



A comparative study of reaction kinetics of in-situ chemical polymerization of polypyrrole onto various textile fibres



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ABSTRACT

An attempt has been made to prepare electrically conductive fibres by coating common textile fibres such as cotton, wool, silk, polyester, nylon, acrylic, and polypropylene with polypyrrole by in-situ chemical polymerization of pyrrole using FeCl_3 as oxidant and p-toluene sulphonic acid as dopant in aqueous medium. Effects of the pyrrole, oxidant and dopant concentrations and substrate textile fibres on reaction kinetics of the in-situ chemical polymerization are investigated. A comparative analysis is done to find out the most suitable textile fibre as substrate for in-situ polymerization of pyrrole. Highest polypyrrole add-on per unit surface area have been observed in case of wool fibre (12.094 g^{-2}) followed by cotton (5.125 g^{-2}) and silk (4.621 g^{-2}). Whereas, the synthetic fibres show comparatively lower polypyrrole add-on than that of the natural fibres. This difference in add-on may primarily be attributed to interaction between these fibres with polypyrrole, which lead to improved fixation of polypyrrole on these fibre surfaces. The chemical interaction is investigated by FTIR and DSC analysis of the control and the polypyrrole coated fibres. Significant chemical interaction has been found in case of wool, silk, cotton and polyester fibres in terms of formation of hydrogen bonds.

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1. Introduction

Since long, various approaches have been adopted for the preparation of electro-conductive textiles such as metal coating, inserting metal wire and fibres inside the textile structure, blending metal fibres during spinning and weaving, etc. [1–4]. Studies have also been reported on graphene incorporated textile substrates where conventional dip and dry technique has been adopted in order to modify the textile material to be electrically conductive [5–8]. All these researches mainly focus on the development of the process of incorporation of graphene into cotton and polyester textiles and evaluation of some properties of the modified textiles. The modification process is quite complicated and time consuming. Most of the researchers used multiple dipping cycles to achieve higher graphene add-on and yet the electrical conductivity obtained is quite poor. Most promising trend is the treatment of textiles with conducting polymers. Conductive polymers such as polypyrrole (PPy), polythiophene, and polyaniline have good electrical conductivity ($10\text{--}10^4 \text{ S/cm}$), easy processability and diversified functional properties that make them attractive to researchers in recent years. Successful application of these polymers on textile materials will yield non-metallic polymeric electro-conductive textiles, which will possess synergistic properties of both the conductive polymer as well as textiles and open up many potential application areas.

Random studies are conducted on applications of PPy onto textile substrates by various methods [9–13]. Among them, in-situ chemical polymerization process in presence of textile substrate has been considered as most suitable due to its simplicity and suitability for large-scale production. By this process, individual textile fibres are coated with self-assembled layer of PPy by adsorption at liquid–solid interface [14–16].

The selection of pyrrole (Py), oxidant and dopant concentrations for in-situ chemical polymerization has been random so far [17–21]. There is lack of information in literature about the influence of these reagent concentrations on reaction kinetics of in-situ polymerization of pyrrole. The presence of textile fibres in reaction bath during in-situ polymerization may influence the reaction kinetics. As the chemical nature of common textile fibres such as cotton, wool, silk, polyester, nylon, acrylic, etc. are different; they may influence the reaction kinetics differently. The selection of substrates has so far been purely by the choice of the researchers. Only few researchers tried to identify the effect of textile substrate on kinetics of PPy formation. Gregory et al. found some influence of textile substrate on in-situ polymerization of pyrrole [22]. However, later on, Ferrero et al. used polyester textiles and found no influence of it on in-situ chemical polymerization of pyrrole [23]. These results seem to be contradictory and needs detail and comprehensive study to understand the fact. Intermolecular interaction between these textile fibres and polypyrrole may lead to the uniform and durable coating, which will yield highly electro-conductive fibres.

In the light of the above discussion, the aim of the present study is to investigate the influence of reagent concentrations and substrate fibres

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on reaction kinetics of in-situ polymerization of pyrrole, to find out the most suitable reaction conditions and substrate fibres for obtaining durable and coherent PPy coating onto textiles. An attempt has been made to investigate the intermolecular interaction between these textile fibres and polypyrrole.

2. Materials and methods

2.1. Materials

Raw cotton (1.5 den), raw wool (6 den), raw silk (4 den), virgin polyester (6 den), virgin nylon 6 (6 den), virgin acrylic fibres (6 den) and virgin polypropylene (6 den) fibres are selected as substrate of in-situ chemical polymerization of pyrrole. These fibres are procured from Dev Woolen Mills, Ludhiana, India; Himachal Silk Mills, Himachal Pradesh, India; and Reliance Industries Ltd., India.

2.2. Chemicals

Pyrrole (Leonid Chemicals, Bangalore, India), Ferric chloride (FeCl_3) (Qualigens Fine Chemicals, Mumbai, India), p-toluene sulphonic acid (PTSA) (S D fine-chem Ltd., Mumbai, India), Caustic soda (NaOH) (S D fine-chem Ltd., Mumbai, India), Sodium carbonate (Na_2CO_3) (S D fine-chem Ltd., Mumbai, India), Sodium dithionite (Qualigens Fine Chemicals, Mumbai, India), Lissapol N (S D fine-chem Ltd., Mumbai, India), Oleate soap (S D fine-chem Ltd., Mumbai, India). All these chemicals are laboratory grade and used as received.

