

# STUDIES ON VAPOUR PHASE POLYMERISATION OF PYRROLE AND ELECTROCONDUCTIVE TEXTILES PREPARED THEREFROM FOR SENSING APPLICATION

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## Abstract

Electroconductive textiles or e-textiles are a group of promising new materials that are becoming very important for their potential use in the areas of electromagnetic shielding, electrothermal generators, electrostatic dissipation, sensors and actuators, and data transfer, to name a few. The current research work explores electrochemical polymerisation and vapour phase polymerisation methods in depth with the aim to develop a fast and industrially scalable process for large-scale fabrication of flexible and non-metallic electroconductive textiles.

In electrochemical polymerisation, the rate of polymerisation and the polymer deposition can be controlled quite precisely by controlling the applied voltage or system current during polymerisation. The solution used for electrochemical polymerisation typically contains one or more compounds in addition to the monomer. These compounds are added with the aim to either achieve low electrical resistance polymer deposition or to obtain a higher polymer add-on. However, there has been an ambiguity in the role of the compounds. Understanding the actual role of the compounds, as to whether they function as a dopant or an electrolyte, is crucial. The current study compares sodium nitrate, sodium 2-anthraquinone sulfonate (AQSA-Na), p-toluene sulfonic acid (pTSA) and oxalic acid to understand the role of different compounds in electrochemical polymerisation. A metric called charge utilization factor (CUF) is defined to characterise each chemical. A suitable combination of a low CUF compound with a high CUF compound turns out to be a better option for achieving low electrical resistance when applied over a polyester fabric. The only hindrance is that it requires a conductive substrate connected with a metallic back electrode.

Vapour phase polymerisation (VPP) is another method used to develop electroconductive fabric. It is a single-step process and does not require a solvent for the dissolution of pyrrole, which makes it eco-friendly. In this work, a glass setup is designed and fabricated in-house to perform VPP on fabric under a controlled environment. The setup consists of separate sections for boiling pyrrole and for vapour deposition on the sample. The effects of oxidant ( $\text{FeCl}_3$ ) concentration, dopant (pTSA) concentration, polymerisation time and pyrrole temperature are studied. The experimental data reveal that the oxidant concentration and the rate of vapour formation play a decisive role in determining the surface resistance of polypyrrole-coated fabric. Quite interestingly, a suitable selection of process parameters yields an electroconductive fabric with a surface resistance of around  $220 \Omega$ . Ultrasonication is carried out to assess the surface adherence of polypyrrole to the fabric. After 10 min of sonication, the fabric resistance increases sharply due to the removal of polymer from the surface, as confirmed also by air permeability and spectrophotometry. The fabric, due to its flexible nature, does not show significant change in resistance during bending and slight turning up to  $20^\circ$  along its axis.

Further, a new process is developed to obtain a faster process by combining electrochemical polymerisation and vapour phase polymerisation. This novel process, which has been named electrically assisted chemical vapour polymerisation (eCVP), offers the flexibility of applying a voltage (up to 12 V) during polymerisation to boost the formation of polypyrrole. The glass

setup used for VPP is modified, and a power supply is connected in series to the fabric sample. Experimental data reveal a decrease in resistance with increasing polymerisation time and the applied voltage. The oxidant ( $\text{FeCl}_3$ ) concentration also plays an equally important role. This novel process results in a higher polypyrrole add-on and over 50 % reduction in surface resistance compared to the VPP method within 3 min time. It also shows the possibility of scale-up for large-scale continuous production of electroconductive fabric.

The polypyrrole-coated fabric prepared by using eCVP is used for fabrication of interdigitated capacitive sensors (IDS). Different designs are prepared to obtain an efficient design of the sensor by changing the width and the number of fingers. Thus, multiple designs with varying capacitance are obtained. The capacitive behaviour of IDS is evaluated at different frequencies from 20 Hz to 2 MHz, and the most stable and cost-effective design is selected. The prepared IDS is then used for RH sensing and compared with commercially available sensors. The polypyrrole-coated fabric also shows a change in the resistance when exposed to acetone and ammonia vapours. Since the behaviour of textile fabrics often depends on the environment, one sensor is prepared with polyethylene lamination to protect it from environmental changes. The capacitance vs frequency behaviour of laminated and unlaminated sensors show that lamination does not affect the working of the IDS for humidity sensing.

At the end, a prototype machine is designed and developed for the preparation of electroconductive fabric continuously. The machine is used to polymerise polypyrrole over nylon, cotton, and polyester fabrics at different speeds. The pre-treated fabric is dipped in the oxidant solution, followed by squeezing. This oxidant-rich fabric is exposed to pyrrole vapours in the reaction chamber. During the exposure of the fabric to pyrrole vapours, an electrical voltage is applied simultaneously to the fabric. The successful preparation of electroconductive fabric by employing the prototype machine demonstrates the possibility of scaling up the eCVP process for large-scale production of electroconductive textiles.