



New prospects in pretreatment of cotton fabrics using microwave heating



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ABSTRACT

As microwaves are known to give fast and rapid volume heating, the present study is undertaken to investigate the use of microwave heating for pretreatment cotton fabrics to reduce the pretreatment time, chemicals and water. The onset of the microwave heating technique on the physicochemical and performance properties of desized, scoured and bleached cotton fabric is elucidated and compared with those obtained on using conventional thermal heating. Combined one-step process for desizing, scouring and bleaching of cotton fabric under microwave heating was also investigated. The dual effect of adding urea, (as microwave absorber and hydrogen peroxide activator) has been exploiting to accelerate the pretreatment reaction of cotton fabric. DSC, FT-IR and SEM have been used to investigate the onset of microwave on the morphological and chemical change of cotton cellulose after pretreatment and bleaching under microwave heating. Results obtained show that, a complete fabric preparation was obtained in just 5 min on using microwave in pretreatments process and the fabric properties were comparable to those obtained in traditional pretreatment process which requires 2.5–3 h for completion.

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1. Introduction

Chemical pretreatment and bleaching of cotton based fabric may be broadly defined as a procedure mainly concerned with the removal of natural as well as added impurities in fabric to a level necessary for good whiteness and absorbency by utilizing minimum time, energy and chemical as well as water (Segal & Wakelyn, 1988, Karmakar, 1999).

Unless the fabric is uniform in whiteness, absorbency, chemical composition and has low levels of impurities, it is unlikely that it will take up dye or finish in a uniform way or to the maximum extent possible. Conventional pretreatments of woven cotton fabrics normally consists of three stages: desizing scouring and bleaching, for knitted cotton fabrics, pretreatment is a two stages process: scouring and bleaching (Segal & Wakelyn, 1988).

The idea of microwave (MW) application for textile finishing processes first originated in the 1970s when cellulose fabrics were treated with Durable Press (DP) finishing agents and cured in the microwave oven. Although these first results were promising, the idea was abandoned until 1995, when Miller patented his pre-set process without being aware of the earlier patent. Both cases involved garment microwave treatment, but they were abandoned

because the efforts to control the process failed (Hou, Wang, & Wu, 2008).

Microwave dielectric heating is based on activation of polar molecules in treated medium (polarization phenomenon). In a microwave electromagnetic field oscillating at 2.5 GHz, which is a preferred frequency for heating applications, the charge changes polarity nearly five billion times per second. Under the influence of a high frequency.

Microwave heating has been proved to be more rapid, uniform, efficient and easily penetrate to particle inside, few work has been mentioned in the literature describing the feasibility of application of microwave in desizing, scouring, bleaching, dyeing and finishing of cotton based fabric (Gacen & Cayuela, 2003; Haggag, El-Sayed, & Allam, 2007; Kale & Bhat, 2011; Zhao, Min, & He, 2011; Ibrahim, El-Sayed, & Ameen, 2010; Katovic, Vukusic, Grgac, Kovacevic, & Schwarz, 2009). However, the results obtained from these reports are variable and further lab scale research is necessary to optimize the application of microwave in pretreatments of cotton based textile in order to pave the way for rendering microwave heating technique commercially successful.

The present study is undertaken to investigate the advantage of microwave heating in desizing, scouring and bleaching (pretreatment) of cotton fabric. The onset of the microwave heating technique on the physicochemical and performance properties of treated cotton fabric will elucidate and compared with those obtained using conventional (currently used) thermal heating. The intention with the use of microwaves is to reduce the pretreatment

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time, chemical and water, as microwaves are known to give fast and rapid volume heating.

2. Experimental

2.1. Materials

2.1.1. Cotton fabric

Greige 100% woven cotton fabric (100 g/m²) was kindly supplied by Misr Company for Spinning and Weaving, Mehala El-Kubra, Egypt. Chemical analysis carried out on the greige fabric showed that the fabric warps were sized with starch-based sizing agent.

2.2. Chemicals

Sodium hydroxide, ammonium persulphate, urea and acetic acid were of laboratory grade chemicals. A non-ionic wetting agent based on an ethylene oxide condensate (Egyptol®), hydrogen peroxide (30% w/w), sodium silicate were of technical grade chemicals.

2.3. Desizing

In this research, padding technique was applied to affect desizing of cotton fabrics under microwave heating instead of conventional thermal heating. The experimental technique was adopted as follows:

Cotton fabrics were padded in aqueous solution containing, ammonium persulphate (3 g/L), Egyptol® (5 g/L), NaOH (2 g/L). The samples were then squeezed to a wet pick up of 100%. The samples were put in polyethylene packages then subjected to microwave heating in microwave oven for different times and different microwave powers. After desizing, the samples were washed several times with boiling water, then with cold water, and next dried in ambient conditions.