2.3. Sample pretreatment

Raw natural fibres (cotton, wool, silk) have natural wax and impurities. The synthetic fibres (polyester, nylon, acrylic and polypropylene) also have oils and impurities which are applied or acquired during their manufacturing. These oil, impurities and wax need to be removed from fibre surface before in-situ chemical polymerization by suitable pretreatment process such as scouring or degumming.

Raw cotton fibres are scoured with a solution of 4% sodium hydroxide (NaOH) and 2% sodium carbonate (Na_2CO_3) at 130 °C for 4 h. Material to liquor ratio is kept 1:20.

Scouring of raw wool is carried out with a solution of 3% detergents (oleate soap) and 2% sodium carbonate at 50 °C for 15 min.

Silk fibres are degummed in alkaline solution of 1% NaOH, 8 g l⁻¹ soap, 1 g l⁻¹ non-ionic surfactant (Lissapol N) at 90 °C for 1 h with a material to liquor ratio of 1:30.

The scouring of polyester, nylon, acrylic and polypropylene is carried out in alkaline solution of 2 g l⁻¹ caustic soda, 1 g l⁻¹ soda-ash, and 1 g l⁻¹ Lissapol N with a treatment time of 1 h at a temperature of 80 °C.

After scouring, all fibres are washed with 0.001 N acetic acid solution to neutralize the alkali and then rinsed thoroughly.

2.4. Preparation of monomer and oxidant bath

The monomer solution is prepared by dissolving pyrrole in de-ionized water and oxidant solutions is prepared by dissolving FeCl_3 and PTSA in de-ionized water separately. Material to liquor ratio of 1:20 is maintained for both the cases. Concentration of pyrrole, FeCl_3 and PTSA used in the experiment are in the range of 0.5 M to 2.5 M, 1 M to 2.5 M, and 0.1 M to 1 M respectively. Selection of these ranges of concentration is based on the literature where the previous researchers reported satisfactory results. At very low concentration either reaction will not occur or become slower. With very high concentration, uncontrolled growth and over oxidation of polypyrrole occur leading to poor polymer properties.

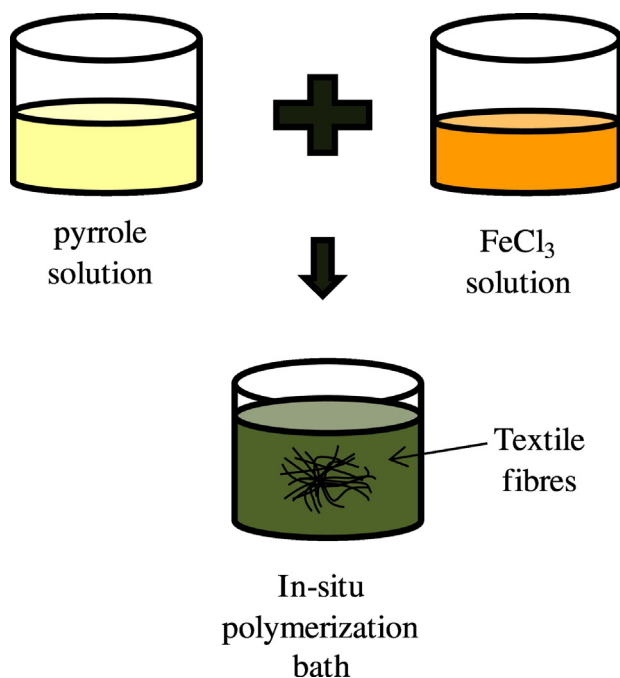


Fig. 1. In-situ chemical polymerization.

2.5. In-situ chemical polymerization of pyrrole

A single bath process is followed for in situ chemical polymerization of pyrrole as shown in Fig. 1. At first, oxidant and monomer solutions are cooled down separately to 5 °C in a cryostat. After cooling, they are poured and mixed together in another container having predetermined weight of textile fibres. The mixture is stirred continuously, oxidative polymerization of pyrrole starts and PPy begins to precipitate in solution in bulk and deposit on the fibre surface as well. After a polymerization time of 1 h, 1 M sodium dithionite solution is added to stop the reaction. Sodium dithionite reduces FeCl_3 to FeCl_2 and as a result, oxidative polymerization of pyrrole stops. Then, synthesized bulk PPy and PPy coated fibres are filtered out from the solution and washed thoroughly by de-ionized water and filtered to collect the loose PPy and PPy coated fibres. The filtrate PPy and coated fibres are oven dried at 80 °C for 3 h before measurement. The same polymerization process and reaction condition

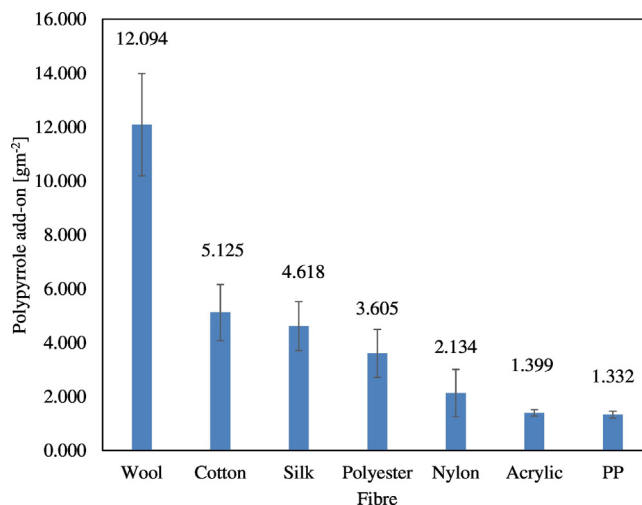


Fig. 2. Polypyrrole add-on per unit surface of different textile fibres.

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