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Graft copolymerization of *N*-vinylpyrrolidone onto stearyl alcohol to impart water repellency and antibacterial properties for cotton/polyester fabric

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ABSTRACT

A PVP/SA adduct was prepared by reacting of N-vinylpyrrolidone with stearyl alcohol in presence of ammonium persulphate as initiator in different preparation reaction conditions. The optimum conditions to prepare that adduct are VP/SA, 25%; LR, 1 l/kg; reaction temperature, 80 °C and reaction time, 40 min. The graft copolymerization of N-vinylpyrrolidone onto stearyl alcohol was confirmed by the FTIR analysis. The TEM image of the prepared adduct emulsion shows that its particle size ranges from 45 to 173 nm. Finishing of cotton/polyester fabric sample with easy care finishing bath containing 80 g/l of that adduct emulsion results in an increasing in the tensile strength, water repellency rating, antibacterial properties, softness degree, and stiffness along with a reduction in the resiliency of treated fabric. The surface of the prepared adduct treated fabric was characterized via scanning electron microscope.

1. Introduction

In the last decades, there is an urgent need for developing the textile products to satisfy the customers' demands. Indeed, textiles having performance, functional and comfort properties are highly desired and that properties can be achieved by means of chemical and biotechnological finishes. The chemical finishing is capable of affording finished fabrics with one or multi-functional properties such as water and oil repellency, antimicrobial properties, pleasant smell, ultraviolet protective properties, self-cleaning, etc [1–9].

In summary, finishes that have repelling properties for water and oil are important for finishing of raincoats as well as imparting repelling properties for carpets and furniture to enable easier cleaning from water or oil based soils. A textile repellent finish repels water or oil by reducing the free energy at fiber surfaces [10–12]. Water repellency can be achieved by means of paraffin repellents, stearic acid–melamine repellents, silicone repellents and fluorocarbon-based repellents. Paraffin based finishes are emulsions containing aluminum or zirconium salts of hydrophobic substance, such as paraffin wax or wax-like substances such as high molecular weights fatty acids and alcohols [1,7,8]. Furthermore, microorganisms results in harmful effects for textiles and wearers. They cause staining, unpleasant odor as well as reduction in mechanical strength for textiles and on the other hand they act as a source for cross infection for textiles wearers. Accordingly, the antimicrobial textile finishes are necessary to minimize such harmful effects. Many antimicrobial finishes can be used to impart textiles with antimicrobial properties such quaternary ammonium compounds, biguanides, alcohols, phenols, aldehydes, metal ions, metals or metal oxides nano-particles, organometallic compounds, chitosan, etc [2,4,5,9,12].

Meanwhile, poly (*N*-vinyl-2-pyrrolidone) (PVP) is a water-soluble, nontoxic, synthetic polymer. PVP polymers are film formers, protective colloid and suspending agents, dye-receptive agents, binders, stabilizers, detoxicants, and complexing agents [2,5,7,9]. PVP has a limitation of the lack of a reactive group that can be undergo chemical modification [2,5,7,9]. Previous studies have reported the feasibility of the free radical polymerization of PVP or N-vinylpyrrolidone monomers (VP) in the presence of substrates having hydroxyl groups such as stearyl alcohol (SA) [7], cellulose [2,9], starch [13], gums [14,15], chitosan [16], etc. The present study aims to synthesis and characterize a new adduct based on the free radical polymerization VP in presence of SA as well as to investigate the potential application of such adduct emulsion as water repellent as well as antibacterial finish for cotton/polyester blended fabric.

2. Experimental

2.1. Materials

Bleached cotton/polyester (50/50) blended fabric of plain weave structure, weight of 105 g/m^2, count (Ne) of 40/1and thickness of

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0.24 mm was supplied by Misr Spinning and Weaving Co., Mehalla ELKobra, Egypt. Arkofix® NG, aqueous solution of dimethyloldihydroxyethylene urea (DMDHEU), kindly supplied by Clariant, Egypt, was used. Egyptol® (a nonionic detergent, based on ethylene oxide condensates) provided by the Egyptian Company for Starch, Yeast and Detergents, Alexandria, Egypt, was used. N-vinylpyrrolidone (VP) supplied by Sigma Aldrich was used. Stearyl alcohol (SA) of purity 95%, ammonium persulphate (APS), hydroquinone as antioxidant and isopropanol were all of laboratory grade chemicals.

2.2. Methods

2.2.1. Graft copolymerization of N-vinylpyrrolidone onto stearyl alcohol

The PVP/SA adduct was prepared as follows: 5 g of stearyl alcohol was melted at 60 °C in 250 ml round flask equipped with a condenser in a thermo stated water bath. To that melt, a VP aqueous solution of specific molar ratio to SA (0–50%) was added followed by stirring and raising the temperature to a specific temperature (60–90 °C). After that, freshly prepared APS aqueous solution of specific concentration (0.0276–0.2656 mmol) was added to the reaction medium, keeping the LR in the reaction medium within the range of 0.6–1.5 l/kg, and then the reaction medium was left for a certain time (15–60 min) until the completion of the polymerization reaction. Finally, the products of the polymerization reaction were immediately cooled to room temperature and 500 ppm of hydroquinone as inhibitor was added before measuring the unsaturation content. It must be mentioned that the reaction product completion and after cooling consists of two layers; a clear aqueous layer and a white solid layer.

