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# An innovative SA/PVP hybrid for attaining water repellent cotton/polyester blended fabric

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**Abstract:** A novel SA/PVP hybrid was synthesized by the reaction of a high concentrated aqueous solution of poly (N-vinyl-2-pyrrolidone) (PVP) with stearyl alcohol (SA) in presence of ammonium persulfate (APS). Results obtained demonstrate that the optimum reaction conditions to synthesis that hybrid is PVP molecular weight (40000 Dalton), PVP concentration (60%), APS/PVP weight ratio (50%), PVP/SA weight ratio (20%), reaction time (55 min) and reaction temperature (80 °C). Upon dispersing that hybrid in water a stable white emulsion was obtained. Moreover, padding cotton/polyester blended fabric in finishing bath containing 60 g/l of the hybrid emulsion as well as 60 g/l of DMDHEU as crosslinker followed by drying at 100 °C/5 min and curing at 150 °C/3 min acquires the finished fabric water repellency rating of 80. On the other hand, the prepared SA/PVP hybrid was characterized by investigation of its IR spectrum as well as the TEM image of its emulsion. Meanwhile, the Scanning Electron Microscope images of untreated and hybrid emulsion treated fabric were investigated. **Keywords:** Polyvinyl pyrrolidone; stearyl alcohol; water repellent fabric; textile finishing.

### 1. Introduction

Poly (N-vinyl-2-pyrrolidone) (PVP) is a water soluble synthetic polymer of extremely low toxicity. It is a film former, protective colloid and suspending agent, stabilizer and complexing agent<sup>1</sup>. PVP has a wide range of industrial applications including biomedical, pharmaceutical and textiles applications. In the textiles field, PVP can be used as a sizing agent, binder, crosslinking agent for cellulosic fabrics, dye-receptive agent and dyestuff dispersant<sup>1-5</sup>. PVP does not possess a reactive group that can undergo chemical modification<sup>5</sup> but it can be crosslinked by heating in air at  $150 \, {}^{\circ}C^{3}$ , gamma and ultraviolet radiation<sup>6,7</sup> or in the presence of potassium persulfate<sup>8</sup>. Previous studies reported that, the crosslinking process of PVP in the presence of persulfate anion proceeds probably by chain scission in the polymer chain beside other competing reactions<sup>4,7,8</sup>.

On the other hand, water repellent finishes are highly demanded to achieve water repellent textiles. They find applications in rain-wear, medical bandages, tents, etc<sup>9</sup>. Water repellent finishes perform their function by reducing the free energy at fiber surfaces. Water repellency can be performed using Paraffin repellents, stearic acid–melamine repellents, silicone water repellents and fluorocarbon-based repellents<sup>9,10</sup>.

The present work aims to synthesis and characterize of a novel SA/PVP hybrid that can be dispersed in water and functionalized as a water repellent finish for cotton/polyester blended fabric.

### 2. Experimental

### 2.1. Materials

Bleached cotton/polyester (50/50) blended fabric of plain weave structure, weight of 125 g/m<sup>2</sup> and count (Ne) of 30/1was and thickness of 0.31 mm supplied by Misr Spinning and Weaving Co., Mehalla EL-Kobra, Egypt. Arkofix<sup>®</sup> NG, aqueous solution of dimethyloldihydroxyethylene urea (DMDHEU), kindly supplied by Clariant, Egypt. Egyptol<sup>®</sup> (a nonionic detergent, based on ethylene oxide condensates provided by the Egyptian Company for Starch, Yeast and Detergents, Alexandria, Egypt) was used. PVP of different molecular weights, 10000, 30000 and 40000 Dalton supplied by Sigma Aldrich. PVP of molecular weight 1,300,000 Dalton and crosslinked PVP supplied by Across Organics, USA. Stearyl alcohol (SA) of purity 95%, ammonium persulfate (APS) as initiator, tertiary butylhydroquinone (TBHQ) as antioxidant, tetrahydrofuran (THF) and acetone were all of laboratory grade chemicals.

### 2.2. Methods

#### 2.2.1. Preparation of SA/PVP hybrid

The PVP/SA hybrid was prepared as follows: specific weight 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 thermostated PVP aqueous solution of specific weight ratio to SA was added followed by stirring and raising the temperature to a specific temperature. After that, freshly prepared APS aqueous solution of specific weight ratio to PVP was added to the reaction medium, keeping the final PVP concentration in the reaction medium within the range of 30-70% according to the reaction conditions, and then the reaction medium was left for a certain time until the completion of the reaction.

