† Department of Physics, Sonopant Dandekar College, Palghar, Maharashtra, India; [bhimraos68@gmail.com](mailto:bhimraos68@gmail.com)

‡ Department of Physics, Sonopant Dandekar College, Palghar, Maharashtra, India; [rajutandel92@gmail.com](mailto:rajutandel92@gmail.com)

♦ Department of Physics, Thakur College of Science and Commerce, Kandivali, Mumbai, Maharashtra, India; [drgiteshpadhye@gmail.com](mailto:drgiteshpadhye@gmail.com)

\*\* Department of Physics, N. B. Mehta Science College, Bordi, Dahanu, Maharashtra, India; [tnghorude@gmail.com](mailto:tnghorude@gmail.com)

samples before and after washing. The key findings shed light on the effects of washing on the chemical structure of gas sensing materials, which is critical for maintaining sensor performance. This paper discusses the gas sensing mechanism and configuration of the sensor. Its threshold limit value (TLV) is 25 ppm and hence detection at low ppm concentration has become the focus of research.

**Keywords:** Gas sensor, Conducting Polymer, NO<sub>2</sub> gas, Polypyrrole, TLV

## 1. Introduction

Over the past few years, there has been a growing interest in the development of fabrics with novel qualities. Smart conductive fabrics have emerged as a promising domain within the realm of smart textiles, captivating researchers, engineers, and designers alike. These fabrics possess the remarkable ability to interact with their surrounding environments or the user, opening up exciting possibilities for applications in healthcare, sports, automotive and aviation [1]. The rapid development of smart materials has paved the way for revolutionary advancements in various industries. Smart conducting fabrics, characterized by their ability to detect and respond to ambient circumstances or stimuli, have garnered significant attention. Smart conductive fabrics can conduct electricity which enhances its value [2]. Furthermore, smart fabrics are frequently required to work, and their quality influences the robustness, washability, reusability, and durability of fabrics. These are fabrics that interact with the surrounding environment or the user [3].

Conductive polymers are organic polymers with the ability to conduct electricity and expressing conductive or semi-conductive characteristics[4]. They have played a major role in the production of smart textiles [5]. Currently, there are over 25 known conductive polymers. They form a fascinating family of materials that combine some of the electrical characteristics of metals with some of the mechanical characteristics of plastics [6]. These polymers have gained popularity as conductive materials due to their low weight and low cost, relatively high adjustable electrical conductivity due to doping process [7] with flexibility, biocompatibility, ability to be specific to have a sensing and actuating operation, and ease of preparation [8].

The enhanced conductivity of polymer-based conductive fabrics led to their widespread recognition [9]. One of the excellent polymer, Polypyrrole (PPy), has significant role in the preparation of the conductive fabrics [10]. PPy is the optimum choice for production of conductive fabric due to their unique properties [14]. PPy has not been effectively explored until Kim *et al.* adopted In Situ Chemical Polymerization to generate the viscoelastic characteristics of PPy [15]. Many scientists are now focusing on polymerizing PPy on fabric substrates [11], cotton fabrics stand out among other textiles due to its softness and moisture absorbing ability [12].

There are numerous applications for conductive fabrics, including biomedical materials [13], flexible supercapacitors [14], strain sensors [5], and electromagnetic shielding [15]. Gas detection [16] and concentration monitoring have become more vital in order to safeguard against human exposure to harmful gases such as NO<sub>2</sub> [7], both at the home and industries. In conclusion, NO<sub>2</sub> is a hazardous gas with detrimental effects on both human health and the environment. Efforts to reduce NO<sub>2</sub> emissions and improve air quality are crucial for protecting public health and preserving the well-being of our planet [16]. Researchers are currently focusing on detecting low parts per million (ppm) concentrations of nitrogen dioxide due to its threshold limit value (TLV) being set at 25 ppm.

This paper discusses the preparation of electrically conductive fabrics by depositing PPy onto cotton fabric. The influence of reaction conditions on the properties of the resulting fabric was investigated systematically. The characterization of PPy-coated fabric was performed using Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and conductivity measurements evaluated using the two-probe method. In addition, the sensitivity of a gas sensor based on smart conductive cotton fabric to NO<sub>2</sub> was assessed.

## **2. Materials and Methods:**

### **2.1. Materials:**

The material being used was a simple cotton cloth (Bleached Cotton Print Cloth) acquired from Bombay Rayon Fashions Ltd., Mumbai. The Pyrrole (Py) purchased from Spectrochem (India) Pvt. Ltd.