2.4. Scouring

Two techniques currently used by the industry were investigated for scouring the desized cotton fabric, exhaustion and pad-steam. In the exhaustion technique, 100 g of desized cotton fabric was treated with an aqueous solution containing NaOH (6 g/L), Egyptol® (2 g/L) using a material to liquor ratio (LR) of 1:30. The solution containing the fabric were introduced in specified beaker glass designed for our microwave oven, then the solution were heated with microwave under different power and for different time.

In the pad-steam technique, the samples were padded in an aqueous solution containing NaOH (50 g/L), Egyptol® (2 g/L), ionic detergent (1 g/L), then squeezed to a wet pick-up of 100%. The samples were introduced in polyethylene package and sealed to avoid dryness, then heated in the microwave oven.

After scouring by either of the two techniques, the samples were washed several times with boiling water and cold water, and finally dried in ambient conditions.

2.5. Bleaching

Similar to scouring, two techniques were used to affect bleaching, namely exhaustion and pad steam. In the exhaustion method, scoured cotton fabrics were treated with an aqueous solution containing H₂O₂, (8 g/L), sodium silicate (2 g/L), NaOH (2 g/L). A material to liquor ratio of 1:30 was used, and the bleaching process was carried out under different microwave power for different time. The fabric was then washed several times with boiling water, then with cold water and finally dried in ambient conditions.

In the pad-steam method, the fabric samples was padded in a solution containing H₂O₂ (10 g/L) and sodium silicate (5 g/L), NaOH, (2 g/L). The fabric was then squeezed to a wet pick-up of 100%. The samples were then introduced in polyethylene package and sealed to avoid dryness then heated in the microwave oven. After bleaching, the sample was washed several times with hot water, then with cold water, and finally dried in ambient conditions.

2.6. One-step process for desizing, scouring and bleaching of cotton fabrics under microwave heating

In this report, we have investigated only pad-steam process for combined desizing scouring and bleaching of cotton fabric. The experiment techniques was adopted as follows: grey 100% cotton fabric was padded in aqueous solution containing NaOH, (50 g/L), Egyptol®, (10 g/L), H₂O₂ (10 g/L) then squeezed to a wet pick up 100% and put in sealed polyethylene package. The fabric was subjected to microwave heated at different power and for different time. After the treatment time, the sample was washed several times with boiling water, then with cold water, and finally dried at ambient conditions.

2.7. Testing and analysis

Loss in fabric weight was expressed as the percentage with respect to the initial weight of the fabric.

Water absorbency was monitored according to an AATCC Test Method 39–1980 (Evaluation of Wettability) (AATCC, 1980). The time (in seconds) between the contact of water drop with the fabric and the disappearance of the water drop into the fabric called wetting time. The shorter the wetting time, the better the fabric absorbency.

Tensile strength was determined by the strip method according to ASTM, Standard Test Method “Breaking Load and Elongation of Textile Fabric”, D-1682-94 (1994).

Whiteness was evaluated with a Color-Eye 3100 Spectrophotometer from SDL Inter.

Residual starch after desizing was evaluated using iodine spot test.

FTIR: Perkin Elmer, system 2000FT-IR-USA.

DSC: Differential scanning calorimeter—Shimadzu-50—USA.

SEM: Samples mounted on aluminum stubs, and sputter coated with gold in a 150 Å sputter (Coated Edwards), and examined by Jeol (JXA-840A) Electron Probe Microanalysis (Japan), magnification range 35–10,000, accelerating voltage 19 kV.

3. Results and discussion

3.1. Desizing of cotton fabric using microwave heating

100% grey cotton fabrics were desized with ammonium persulphate under the effect of microwave heating as alternative eco-friendly thermal heating. The treated fabrics were evaluated for loss in fabric weight and residual starch using iodine test. Results obtained are set out in Table 1.

Results obtained from Table 1 indicate that, approximately complete removal of starch sizing agent from the cotton fabric can be obtained after heating the treated samples in microwave oven at power 800 W for 2 min. A similar result are obtained with conventional heating using the same concentrations from the reagent but for 45 min (Segal & Wakelyn, 1988).