### 2.2.2. Formation of the SA/PVP hybrid emulsion

At the end of the reaction, specific volume of hot distilled water at 70°C is added to the reaction products followed by stirring the mixture, using a strong homogenizer, for 3 minutes to form homogeneous oil in water hybrid mother emulsion. Moreover, to obtain an emulsion with specific concentration, the mother emulsion is diluted with water with stirring. On the other hand, to enhance the storage life of the prepared emulsion up to 3 months, tertiary butylhydroquinone may be added as antioxidant to the freshly prepared hybrid emulsion followed by stirring the mixture for extra 2 minutes.

### 2.2.3. Purification of SA/PVP hybrid for IR analysis

At optimum conditions of reaction, 5 grams of the SA/PVP hybrid product was prepared, as mentioned above, and left to cool without homogenization where two phases were formed. The first one is a solid white phase whereas the other phase of the product is a very viscous pale yellow paste. The latter was carefully separated from the reaction medium, washed with a mixture of THF/acetone for five times followed by filtration [11], and finally dried and then stored over CaCl<sub>2</sub> in a desiccator for at least 48 hour before IR analysis (product 1). The former was washed well several times with hot distilled water (40  $^{\circ}$ C), dried and finally stored over CaCl<sub>2</sub> in a desiccator for 24 hour before IR analysis (product 2).

### 2.3. Fabric treatment

Fabric samples of 30X30 cm<sup>2</sup> were padded twice in solutions containing DMDHEU (0 - 80 g/l) as a crosslinker and hybrid emulsion (0 - 80 g/l), to a wet pick up of ca 100 %. The padded samples were dried at 100°C/3 min in Wenner Mathis AGCH-8155 oven and then cured at 150  $^{\circ}$ C/ 3 min. The finished fabrics were then washed with distilled water at 50  $^{\circ}$ C for 10 minutes, thoroughly rinsed and finally dried for testing.

### 2.4. Testing and analysis

- Infra Red (IR) spectroscopy was carried out using Bruker IR Spectrometer.
- 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.

- Water repellency rating (WRR) was performed using the spray test as described by AATCC Test Method 22-1989.
- 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 5, 10 and 15 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.
- Scanning Electron Microscope (SEM) images of the treated and untreated fabric samples were obtained Using SEM Model Quanta 250 FEG, FEI Company, Netherlands.

### 3. Results and discussion

#### 3.1. Tentative mechanism

Previous studies<sup>12,13</sup> reported that ammonium persulphate (APS) decomposes in aqueous medium to generate SO<sub>4</sub><sup>--</sup> and HO<sup>-</sup> free radical species (equations 1 and 2). Thus, it is expected that when an aqueous reaction medium containing PVP, SA as well as APS is agitated, macroradicals of SA and PVP will be formed (equations 3 and 4) giving rise a formation of crosslinked PVP<sup>7,8,14</sup> as well as SA grafted with PVP (equations 5)<sup>12-14</sup>. The later serves as a self emulsifiable for the reaction products beside to the merit of the crosslinked PVP that accelerate the disintegration of SA in aqueous formulations and stabilize the resulted emulsion<sup>15</sup>. For simplicity, grafted SA will be designated by SAPVP species. In addition to the aforementioned products, the reaction products may include oxidative degraded PVP and SA chains beside unreacted starting materials, i.e. SA and PVP; all in state in a state of entanglement<sup>7,8,12-14</sup>. For convenience, the reaction products will be referred to as "SA/PVP hybrid". The following equations may represent the formation of SA and PVP macroradicals as well as SAPVP species.



where R represents SO<sup>-4</sup> and HO free radicals.

iii) Graft of SA with PVP (i.e. formation of SAPVP species)



On the other hand, treating cotton/polyester blended fabric with finishing bath containing the emulsion of SA/PVP hybrid, DMDHEU as crosslinker and the remaining decomposed APS as a catalyst, the PVP segments, that having cationic properties<sup>3</sup>, of the SAPVP species will orient themselves as heads towards the

partially negatively charged cellulosic fibers thereby rendering the hydrophobic tail to orient perpendicularly away from the surface and thus provide a hydrophobic layer on the fabric surface.