2.2.2. Formation of PVP/SA adduct emulsion

To prepare a specific concentration of the PVP/SA adduct emulsion, a specific volume of hot distilled water at 70 °C is added to the reaction product followed by stirring the mixture using a strong homogenizer for 3 min to form a homogeneous oil in water emulsion. For the purpose of IR analysis of PVP/SA adduct, the reaction product after completion was immediately, i.e. without the addition of hydroquinone, poured in 50 ml isopropanol and stirred for 30 min followed by solvent evaporation, drying and storing over $CaCl_2$ in a desiccator for at least 48 h before IR analysis.

2.3. Fabric treatment

Fabric samples of $30 \times 30\,cm^2$ were padded twice in solutions containing DMDHEU (0–80 g/l) as a crosslinker, ammonium persulfate (6 g/l) and hybrid emulsion (0–100 g/l), to a wet pick up of ca 100%. The padded samples were firstly dried at 100 °C/3 min in Wenner Mathis AGCH-8155 oven and then cured at 150 °C/3 min. The finished samples were then washed with distilled water at 50 °C for 10 min, thoroughly rinsed and finally dried for testing.

2.4. Testing and analysis

- The extent of polymerization, expressed as percentage total conversion, %TC, was determined by assessing the unsaturation (double bonds) content before and after polymerization [17].
- Water repellency rating (WRR) was performed using the spray test as described by AATCC Test Method 22–1989.
- Antimicrobial activity of control and finished cotton fabric were tested, expressed in the inhibition zone per millimeters, according to the disc diffusion method, AATCC 147–2004. The antibacterial activities of the untreated blank as well as finished fabrics were tested against the following bacteria:

  Gram-positive bacteria: *Staphylococcus aureus* (SA).

  Gram-negative bacteria: *Escherichia coli* (EC).

- Dry wrinkle recovery angle (WRA) was determined according to ASTM D-1296-98.
- The tensile strength (TS) of the finished fabric sample was tested in the warp direction according to ASTM D-2256-98.
- Stiffness (S) was determined in the warp direction according to ASTM Test Method D 1388-96 using the cantilever apparatus.
- Surface roughness (SR) was measured using a Surfacoder 1700a.
- Durability to wash was assessed by subjecting the fabric to 10 laundering cycles. Each laundering cycle consists of washing the fabric sample with hot water at 50 °C for 10 min using 2 g/l nonionic detergent followed by rinsing and air drying at ambient conditions.
- Infra Red (IR) spectroscopy was carried out using JASCO FTIR Spectrometer.
- Scanning Electron Microscope (SEM) images of the treated and untreated fabric samples were obtained using SEM Model Quanta 250 FEG, FEI Company, Netherlands.
- The particles size of the hybrid emulsion were obtained by transmission electron microscope (TEM) using a JEOL, JEM 2100 F electron microscope at 200 kV.

3. Results and discussion

3.1. Tentative mechanism

Previous literatures [17,18] reported that ammonium persulphate decomposes in aqueous medium to generate free radical species ($SO^{\bullet}{}_4^-$ and $HO^{\bullet}$ that will be denoted by $R^{\bullet}$) which can initiate polymerization reaction of VP in presence of SA as represented in the following equations:

$$S_2O_8^{2-} \rightarrow 2\,SO_4^{\bullet -} \quad (1)$$

$$SO_4^{\bullet -} + H_2O \rightarrow HSO_4^- + {}^{\bullet}OH \quad (2)$$

$$VP + R^{\bullet} \rightarrow RH + VP^{\bullet} \quad \text{(Activated VP molecule)} \quad (3)$$

$$VP^{\bullet} + n\ VP \rightarrow PVP \quad \text{(PVP homopolymer)} \quad (4)$$

$$SA - OH + R^{\bullet} \rightarrow SA - O^{\bullet} + RH \quad \text{(Activated SA molecule)} \quad (5)$$

$$SA - O^{\bullet} + VP \rightarrow PD - O - VP^{\bullet} \xrightarrow{n\,VP} PVP - g - SA \quad \text{(PVP grafted SA)} \quad (6)$$

Consequently, the final product of the polymerization reaction is a mixture of PVP homopolymer, PVP grafted SA and un-grafted SA (intact and oxidized) in a state of entanglement with each other. This mixture will be referred to as PVP/SA adduct. Moreover, the PVP grafted SA will act as a self emulsifiable for the reaction products, i.e. the un-reacted SA, beside the PVP homopolymer that accelerate the disintegration of SA in aqueous formulations and stabilize the resulted emulsion [2,7,9]. In addition, treating cotton/polyester blended fabric with finishing formulation containing the prepared PVP/SA adduct emulsion, DMDHEU as crosslinker and ammonium persulphate as a catalyst, brings about a water repellent fabric. Thus, by virtue of the cationic properties of the PVP segments [7] of the PVP grafted SA species they will orient themselves as heads towards the partially negatively charged cellulosic fibers [7,19] thereby rendering the hydrophobic tail to orient perpendicularly away from the surface and thus provide a hydrophobic layer on the treated fabric surface.