3.2. Scouring of cotton fabric using microwave heating

The loom state cotton fabric contains about 8–12% natural impurities of total weight of the fibre. These impurities mainly consist

Table 1

Effect of microwave time and power on loss of fabric weight after desizing using padding techniques.

| Microwave power (W) | Loss in fabric weight (%) (residual starch) | | | |
|---------------------|---|---------|---------|---------|
| | 1 min | 2 min | 3 min | 4 min |
| 20 | 7.7 (++) | 8.5 (+) | 8.6 (+) | 8.7 (–) |
| 40 | 8.4 (+) | 8.0 (+) | 8.5 (–) | 8.4 (–) |
| 60 | 8.5 (+) | 7.8 (–) | 7.9 (–) | 8.0 (–) |
| 80 | 8.7 (+) | 8.9 (–) | 8.9 (–) | 8.1 (–) |
| 100 | 8.0 (+) | 7.9 (–) | 8.0 (–) | 8.2 (–) |

Conditions used: Grey 100% cotton fabrics were padded in aqueous solution containing: ammonium persulphate, 3 g/L; NaOH, 2 g/L; Egyptol®, 5 g/L, squeezing to 100% wet pick up.

(++) high amount of residual starch (–) no residual starch.

of waxes, proteins, pectic substances and mineral matters (Segal & Wakelyn, 1988, Karmakar, 1999). In addition to this, the mechanically held impurity called 'motes' are present containing seed-coat fragments, aborted seeds and leaves etc., that cling to the fibre.

Scouring is a purifying treatment of textiles. The scouring process consists of an alkali treatment in the presence of wetting agent. The objective of scouring is to reduce the amount of impurities sufficiently to obtain level and reproducible results in dyeing and finishing operations. It may be said that good scouring is the foundation of successful finishing (Segal & Wakelyn, 1988, Karmakar, 1999).

Two techniques currently used by the industry were investigated for scouring the desized cotton fabric, exhaustion and pad-steam. The efficacy of scouring process was determined by measuring the loss in fabric weight and wettability after scouring. Results obtained are set out in Table 2.

It could be emphasized from Table 2 that, on using pad-steam technique, a complete wax and impurities removal are obtained when the desized cotton fabric were scoured with 50 g/L NaOH then heated under the effect of microwave for 4 min. This is evidenced by higher water absorbability of the scoured fabrics (4 s).

Table 2

Effect of microwave time and power on loss in weight and wettability of fabric after scouring using exhaustion or pad-steam technique.

| Microwave power (W) | Loss in fabric weight (%) wettability (s) | | | | | | | |
|---------------------|---|---------|---------|---------|---------------------|---------|---------|---------|
| | Using exhaustion technique | | | | Pad-steam technique | | | |
| | 1 min | 2 min | 3 min | 4 min | 1 min | 2 min | 3 min | 4 min |
| 20 | 4 (12) | 5 (10) | 5 (9) | 5 (5) | 3.5 (9) | 4 (7) | 4.1 (6) | 4.5 (3) |
| 40 | 3.4 (10) | 4.5 (9) | 4.7 (7) | 4.8 (4) | 3.7 (8) | 4.2 (7) | 4.5 (5) | 4.6 (1) |
| 60 | 4.0 (9) | 4.1 (6) | 4.2 (4) | 4.4 (2) | 4.0 (4) | 4.1 (5) | 4.2 (2) | 4.4 (2) |
| 80 | 4.5 (6) | 4.7 (5) | 5.0 (4) | 5.4 (2) | 4.5 (3) | 4.7 (3) | 5.0 (2) | 5.4 (2) |
| 100 | 4.4 (5) | 4.6 (4) | 4.7 (4) | 4.9 (1) | 4.4 (3) | 4.6 (3) | 4.7 (2) | 4.9 (1) |

Conditions used: Grey 100% cotton fabrics were padded in aqueous solution containing: NaOH, Egyptol®, 2 g/L, 1 g/L ionic detergent, squeezing to 100% wet pick up. Values in brackets represent wettability time of the fabric (s).

Table 3

Effect of microwave time and power on whiteness index of the 100% cotton fabric after bleaching using pad-steam techniques.

| Microwave power (W) | Whiteness index | | | | | | | |
|---------------------|----------------------------|-------|-------|-------|---------------------|-------|-------|-------|
| | Using exhaustion technique | | | | Pad-steam technique | | | |
| | 1 min | 2 min | 3 min | 4 min | 1 min | 2 min | 3 min | 4 min |
| 20 | 40 | 43 | 45 | 45 | 52 | 52 | 58 | 58 |
| 40 | 47 | 45 | 49 | 49 | 52 | 56 | 60 | 63 |
| 60 | 47 | 51 | 55 | 52 | 57 | 60 | 61 | 65 |
| 80 | 46 | 52 | 55 | 55 | 60 | 62 | 64 | 65 |
| 100 | 48 | 54 | 55 | 60 | 60 | 63 | 64 | 67 |

WI grey cotton fabric (Blank) = 21.06.

Conditions used: See Section 2.