Needless to say that the presence of the DMDHEU in the aforementioned finishing bath enhances significantly the water repellency of the treated fabrics by fixation of SA.OH onto the finished fabric surface (equation 6) as well as coating that fabric with the crosslinked stearyl alcohol (equation 7).



Beside the above interactions, a competitive reaction of crosslinking of cotton cellulose may be also occurred (equation 8).



Table 1 may support that tentative mechanism as it can be seen that good emulsion was formed and water repellent fabric was achieved only when SA reacts with PVP in presence of APS as initiator.

Table 1: Effect of reaction ingredients on the emulsion state and WRR of the treated fabric.

| Reaction ingredients | Emulsion state                    | WRR |
|----------------------|-----------------------------------|-----|
| SA + APS             | No emulsion                       | 0   |
| SA+ PVP              | No emulsion                       | 0   |
| SA + crosslinked PVP | No emulsion                       | 0   |
| *SA + PVP+ APS       | Emulsion with large particle size | 50  |

Fabric finishing conditions: PVP molecular weight, 40000 Dalton; PVP or crosslinked PVP/SA weight ratio, 15%; APS/PVP weight ratio, 50 %; PVP or crosslinked PVP conc., 60%; reaction time, 45 min; reaction temp., 70 °C.

Synthesis reaction conditions: \*The fabric sample is padded in finishing bath containing 60 g/l DMDHEU as well as 60 g/l of the emulsion to a wet pick up of 100 %, dried at 100  $^{\circ}$ C/5 min then cured at 150  $^{\circ}$ C/3 min.

### 3.2. Factors affecting the preparation and performance of SA/PVP hybrids as water repellent finishes

Given below are the major factors affecting the preparation of SA/PVP hybrids along with the feasibility of utilization of their emulsions as water repellent finishes for cotton/polyester blended fabric.

### 3.2.1. APS/PVP weight ratio

Table 2 shows the effect of APS concentration on the emulsion state of the prepared hybrids as well as the water repellency rating of the treated fabric samples. It is clear that, for a given set of treatment conditions, increasing the APS concentration in the synthesis reaction medium up to 50 %, based on weight of PVP, is accompanied by an enhancement in both the emulsion state of hybrids as well as the WRR of the treated fabric that reaches to a maximum value of 50. The matter that could be attributed to increasing of free radical species in the reaction medium which in turn enhances the formation of SAPVP species and consequently improves both the emulsion state as well as the water repellency rating of treated fabrics<sup>6</sup>. Moreover, increasing of APS/PVP ratio beyond 50%, within the range studied, does not affect either the emulsion state or the WRR of

the treated fabric remaining it at the value 50 and suggesting that the higher APS/PVP ratio is accompanied by a rapid rate of termination<sup>16-19</sup>.

| Table 2: Effect of APS/PVP weight ratio on t | the emulsion state | and the water | repellency i | rating of tre | ated |
|--|--------------------|---------------|--------------|---------------|------|
| fabric.                                      |                    |               |              |               |      |

| APS/PVP (%) | Emulsion state                    | WRR |
|-------------|-----------------------------------|-----|
| 0           | No emulsion                       | 0   |
| 40          | No emulsion                       | 0   |
| 45          | No emulsion                       | 0   |
| 50          | Emulsion with large particle size | 50  |
| 60          | Emulsion with large particle size | 50  |

Synthesis reaction conditions: PVP molecular weight, 40000 Dalton; PVP/SA weight ratio, 20%; PVP conc., 50%; reaction temp., 80 °C; reaction time, 45 min.

Fabric finishing conditions: The fabric samples are padded in finishing bath containing 60 g/l DMDHEU as well as 60 g/l of the emulsion to a wet pick up of 100 %, dried at 100  $^{\circ}$ C for 5 min and then cured at 150  $^{\circ}$ C for 3 min.