Also, Iron (III) Chloride ( $\text{FeCl}_3$ ) purchased from Loba Chemie. Ltd. of research grade were used. Deionized water (DI) obtained from a Solar-powered deionization system was utilized for the sample preparation process.

## 2.2. Synthesis of conductive fabric by polymerization technique:

Commercial cotton fabrics were cleaned and dried before usage to get rid of any foreign contaminants (such as starch, oils, waxes, etc.). Py had undergone chemical polymerization to develop fabric samples [17]. We accomplished this process by adopting Py as the monomer and  $\text{FeCl}_3$  as the oxidant in a 1:1 monomer to oxidant ratio[18]. The reaction of PPy formation from Py monomer is illustrated in Figure 1. In summary, 10 ml of Iron Chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ) were added dropwise to 1 ml of aqueous 1M Pyrrole solution. By using a magnetic stirrer, the mixture was constantly stirred for four hours at 5 °C. After the completion of reaction, we dipped a cotton fabric in the solution and subjected to additional swirling for five hours at a low RPM. The fabric was then left in the solution overnight. On the following day, the fabric was stirred for 7-8 hours, removed from the solution and subsequently dried. At this, Initial measurements were taken before washing the sample. Afterwards, the fabrics were washed with DI water and dried once again. Final measurements were recorded after this step. The schematic for sample preparation was illustrated in Figure 2.

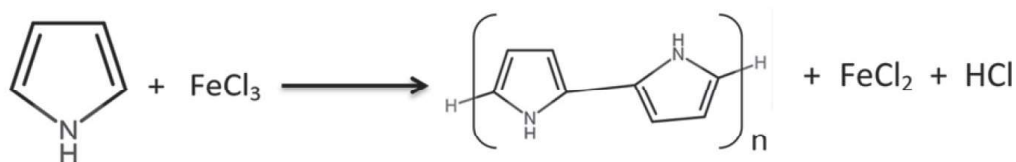


Figure 1: Reaction of formation of polypyrrole from pyrrole

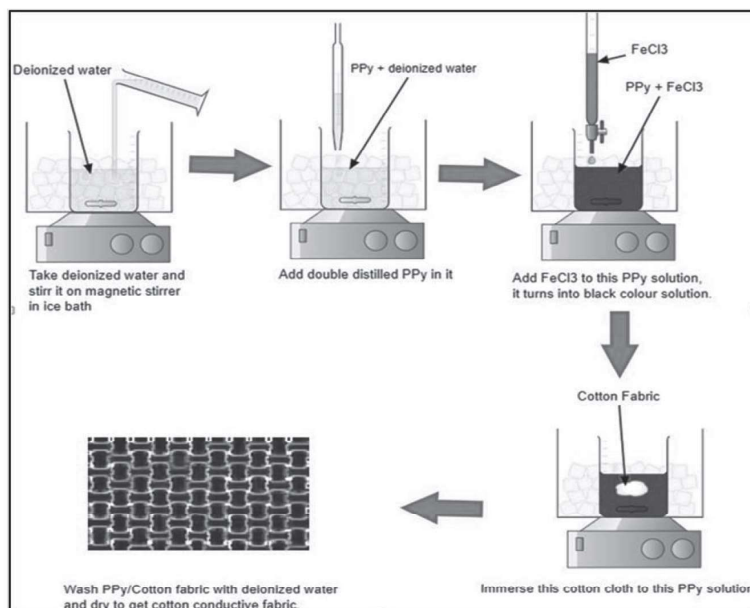


Figure 2: Schematic diagram for the sample preparation

### 2.3. Characterization and Conductivity measurements:

The samples underwent numerous examinations as part of the study. infrared (IR) spectra were obtained using the Attenuated Total Reflectance (ATR) technique on a Bruker ALPHA II FTIR Spectrometer. Both before and after washing, X-ray diffraction (XRD) patterns were analysed in the  $2\theta$  range of 3-90. A four-point probe resistivity device with copper electrodes was used to measure surface resistance under a 5N pressure and two probe method utilised in order to evaluate the dc electrical conductivity. These measurements provided information on the characteristics of the samples both before and after they were washed with DI water.

### 3. Results and Discussions:

PPy polymerization resulted in the production of black cotton fabrics; establish that the polymer completely encapsulated the fabric surface. The actual image of the PPy/Cotton fabric sample is shown in Figure 3. To confirm the polymerization of fabric, various characterizations had done.