Generally and under a conventional steaming, a similar scouring efficacy is obtained after 2 h (Karmakar, 1999).

3.3. Bleaching of cotton fabric using microwave heating

The purpose of bleaching is to destroy this colored material and to confer a pure white appearance to the fibers. Bleaching should also decolorize or remove any residual impurities left by scouring. Bleaching is the linchpin of preparation and today really means bleaching with hydrogen peroxide (peroxide) (Karmakar, 1999).

Table 3 shows the whiteness index of cotton fabrics after bleaching using pad steam technique and under microwave heating. Results of Table 3 make it clear that the fabric whiteness index (WI) increase as the microwave power and the exposure time increase. A good WI is obtained after 3 min at 800 W microwave power. A comparable result is obtained when the scoured cotton fabric was bleached under a conventional steaming process but for much longer time (60 min) (Segal & Wakelyn, 1988).

3.4. One-step process for combined desizing, scouring and bleaching process under microwave heating

Now-a-days all efforts in the field of pre-treatment processes of textiles are directed towards shortening and simplification of the treatment (Segal & Wakelyn, 1988). In the conventional preparation, the desizing, scouring and bleaching processes are carried out separately at high temperatures, requiring the use of large amount of thermal energy. In order to minimize energy consumption it has become necessary to combine several pre-treatment stages or by shortening the pretreatment time.

Table 4 shows the relation between the microwave power and time on the loss of the fabric weight, wettability and WI, respectively, when the three processes were combined in one-step process and carried out using microwave heating. The results indicated that, an accepted fabric performance were obtained when the three steps were combined in one step process.

Table 4

Effect of microwave time and power on loss in fabric weight, wettability and whiteness index of the cotton fabric after one-step desizing, scouring and bleaching using pad-steam techniques.

| Microwave power (W) | Parameters | Microwave exposure time | | | |
|---------------------|-----------------|-------------------------|-------|-------|-------|
| | | 1 min | 2 min | 3 min | 4 min |
| 40 | Loss weight (%) | 10.8 | 10.2 | 11.5 | 12.1 |
| | Wettability (s) | 4 | 3 | 2 | 2 |
| | Whiteness index | 43 | 49 | 54 | 56 |
| 60 | Loss weight (%) | 12.4 | 11.9 | 11.4 | 11.6 |
| | Wettability (s) | 3 | 2 | 1 | 1 |
| | Whiteness index | 45 | 51 | 55 | 59 |
| 80 | Loss weight (%) | 11.2 | 11.6 | 12.6 | 12.4 |
| | Wettability (s) | 2 | 2 | 1 | 1 |
| | Whiteness index | 52 | 52 | 62 | 60 |
| 100 | Loss weight (%) | 12.1 | 12.1 | 12.9 | 12.0 |
| | Wettability (s) | 1 | 1 | 1 | 1 |
| | Whiteness index | 52 | 55 | 62 | 65 |

WI grey cotton fabric (Blank) = 21.06.

Conditions used: Grey 100% cotton fabrics were padded in aqueous solution containing: NaOH, 50 g/L, H₂O₂ 10 g/L, Egyptol®, 10 g/L, squeezing to 100% wet pick up.

3.5. Effect of addition of microwave absorption in pretreatment bath

The addition of polar salts (microwave absorber) to the solvent, usually leads to higher heating rates (microwave radiation absorption) for the whole mixture and the energy transfer for whole reaction is rapid, these salts should have good solubility characters to produce homogeneous mixture. In microwave-assisted synthesis, a homogeneous mixture is preferred to obtain a uniform heating pattern. For examples, Urea has C–O and C–N covalent bonds. These bonds are somewhat polar with the most polar bond being C–O (oxygen is more electronegative than N and C). Moreover, Urea is highly soluble in water (around 100 g in 100 mL of water at room temperature) so, it can use successfully as microwave absorber. It has been mention elsewhere that urea acts as peroxide activator during the peroxide bleaching process (Segal & Wakelyn, 1988).

With the above in mind, the dual effect of adding urea, (as microwave absorber and hydrogen peroxide activator) has been exploit to accelerate the pretreatment reaction of cotton fabric. The study will be carried out only with one-step process for desizing scouring and bleaching using pad-steam technique. Results obtained are set out in Table 5.