### 3.2.2. PVP concentration

Table 3 reveals the effect of PVP concentration on the emulsion state of the prepared hybrids as well as the water repellency rating of their treated fabric samples. It is obvious that, increasing the concentration the PVP aqueous solution up to 60 % significantly enhances both the emulsion state as well as the WRR of treated fabric. This can be interpreted in terms of the availability of the polymer molecules in vicinity of the remaining reactants molecules that may increases the extent of formation of SAPVP species leading to an improvement in the emulsion state and consequently a higher WRR of treated fabric<sup>7,8</sup>. Further increase in PVP concentration, within the range studied, has practically no effect on the WRR of treated fabric.

# Table 2: Effect of APS/PVP weight ratio on the emulsion state and the water repellency rating of treated fabric.

| APS/PVP (%) | Emulsion state                    | WRR |
|-------------|-----------------------------------|-----|
| 0           | No emulsion                       | 0   |
| 40          | No emulsion                       | 0   |
| 45          | No emulsion                       | 0   |
| 50          | Emulsion with large particle size | 50  |
| 60          | Emulsion with large particle size | 50  |

Synthesis reaction conditions: PVP molecular weight, 40000 Dalton; PVP/SA weight ratio, 20%; PVP conc., 50%; reaction temp., 80 °C; reaction time, 45 min.

Fabric finishing conditions: The fabric samples are padded in finishing bath containing 60 g/l DMDHEU as well as 60 g/l of the emulsion to a wet pick up of 100 %, dried at 100  $^{\circ}$ C for 5 min and then cured at 150  $^{\circ}$ C for 3 min.

# Table 3: Effect of PVP concentration on the emulsion state and the water repellency rating of treated fabric.

| PVP conc. | Emulsion state                              | WRR |
|-----------|---|-----|
| (%)       |   |     |
| 40        | No emulsion                                 | 0   |
| 50        | Emulsion with large particle size           | 50  |
| 60        | Homogenous emulsion with fine particle size | 70  |
| 70        | Homogenous emulsion with fine particle size | 70  |

Synthesis reaction conditions: PVP molecular weight, 40000 Dalton; PVP/SA weight ratio, 20%; APS/PVP weight ratio, 50 %; reaction temp., 80 °C; reaction time, 45 min.

Fabric finishing conditions: The fabric samples are padded in finishing bath containing 60 g/l DMDHEU as well as 60 g/l of the emulsion to a wet pick up of 100 %, dried at 100  $^{\circ}$ C for 5 min and then cured at 150  $^{\circ}$ C for 3 min.

### 3.2.3. Reaction temperature

The effect of raising the synthesis reaction temperature on the emulsion state of the prepared hybrids and the WRR of their treated fabric samples is shown in Table 4. It is well seen that raising the reaction medium temperature from 60 °C to 90 °C significantly enhances the emulsion state of the prepared hybrids and thereby upgrades the WRR of treated fabric; a point which reflects the positive impact of temperature on the rate of initiation, initiator decomposition, i.e. more free radicals, and the mobility of reactants species in the reaction medium [16-19].

 Table 4: Effect of reaction temperature on the emulsion state and the water repellency rating of treated fabric.

| <b>Reaction temperature</b> | ction temperature Emulsion state            |    |
|-----------------------------|---|----|
| ( <sup>o</sup> C)           |   |    |
| 60                          | No emulsion                                 | 0  |
| 70                          | Emulsion with large particle size           | 50 |
| 80                          | Homogenous emulsion with fine particle size | 70 |
| 90                          | Homogenous emulsion with fine particle size | 80 |

Synthesis reaction conditions: PVP molecular weight, 40000 Dalton; PVP/SA weight ratio, 20%; PVP conc., APS/PVP weight ratio, 50 %; 60%; reaction time, 45 min.

Fabric finishing conditions: The fabric samples are padded in finishing bath containing 60 g/l DMDHEU as well as 60 g/l of the emulsion to a wet pick up of 100 %, dried at 100  $^{\circ}$ C for 5 min and then cured at 150  $^{\circ}$ C for 3 min.

| Reaction time<br>(min) | Emulsion state                              | WRR |
|------------------------|---|-----|
| 10                     | No emulsion                                 | 0   |
| 20                     | Emulsion with large particle size           | 50  |
| 30                     | Emulsion with large particle size           | 50  |
| 45                     | Homogenous emulsion with fine particle size | 70  |
| 55                     | Homogenous emulsion with fine particle size | 80  |
| 70                     | Homogenous emulsion with fine particle size | 80  |

Table 5: Effect of reaction time on the emulsion state and the water repellency rating of treated fabric.