Figure 3: Actual image of PPy/Cotton fabrics

### 3.1. FTIR analysis:

Figure 4 shows the FTIR curves of unprocessed normal cotton fabric, PPy/cotton fabric before and after washing.

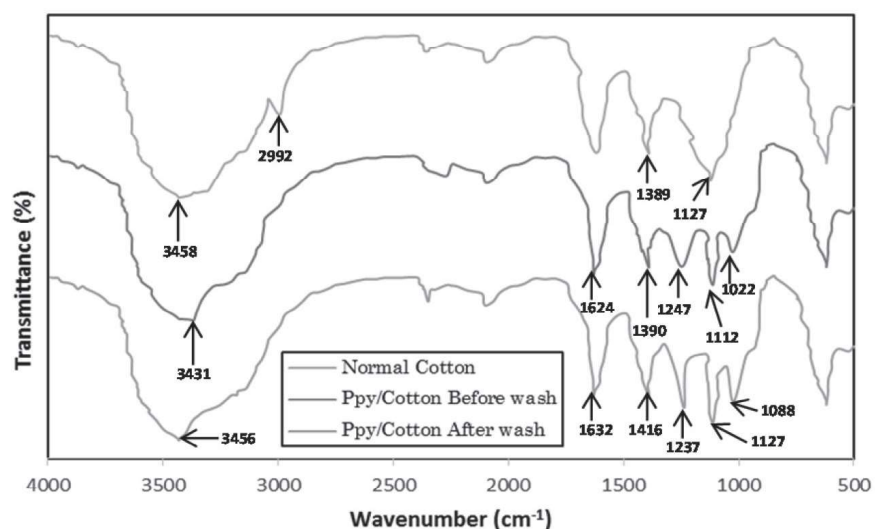


Figure 4: Infrared spectrum for normal cotton and PPy/Cotton fabrics

The FTIR curves of pure cotton have a peak at  $3458\text{ cm}^{-1}$  suggests O-H stretching vibration and peak at  $2992\text{ cm}^{-1}$  is evidence of C-H stretching vibration [19]. The untreated cotton's IR characteristic absorption peaks simultaneously emerged at  $1389$  and  $1127\text{ cm}^{-1}$ . However, at  $2820\text{--}1738\text{ cm}^{-1}$ , the peak values of the PPy/cotton composites are higher than those of cotton. The increasing water-bound polymerization process is the reason of this. Confirming the polymerization of PPy onto

cotton fibers are the group traits displayed in this figure. Before washing the PPy coated sample, the characteristic absorption peaks were seen at 1624, 1390, 1247, 1112, and 1022  $\text{cm}^{-1}$ . Following sample washing, the following absorption peaks were found at 1632, 1416, 1237, 1127, and 1088  $\text{cm}^{-1}$ . Since deionized water removes impurities and grime from the surface, the cotton sample's peaks were easily distinguished. The fundamental vibrations of the polypyrrole ring are responsible for the bands at 1632 and 1624  $\text{cm}^{-1}$  [20], the C-H in-plane vibrations are responsible for the bands at 1247 and 1237  $\text{cm}^{-1}$ , and the C-N stretching vibrations are responsible for the band at 1127  $\text{cm}^{-1}$  [18]. The C-O stretching vibration corresponds to the intense peak vibrations observed at 1022  $\text{cm}^{-1}$ .

### 3.2. XRD analysis:

Figure 5, 6 and 7 depicts the XRD patterns of normal cotton and PPy/cotton fabrics before and after washing. The diffraction of normal unprocessed cotton fabric exhibits characteristic peaks at  $23.7^\circ$  and  $27.4^\circ$ . These are the characteristic peaks of the crystal polymorph I of cellulose [19]. These peaks were appeared for fibers with higher cellulose content, such as cotton or flax [21].