3.6. Characterization of the pretreated fabrics

3.6.1. DSC and TGA

The thermal behavior (DSC and TGA) of grey, desized, scoured and bleached cotton fabrics were analyzed in nitrogen atmosphere in a temperature domain of 25–500 °C are shown in Figs. 1–4, respectively. The initial weight loss amounted 8–8.4% for all four substrates are attributed to the loss in fabric humidity. The major

Table 5

Effect of microwave time on properties of cotton fabrics after desizing, scouring and bleaching in presence of urea using pad-steam technique.

| Microwave time (min) | Wettability (s) | Whiteness index | Tensile strength (N) | Elongation at break (%) |
|----------------------|-----------------|-----------------|----------------------|-------------------------|
| 1 | 3 | 57 | 70 | 22 |
| 2 | 2 | 65 | 70 | 20 |
| 3 | 2 | 68 | 70 | 22 |
| 4 | 2 | 72 | 60 | 19 |

MW power: 720 W (60% from total power).

Conditions used: NaOH, 4 g/L, H₂O₂; sod silicate, 5 g/L; Egyptol®, 8 g/L; 5 g/L; urea, 5 g/L, steaming in sealed polyethylene package*, power of MW adjusted at 60%.

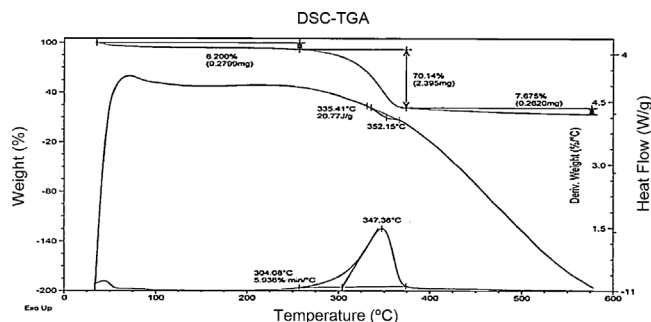


Fig. 1. DSC-TGA of 100% cotton fabric (grey fabric).

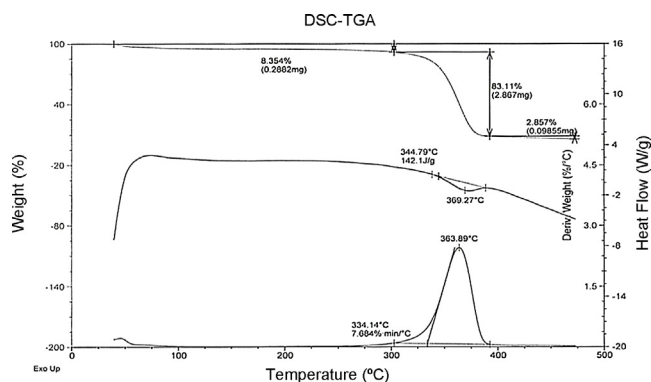


Fig. 2. DSC-TGA of desized 100% cotton fabric using exhaustion technique.

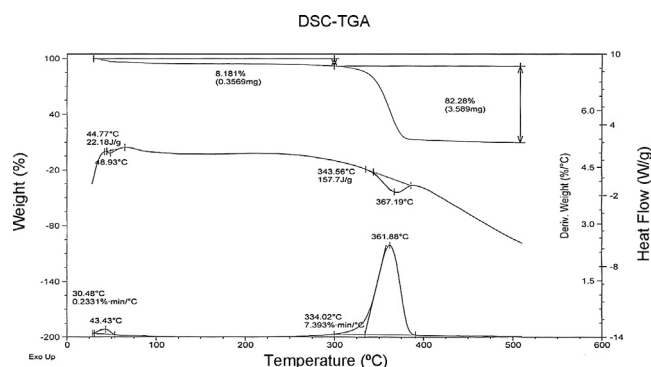


Fig. 3. DSC-TGA of scoured 100% cotton fabric using exhaustion technique.

weight-loss processes starts at 250 °C for the grey cotton fabric (insert in Fig. 1) the maximum weight reduction is observed at 363.9 °C and amounted 70%. At temperatures as high as 375 °C the residual weight of non-decomposed substrate present in the system was 7.0%. Similar results are obtained with desized, scoured

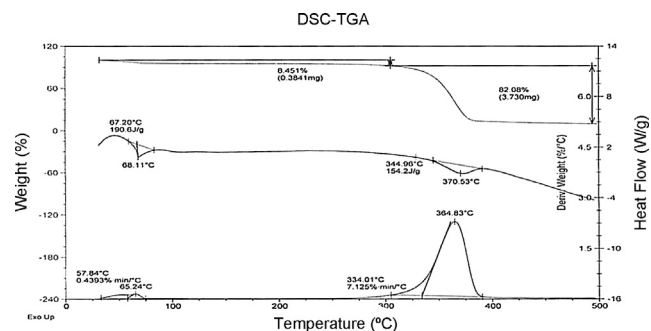


Fig. 4. DSC-TGA of bleached 100% cotton fabric using exhaustion technique.