Synthesis reaction conditions: PVP molecular weight, 40000 Dalton; PVP/SA weight ratio, 20%; PVP conc., 60%; APS/PVP weight ratio, 50 %; reaction temp., 80 °C.

Fabric finishing conditions: The fabric samples are padded in finishing bath containing 60 g/l DMDHEU as well as 60 g/l of mother emulsion to a wet pick up of 100 %, dried at 100  $^{\circ}$ C for 5 min and then cured at 150  $^{\circ}$ C for 3 min.

### 3.2.4. Reaction time

Table 5 shows the effect of prolonging of the synthesis reaction time on the emulsion state of the prepared hybrids as well as the WRR of their treated fabric samples. It is clear that increasing the reaction time from 10 to 55 min brings about an enhancement in the WRR of the treated fabric that reaches to 80, which is a direct result for the gradual improvement of the emulsion state of the prepared hybrids. The longer reaction time, within the range studied, has practically no effect on the emulsion state or WRR of the treated fabric. This could be explained by that during the initial stages of the reaction, 0 - 55 min, the PVP polymer chains as well as the initiator are present at higher concentrations in the reaction medium and under the molecular collision among these reactants the SA/PVP hybrid will be formed. Beyond 55 and up to 70 min, depletion in the

reactants content in the reaction medium will be occurred giving rise to the leveling off of the WRR of the treated fabric [16-19].

### 3.2.5. PVP molecular weight

# Table 6: Effect of PVP molecular weight on the emulsion state and the water repellency rating of treated fabric.

| PVP molecular weight<br>(Dalton) | Emulsion state                                 | WRR |
|----------------------------------|--|-----|
| 10000                            | No emulsion                                    | 0   |
| 30000                            | Emulsion with large particle size              | 70  |
| 40000                            | Homogenous emulsion with fine particle size No | 80  |
| 1,300,000                        | emulsion                                       | 0   |

Synthesis reaction conditions: PVP/SA weight ratio, 20%; PVP conc., 60%; APS/PVP weight ratio, 50 %; reaction temp., 80 °C; reaction time, 55 min.

Fabric finishing conditions: The fabric samples are padded in finishing bath containing 60 g/l DMDHEU as well as 60 g/l of the emulsion to a wet pick up of 100 %, dried at 100  $^{\circ}$ C for 5 min and then cured at 150  $^{\circ}$ C for 3 min.

Table 6 shows the effect of PVP molecular weight on the emulsion state of the prepared hybrids as well as the WRR of their treated fabric samples. The results reveal that, increasing the PVP molecular weight, up to 40000 Dalton, is accompanied by a substantial enhancement in both the emulsion state of the prepared hybrid as well as the WRR of treated fabrics. This may be associated with enhancing of the hydrophilic moieties of the SAPVP species which is responsible for emulsification of SA<sup>20</sup>. Moreover, increasing the polymer molecular weight to 1,300,000 Dalton worse impairs the water repellency of the treated fabric and downs it to zero probably due to increasing of the reaction medium viscosity that hinders molecular collisions of the reactants and consequently lowers the extent of formation of the SAPVP species<sup>21,4</sup>.

### **3.2.6. Emulsion concentration**

Figure 1 shows the effect of emulsion concentration in the finishing bath on the water repellency rating of treated fabric. Obviously, it is seen that increasing the emulsion concentration, 0 up to 60 g/l, results in a significant enhancement in the water repellence rating of the treated fabric. This is a direct result of coating the fabric with a low surface energy film of the solid ingredients of the hybrid emulsion as illustrated above by equations 4 and 5. Beyond the concentration of 60 and up to 80 g/l, a water repellency rating leveling off was observed.



Figure 1: Effect of emulsion concentration on water repellency rating of treated fabric.

Fabric finishing conditions: The fabric samples are padded in finishing bath containing 60 g/l DMDHEU as well as specific weight of the emulsion to a wet pick up of 100 %, dried at 100  $^{\circ}$ C for 5 min and then cured at 150  $^{\circ}$ C for 3 min.



Figure 2: Effect of DMDHEU concentration on water repellency rating of treated fabric.