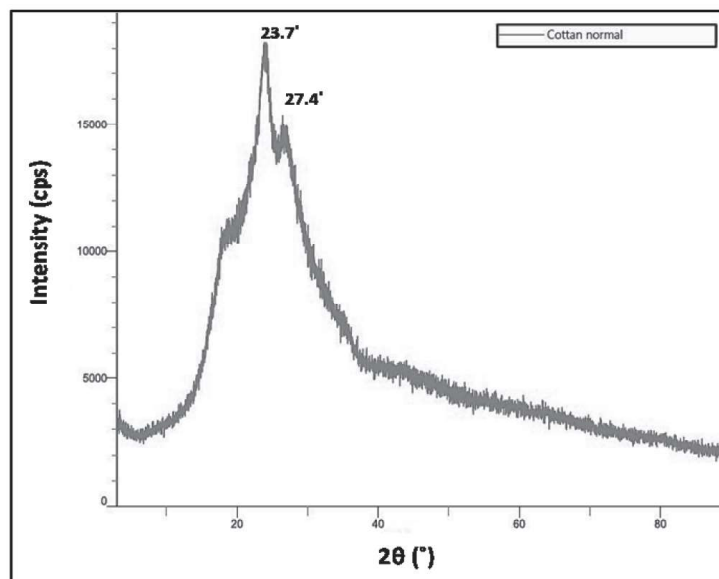


Figure 5: XRD pattern for normal unprocessed Cotton fabric

As the PPy concentration in the sample is so low, the diffraction peaks of PPy/cotton composites before washing were having no prominent

diffraction peaks characteristic of PPy but having one peak at  $27.8^\circ$ . The diffraction peaks intensity of PPy/cotton composites concisely decreases when compared to raw cotton, leading to the PPy being equally dispersed in the sample surface [20].

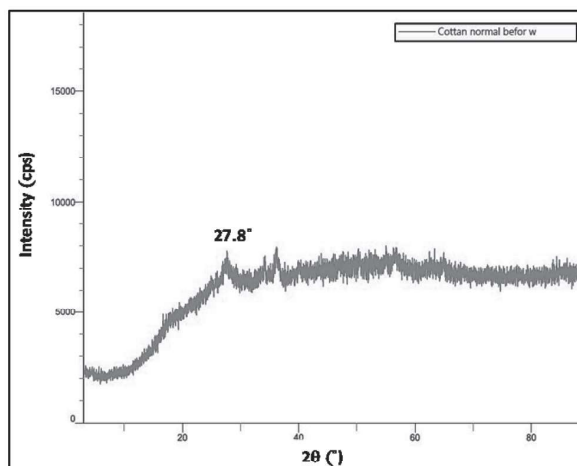


Figure 6: XRD pattern for PPy/Cotton fabric before wash

However, after washing the PPy/cotton fabric sample, the XRD pattern shows distinctive peaks at  $27.32^\circ$  and  $35.71^\circ$ . The highest peak, which has a crystal plane (1 0 4) was observed at approximately  $2\theta = 27.32^\circ$ , signifies the amorphous form of the polymer according to previous literatures [22, 23]. They result from PPy chain dispersion at the interplanar spacing. This is consistent with the previous reports found in the literature [21, 22]. It is observed that the intensity of the diffraction peaks of the PPy/cotton fabric is significantly higher after washing with deionized water than it was before. Thus, the peak at  $2\theta = 27.32^\circ$  is most intense because it indicates the crystal structure's densely packed plane, which effectively scatters X-rays. Some prominent peaks (at  $23.53^\circ$  and  $35.71^\circ$ ) that develop in the fabric sample after washing indicate the sample's crystallinity. This disclosing the sample's partially amorphous nature [22]. Especially in cases when a material is not entirely crystalline, additional peaks in the XRD pattern often appear less strong. Consequently, they might be less significant and challenging to correctly evaluate the planes. Due to the repetitive unit of pyrrole ring and highly aligned polymeric chains, both patterns of PPy/Cotton fabric show a common broad peak at about  $2\theta \approx 27^\circ$ . Further, this broad peak indicates the



intermolecular  $\pi$ - $\pi$  stacking structure and amorphous packing of the developed PPy [20, 21].