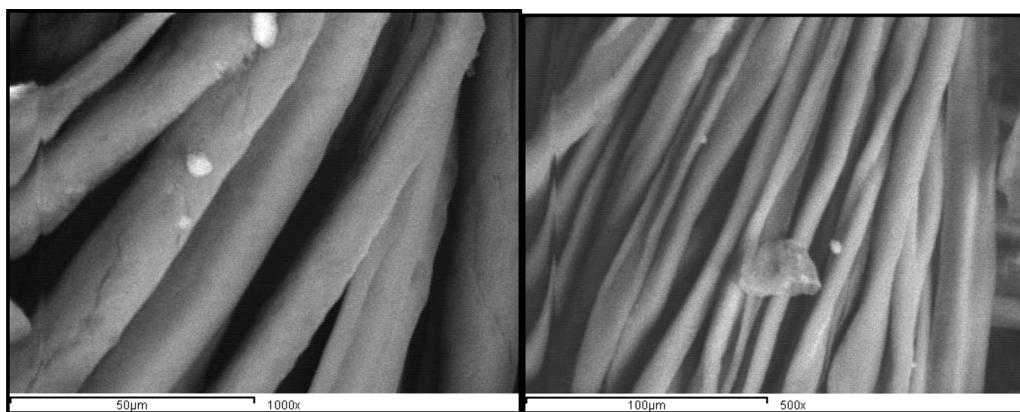


Fig. 5. SEM of untreated 100% grey cotton fabric.

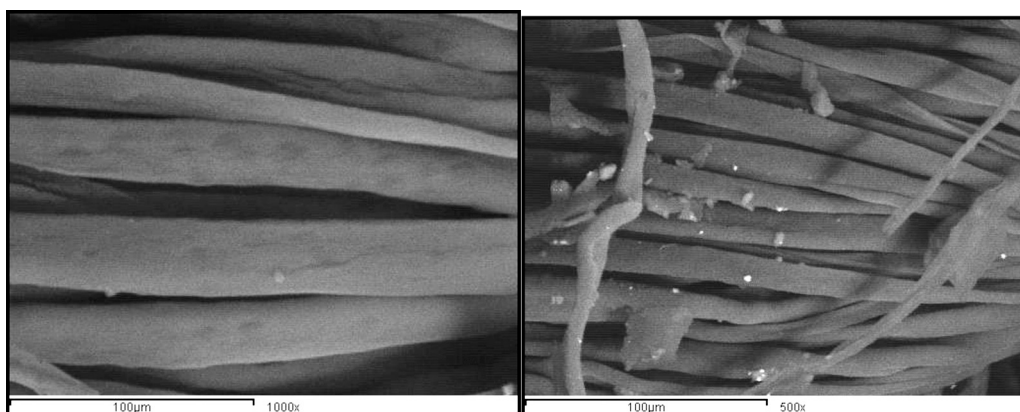


Fig. 6. SEM of desized cotton fabric under microwave heating using exhaustion technique.

and bleached cotton fabrics (Figs. 2–4, respectively). The figures confirm that, cellulose polymer remain approximately unaltered after pretreatment. This is evidenced by similar peak shape and position in DSC chart.

3.6.2. SEM of pretreated fabric

Figs. 5–8 show the scanning electron micrograph (SEM) of grey cotton, desized scoured and bleached cotton fabric, respectively. Fig. 9 shows SEM micrograph of cotton fabric after one-step desizing, scouring and bleaching under microwave heating.

Examination of SEM micrographs of all substrates show typical fibers with twisted smooth ridges and smooth surface

characteristic whereas concave grooves are still appear. The micrograph confirm that, cellulose micro fibril remain approximately unaltered after pretreatment. This is in accordance with the results previously reported (Hou et al., 2008).

3.6.3. FTIR of cotton fabrics after pretreatments under microwave heating using exhaustion technique

Figs. 10–13 shows FTIR analysis of cotton fabrics before and after pretreatment and bleaching under microwave heating. Peaks assignment are set out in Table 6. Results of Figs. 10–13 and Table 6 show a typical cellulose chart with no over-oxidation peak (at