Fabric finishing conditions: The fabric samples are padded in finishing bath containing 60 g/l of the emulsion as well as different concentrations of DMDHEU to a wet pick up of 100 %, dried at 100  $^{\circ}$ C for 5 min then cured at 150  $^{\circ}$ C for 3 min.

### 3.2.7. DMDHEU concentration

The effect of DMDHEU concentration, 0 - 80 g/l, on the WRR of treated fabric samples was monitored in Figure 2; exploiting the acidic properties of the finishing bath resulted from the decomposition of APS during the hybrid synthesis reaction as a catalyst. It is clear from Figure 2 that: i) treating fabric samples with finishing bath containing only the hybrid emulsion, i.e. in absence of DMDHEU, negatively affect the water repellency of that fabric, ii) increasing the DMDHEU concentration in the finishing bath from 25 to 60 g/l significantly enhances the WRR of the treated fabrics reflecting the role of DMDHEU in fixation of the hybrid ingredients onto the fabric surface (equations 4 and 5)<sup>22</sup>, and iii) further increasing of DMDHEU concentration, 60-80 g/l, practically does not affect the WRR of the treated fabric.

### 3.2.8. PVP/SA weight ratio

Upon alteration of the PVP/SA weight ratio, five different SA/PVP hybrids were prepared and their emulsions were designated by hybrids H1 - H5. Table 7 shows the emulsion state of that hybrids as well as some performance properties of their treated fabric samples. It is clear that increasing of PVP/SA weight ratio from 5 to 25 % in the finishing bath results in a significant improvement in the emulsion state of the prepared hybrids, water repellency rating and the surface smoothness of their treated fabric samples along with a noticeable increasing in the stiffness of that fabric samples. This could be related to the enhancement in the extent of formation of SAPVP species upon increasing of the PVP concentration that effectively results in coating the treated fabric surfaces with hydrophobic smooth film. Moreover, increasing stiffness of treated fabric may be attributed to the improvement in the deposited hydrophobic film onto the fabric surface and/or increasing of PVP concentration as a polymer additive in the finishing bath<sup>12,22</sup>. Table 7 depicts also that hybrids H4 and H5 are the most durable for washing that reaches up to 10 washing cycles.

| PVP/SA    | Hybrid      | Emulsion state                              | WRR            | SR    | S    |
|-----------|-------------|---|----------------|-------|------|
| (%)       | Designation |   |                | (µm)  | (mg) |
| Untreated | -           | -   | 0              | 17.65 | 498  |
| 5         | H1          | Emulsion with large particle size           | 50(0)(0)(0)    | 16.44 | 534  |
| 10        | H2          | Emulsion with large particle size           | 50(0)(0)(0)    | 16.05 | 640  |
| 15        | H3          | Homogenous emulsion with fine particle size | 70(50)(0)(0)   | 15.57 | 712  |
| 20        | H4          | Homogenous emulsion with fine particle size | 80(70)(50)(0)  | 15.12 | 748  |
| 25        | H5          | Homogenous emulsion with fine particle size | 80)(70)(50)(0) | 14.83 | 784  |

Table 7: Effect of PVP/SA weight ratio on water repellency rating, roughness and stiffness of treated fabric.

Synthesis reaction conditions: PVP molecular weight, 40000 Dalton; PVP conc., 60%; APS/PVP weight ratio, 50 %; reaction temp., 80 °C; reaction time, 55 min.

**Fabric finishing conditions:** The fabric samples are padded in finishing bath containing 60 g/l DMDHEU as well as 60 g/l of the emulsion to a wet pick up of 100 %, dried at 100 °C for 5 min and then cured at 150 °C for 3 min. WRR: water repellency rating, SR: surface roughness, S: stiffness. Values in parentheses indicate durability of treated fabric to washing after 5, 10, and 15 washing cycles.

Accordingly, it seems that the hybrid H4 is the recommended for imparting good water repelling properties for the cotton/cellulose blended fabric.

Furthermore, now work is underway to exploit TBHQ as antioxidant<sup>23</sup> beside other phenolic antioxidants organic compounds as multi-functional finishes to enhance storage life of the prepared hybrid H4 emulsion as well as upgrade other functional properties of water repellent fabric such as the antibacterial as well as UV blocking properties.