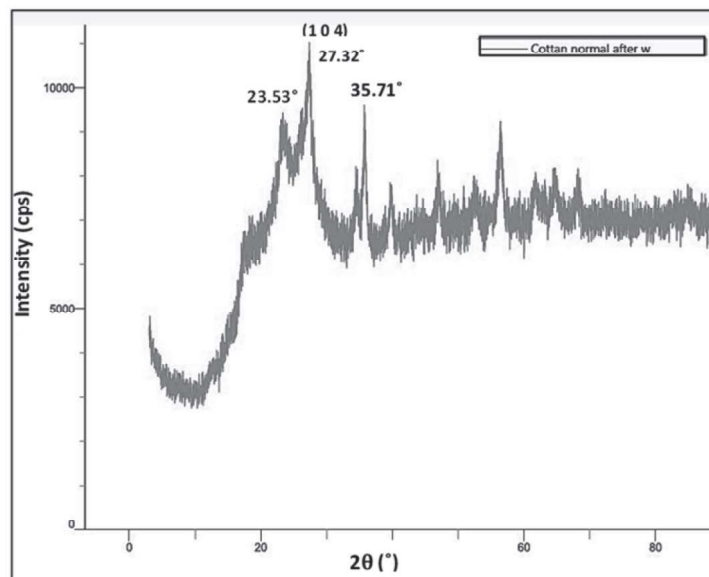


Figure 7: XRD pattern for PPy/Cotton fabric after wash

### 3.3. Electrical properties:

The surface resistance to temperature graph for the PPy/Cotton samples, both before and after washing, is illustrated in Figure 8. Four probes setup were used to measure surface resistance of the cotton sample at various temperatures. The surface resistance of both cotton samples steadily increases, and it is particularly high for the cloth before washing. The pointed probe of the four probe apparatus damages the sample. For this reason, we incorporated two probe method for evaluating electrical conductivity. This approach involves sandwiching a fabric sample between the probes which is having surface area of 1 cm<sup>2</sup> and calculating the electrical conductivity using the formula,

$$\text{Conductivity, } \sigma = \frac{I t}{V A} = \frac{t}{R \times A}$$

Where,  $V$  = voltage applied,  $t$  = Thickness of the sample,  $I$  = current,  $R$  = resistance of the sample,  $A$  = Area of the sample which is in contact with the probes.

Figure 9 and 10 shows the I-V characteristics of the PPy/Cotton sample both before and after washing at different temperatures. We

estimated the slope of the curve at room temperature, using these curves,  $\text{Slope} = \frac{I}{V} = \frac{1}{R}$ . Naturally, it may be inferred that cleaning the PPy/cotton fabric could influence the electrical conductivity of PPy/cotton composites. Any dirt, dust, or pollutants that may have developed on the fabric surface can be eliminated by washing the fabric in deionized water. The electrical conductivity of PPy coated fabric can be improved by eliminating these impurities, also reducing the surface resistance. The calculated electrical conductivity from the values obtained is  $1.45 \times 10^{-2} \text{ S/cm}$  for PPy/Cotton fabric before washing with deionized water. For after washing sample, I-V curve shown in Figure 10, the electrical conductivity increased with the value calculated as  $56.24 \text{ S/cm}$ , which is much higher than the before washing sample.

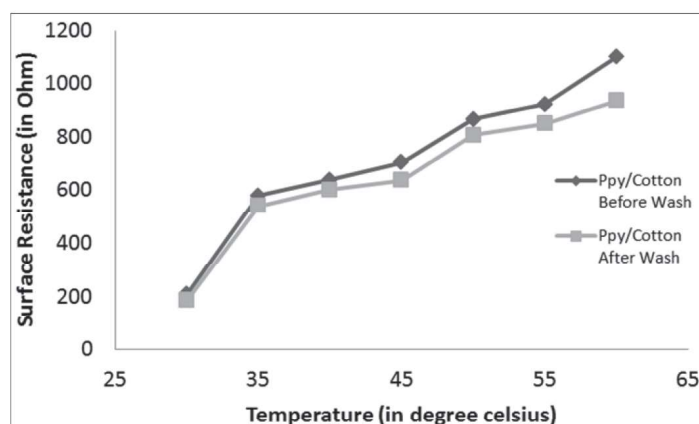


Figure 8: The influence of temperature on surface resistance of PPy/Cotton fabric samples