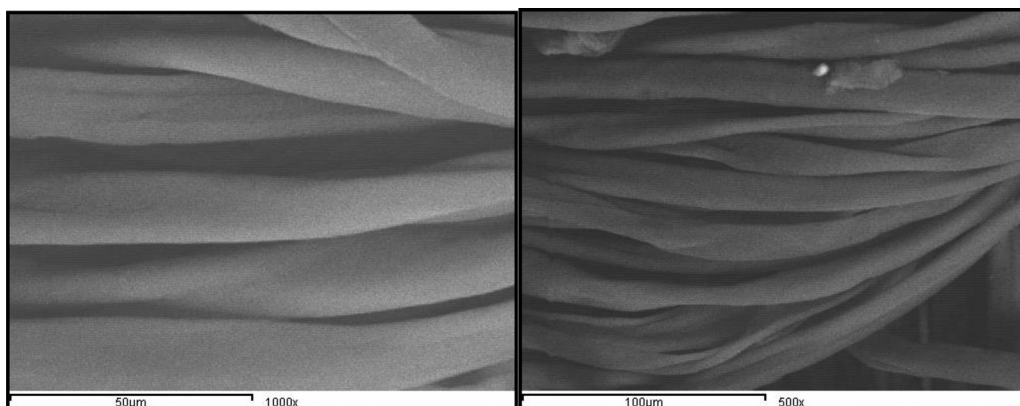


Fig. 7. SEM of scoured cotton fabric under microwave heating using exhaustion technique.

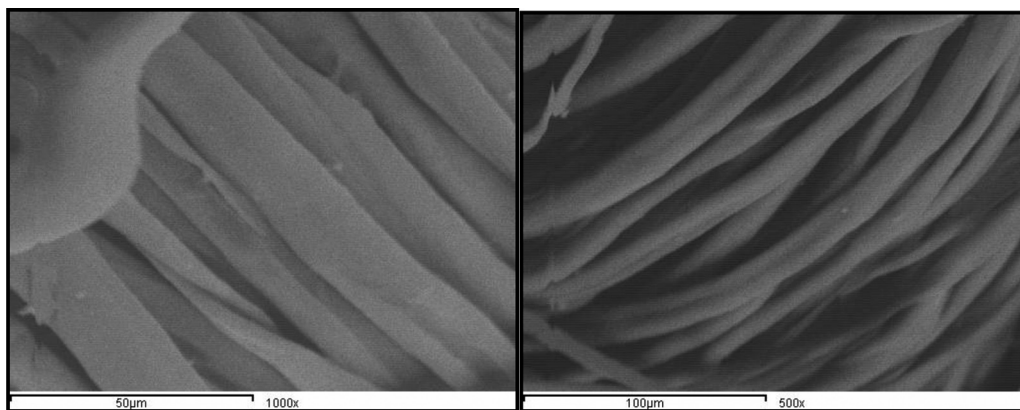


Fig. 8. SEM of bleached cotton fabric under microwave heating using exhaustion technique.

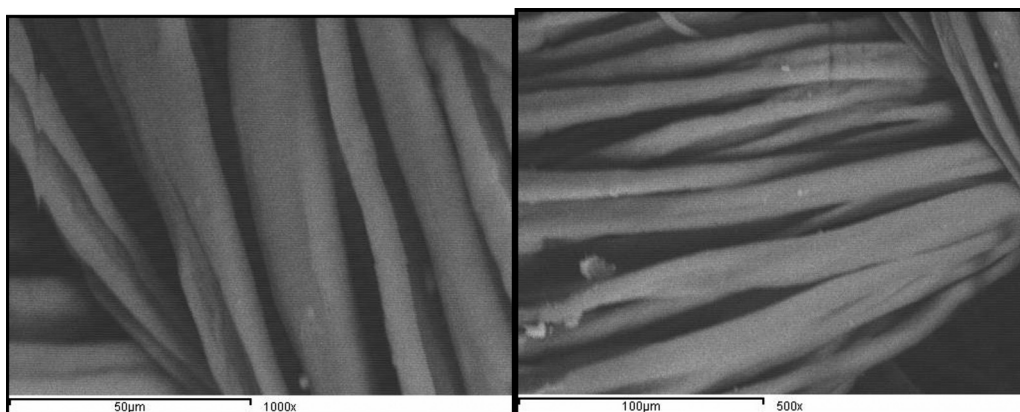


Fig. 9. SEM of cotton fabric after one-step desizing, scouring and bleaching under microwave heating using exhaustion technique.

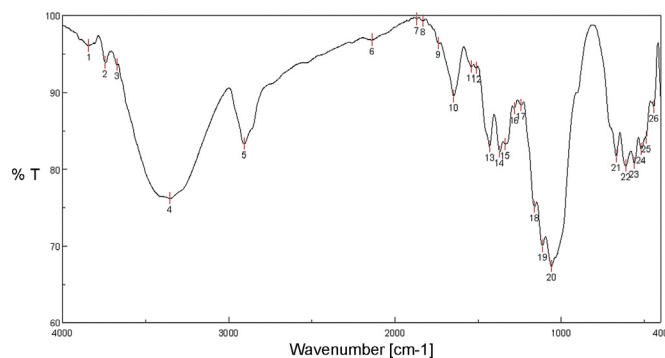


Fig. 10. FTIR of desized cotton fabric under microwave heating using exhaustion technique.