### 3.4. Characterization of SA/PVP hybrid H4 and its treated fabric

### 3.4.1. FTIR spectra

At optimum reaction conditions, a SA/PVP hybrid H4 was freshly prepared and then purified as mentioned in the experimental part to obtain Product 1 and Product 2. Both of these products were subjected to FTIR analysis and their FTIR spectra are represented by Figures 3 and 4 respectively. For comparison, FTIR analysis of the pure PVP (40000 Dalton) as well as SA were also performed and their spectra are represented by Figures 5 and also 4 respectively.



Figure 3: IR Spectrum of Product 1



Figure 4: IR Spectrum of Product 2 or stearyl alcohol.



### Figure 5: IR Spectrum of PVP.

Obviously, Figure 3 includes:

- 1) Peaks resembles to that of PVP spectrum such as:
- Peak at 3430 cm<sup>-1</sup> which is corresponding to hydroxyls from moisture as PVP is a hydrophilic polymer,
- Peak at 2922 cm<sup>-1</sup> which is assigned to the asymmetric stretching vibration of -CH in the skeletal chain of PVP,
- Peak at 1291 cm<sup>-1</sup> which is assigned to the vibration absorption of the C–N bond<sup>4,5</sup>, and
- Peak at 1640 cm<sup>-1</sup> which is corresponding to stretching vibration of C=O of PVP and crosslinked PVP  $_{8,14,24}$ .
- 2) Peaks similar to that of SA spectrum such as:
- Medium peaks at 2850 and 2918 cm<sup>-1</sup> which belong to aliphatic -CH stretching vibration.
- 3) Peaks appear in only the hybrid spectrum:
- Peak at 1116 cm<sup>-1</sup> which is corresponding to stretching vibration of ether bond (C-O-C aliphatic) confirming the formation of SA grafted PVP (SAPVP) and consequently the aforementioned tentative

mechanism. It seems that the SAPVP chains are bound to the crosslinked PVP matrix through the PVP part of SAPVP chain or by entanglement.

On the other hand, it is clearly shown that the FTIR spectrum of Product 2 (Figure 4) is identical to that of stearyl alcohol indicating that it is an unreacted stearyl alcohol.

### 3.4.2. TEM Image

The particles size of the emulsion of Hybrid H4 was determined by the transmission electron microscope (TEM) and represented by Figure 6. It is clear from Figure 6 that the emulsion particles are of size 10 to 50 nm.



Figure 6: TEM image of the Hybrid H3 emulsion.



(a) SEM of untreated cotton/PET (50/50%) fabric.

(b) SEM of treated cotton/PET (50/50%) fabric.



Figure 7: Scanning Electron Microscope images of (a) untreated cotton/PET blended fabric and (b) cotton/PET blended fabric treated with the PVP/SA hybrid emulsion.

### 3.4.3. Scanning Electron Microscope

Figure 7 shows the scanning electron microscope (SEM) micrograph of (a) untreated cotton/PET blended fabric and (b) cotton/PET blended fabric treated with finishing bath containing 60 g/l of the PVP/SA hybrid emulsion as well as 60 g/l of DMDHEU. It is well seen that, there is a homogenous deposited film of the hybrid ingredients onto the surface of the treated fabric (Fig. 7b) compared with that of the untreated sample (Fig. 7a).

### 4. Conclusions

- 1. A novel SA/PVP water repellent finish was synthesized by reacting of high concentrated aqueous solution of PVP with SA in presence of APS as initiator.
- 2. The optimum conditions to prepare the aforementioned hybrid are APS/PVP (50%), PVP concentration (60%), reaction time (55 min), reaction temperature (80°C), PVP molecular weight (40000 Dalton) and PVP/SA weight ratio (20%).
- 3. The prepared hybrid was characterized by investigation of its FTIR analysis after purification and the TEM image of the hybrid emulsion that shows that the emulsion particles are of size 10 to 50 nm.
- 4. Both the untreated as well as the hybrid emulsion treated fabric were characterized using Scanning Electron Microscope analysis.
- 5. Padding Cotton/polyester blended fabric in finishing bath containing 60 g/l of the prepared hybrid emulsion as well as 60 g/l of DMDHEU as a crosslinker followed by drying the padded at 100 °C for 5 min then curing at 150 °C for 3 min acquires the finished fabric water repellency rating of 70.

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