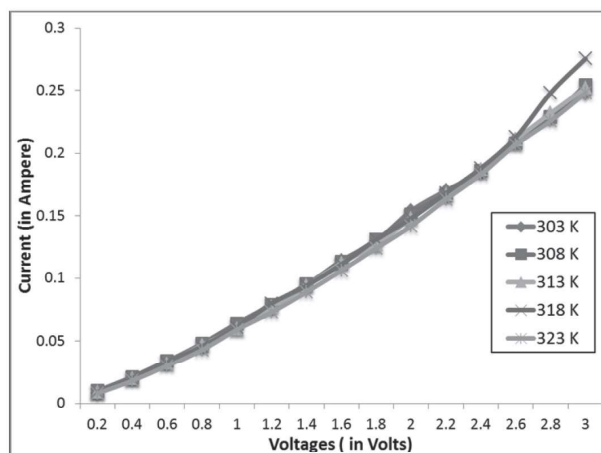


Figure 9: I-V characteristics curve of PPy/Cotton fabric before wash

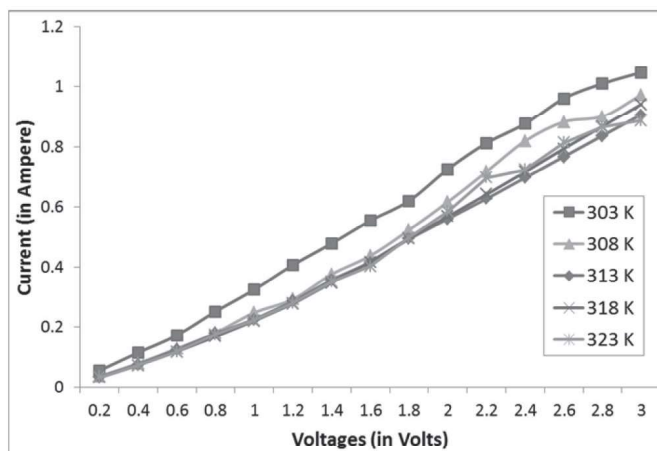


Figure 10: I-V characteristics curve of PPy/Cotton fabric after wash

### 3.4. Fabrication of Gas Sensor:

Finally, these PPy/Cotton fabrics were utilized for gas sensing. Sensors are required to detect and assess the concentration of gaseous contaminants. Here, PPy/ cotton fabric employed in the fabrication of gas sensor devices. The gas detecting setup is depicted in Figure 11.

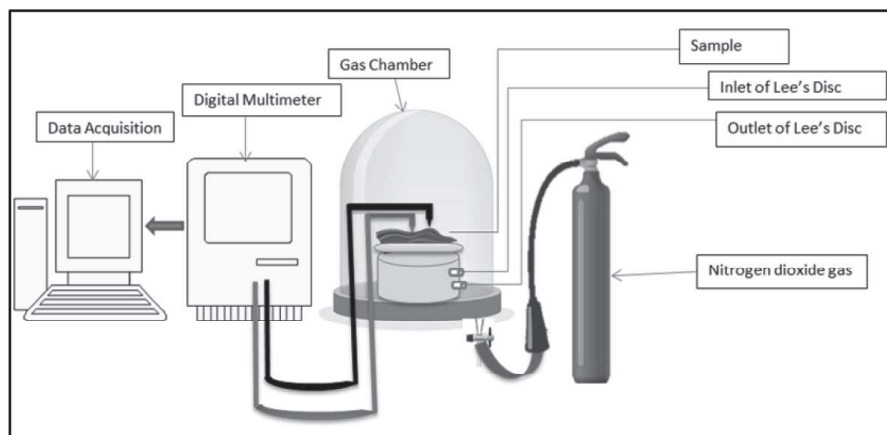


Figure 11: Gas Sensing Setup

By means of a Keithley 2000 Multimeter, the temperature-controlled resistance of a sample of conducting fabric was examined with respect to time. The sensor was subjected to  $\text{NO}_2$  gas concentrations varying from 5 ppm to 100 ppm in the test chamber to determine its sensitivity. The sensitivity of composite fabric was reported for varied concentrations for before and soon after exposure to  $\text{NO}_2$  gas is depicted in Figure 12.

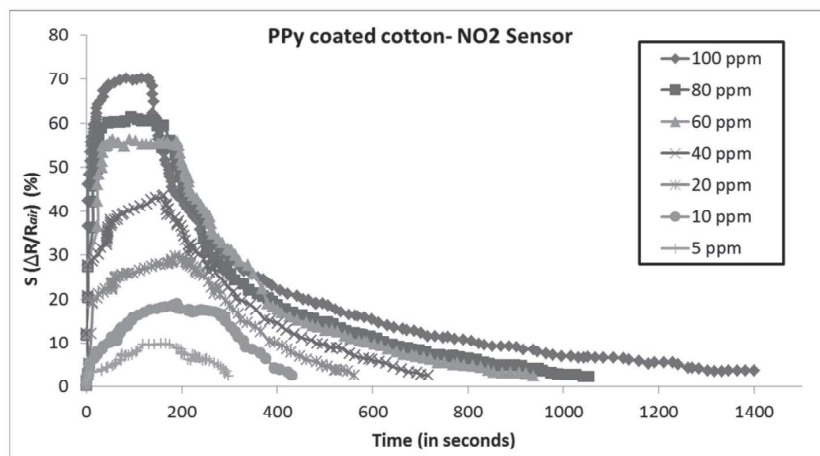


Figure 12: The influence of NO<sub>2</sub> gas Concentration on the Sensitivity of Sensor

The response of the sensor's sensitivity (S) evaluated by,

$$S \% = \frac{|R_{air} - R_{gas}|}{R_{air}} \times 100\% = \frac{|\Delta R|}{R_{air}} \times 100\%$$

The sensitivity of the PPy/Cotton sample sensor to the various concentration of NO<sub>2</sub> gas sensor at 5 ppm to 100 ppm was found to be 4.68 % to 67.31%. This pattern of behaviour corresponded to the NO<sub>2</sub> gas molecules adhering to the PPy coated fabric sample surface. The number of active sites for adsorption increased with the level of NO<sub>2</sub> gas, which improved the sensor's sensitivity.