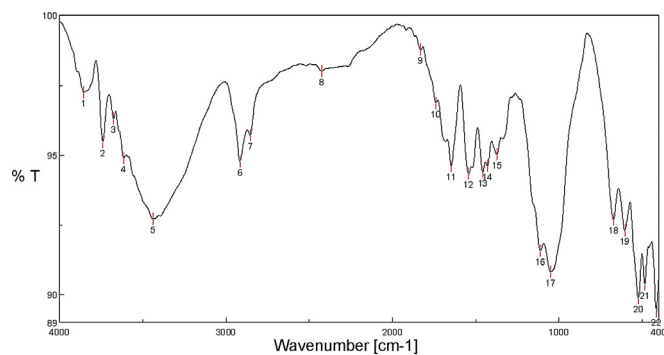


Fig. 11. FTIR of scoured cotton fabric under microwave heating using exhaustion technique.

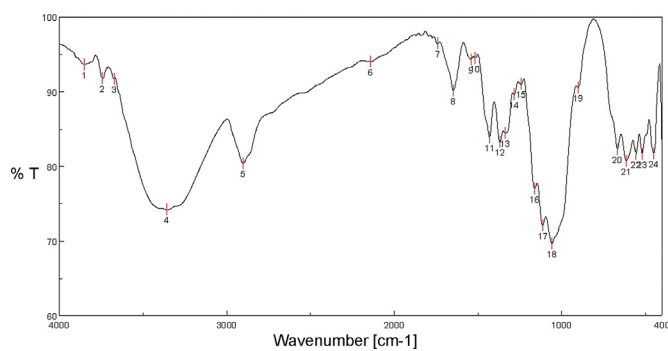


Fig. 12. FTIR of bleached cotton fabric under microwave heating using exhaustion technique.

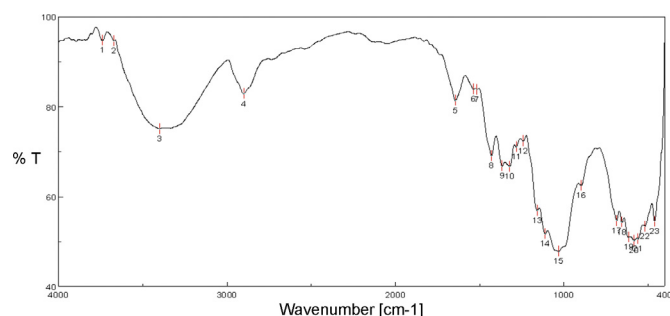


Fig. 13. FTIR of cotton fabric after one-step process for desizing scouring and bleaching under microwave heating using exhaustion.

Table 6
Peaks assignment of cotton fabrics.

| Peak (cm ⁻¹) | Assignment |
|--------------------------|--|
| 3650 | Carboxylic –OH |
| 3330 | Alcoholic –OH stretching |
| 2900 | Aliphatic –CH ₂ stretching |
| 1650 | Absorbed water and hydrogen bond |
| 1428 | Aliphatic –CH ₂ bending |
| 1367 | –CH bending (deformation stretching) |
| 1338 | –OH in plan bending |
| 1316 | –CH wagging |
| 1161 | –C–O–C–asymmetric bridge stretching |
| 1110 | –C–O–H bending of secondary alcoholic |
| 1057 | Asymmetric in plan ring stretching |
| 1033 | –C–O stretching |
| 902 | Asymmetric out-of-phase ring stretch –C ₁ –O–C ₄ β-glucosidic bond |

~2500 cm⁻¹) was observed after pretreatment under microwave heating (Zhbarkov, 1966).

4. Conclusion

Grey cotton fabric was desized, scoured and bleached under microwave heating as green alternative to conventional thermal heating. The treatments were carried out as per exhaustion or pad batch techniques. Combined one-step process for bleaching of grey cotton fabric was also investigated under microwave heating. The

onset of the microwave heating technique on the physicochemical and performance properties of treated cotton fabric will elucidate and compared with those obtained using conventional (currently used) thermal heating. Results obtained show that, a complete fabric preparation was obtained in just 5 min on using microwave in pretreatments process and the fabric properties were comparable to those obtained in traditional pretreatment process which takes 2.5–3 h for completion. TGA–DSC, SEM and FTIR analysis confirmed that, cellulose micro fibril remain approximately unaltered after pretreatment under microwave heating.

Based on the above results, it could be emphasized that, microwave heating is a promising green alternative tool can be used pretreatment cotton fabrics.

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