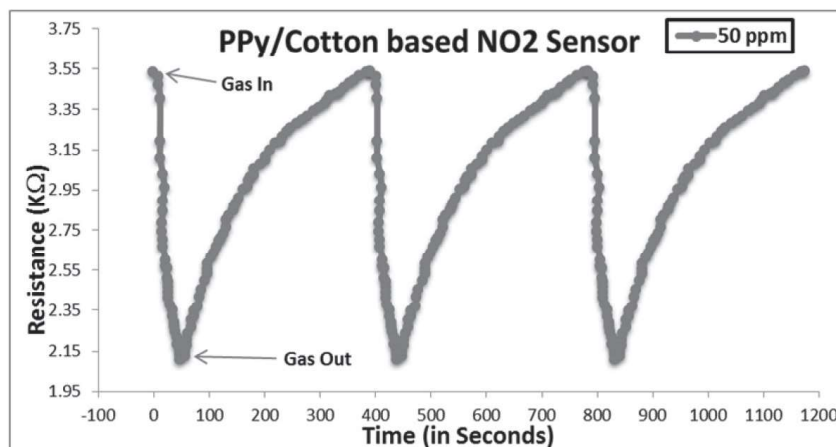


Figure 13: Resistance characteristics of NO<sub>2</sub> sensor at 50 ppm gas concentration

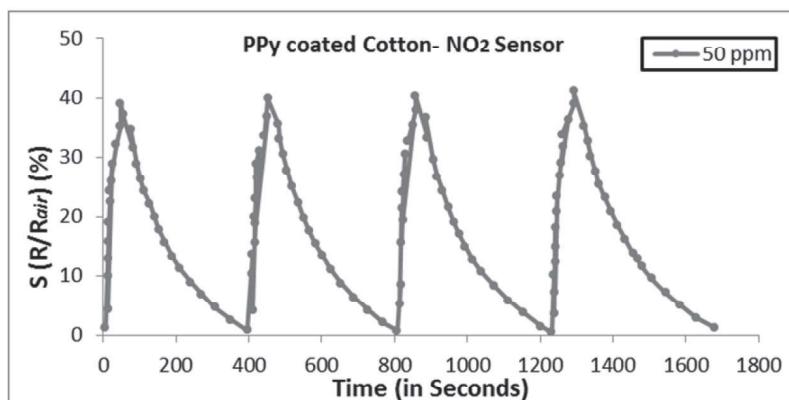


Figure 14: Sensitivity of NO<sub>2</sub> gas sensor at 50 ppm gas concentration

We obtained the repetitive response of PPy/Cotton sensor to 50 ppm of NO<sub>2</sub> gas. For these ppm value, the resistance before and after exposure of NO<sub>2</sub> gas with respect to time is recorded in Figure 13 and sensitivity of NO<sub>2</sub> sensor at 50 ppm with function of time is shown in Figure 14. It is noteworthy that the PPy coated cotton fabric gas sensor responds well to NO<sub>2</sub> gas, especially at a concentration of 50 ppm. This trend in Figure 13 also indicates that it is n-type sensor. Furthermore, the observed linearity of response in the range of 5 to 100 ppm NO<sub>2</sub> gas concentration is an astonishing. Figure 15 showing response and recovery time of the PPy/Cotton based NO<sub>2</sub> gas sensor. Recovery time of the sensor goes on rising with the increasing concentration of NO<sub>2</sub> gas sensor while the response time falling. A critical feature of a gas sensor is its linearity of response, which suggests that the target gas concentration may be accurately associated with the sensor's output. This implies that the PPy coated cotton fabric sensor can be an effective device for tracking NO<sub>2</sub> gas concentrations within the permitted range.

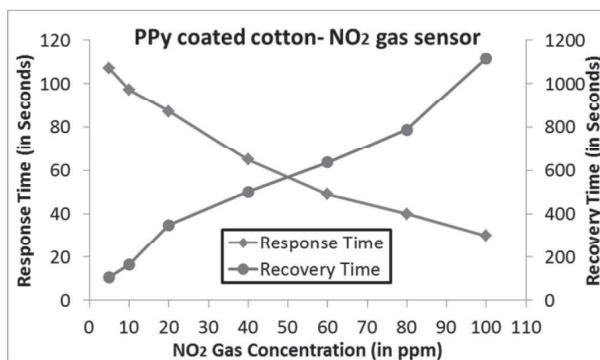


Figure 15: Response time and Recovery time of the NO<sub>2</sub> gas sensor

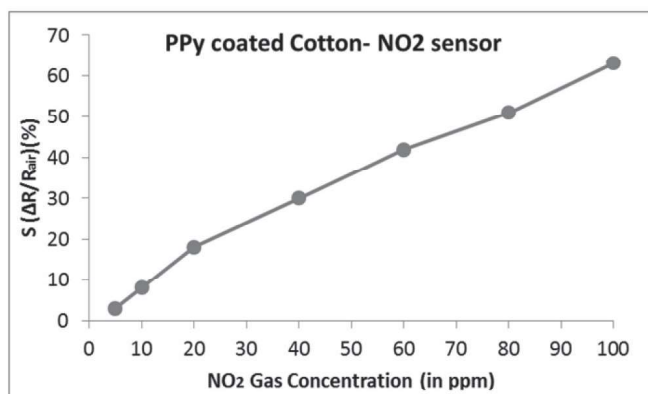


Figure 16: Sensitivity of the PPY coated NO<sub>2</sub> gas sensor with respect to the NO<sub>2</sub> gas concentration

Figure 16 displays the sensitivity of the NO<sub>2</sub> gas sensor at various concentrations. The sensor's sensitivity increased along with the concentration of NO<sub>2</sub> gas. The positive finding of our study is that the PPy-coated cotton fabric sensor becomes more sensitive to changes in NO<sub>2</sub> concentration. An effective method to remove contaminants or residues from the cotton fabric coated with PPy is to wash the sample with deionized water. By cleaning the sample surface, more adsorbing sites may become visible or activated, improving the sensor's ability to interact with and detect NO<sub>2</sub> molecules. Furthermore, the washing process may impact the surface of the PPy coating, making it more reactive and favourable for NO<sub>2</sub> adsorption. This alteration most likely plays a role in the observed rise in sensitivity to elevated NO<sub>2</sub> levels. The sensor's constant and linear response to NO<sub>2</sub> gas concentration provides as additional proof for its suitability for gas sensing applications. In gas sensors, linearity is especially important since it makes calibration procedures easier and allows for accurate quantification of gas concentrations.

#### 4. Conclusions

In summary, our study successfully demonstrates the preparation of PPy-coated cotton fabrics through in situ chemical polymerization. Comprehensive characterizations, comprising FTIR and XRD supported the interactions of FeCl<sub>3</sub> and PPy on textiles, revealed structural modifications and more effective PPy dispersion after washing. Even after washing, the PPy coated sample shows superior electrical conductivity with values  $1.45 \times 10^{-2}$  S/cm before and 56.24

S/cm after, indicating that cleanliness is crucial for the most excellent sensor functioning.

Gas sensing experiments demonstrated the sensor's exceptional sensitivity to NO<sub>2</sub> gas concentrations, which ranges from 4.68% to 67.31% for 5 ppm to 100 ppm. Its potential for dependable monitoring is highlighted by its repeatability to 50 ppm of NO<sub>2</sub> gas and positive correlation with NO<sub>2</sub> gas concentration. Its believability for use in gas sensing applications is increased by the linearity that has been observed in the 5 to 100 ppm range that is this sensor works well at very low ppm too. The work underlines the value of thorough material characterisation and the efficiency of easy cleaning techniques in improving sensor performance. These findings open up new avenues for the development of advanced textile-based devices and highlight the potential for further research in this exciting field.

#### **5. Author contribution Statement:**

**Vinod S. More:** Conceptualization, Methodology, Writing – original draft. **B.K.Sakhare:** Literature Survey, Data interpretation. **R.P.Tandel:** Experimentation, Data collection. **Dr.G.G. Padhye:** Visualization, Supervision. **Dr.T.N.Ghorude:** Supervision, validation.

#### **6. Conflict of Interest:**

The authors state that they have no any conflicts of interest or relationships that may seem to have influenced the work described in this study.

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