

## Research Article

# A Modified Hydrolysis Method of Decolorizing Reactive-Dyed Polycotton Waste Fabric and Extraction of Terephthalic Acid: A Perspective to Reduce Textile Solid Waste

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To manage the polycotton textile-waste fabric (PCWF), a modified alkaline hydrolysis method is used for decolorization and separation of polyester as terephthalic acid (TPA). The effects of optimum conditions on TPA yield (%) have been determined to be  $97.66 \pm 1.94\%$ . Dye degradations and K/S values are measured by UV-visible spectrophotometer. K/S value of PCWF is 37.06 and separated cotton fabric (SCF) is 0.035, respectively. The chemical functionalities and crystallinity of PCWF, SCF, and TPA are determined by using FTIR and XRD. FTIR peak values are  $1684\text{ cm}^{-1}$ ,  $1574\text{ cm}^{-1}$ ,  $1512\text{ cm}^{-1}$ ,  $1280\text{ cm}^{-1}$ , and  $1425\text{ cm}^{-1}$  that prove transformation of polyester to TPA. XRD peaks confirm polyester conversion to TPA, and the values are 17.4, 25.13, 28.12, 29.09, and 38.7. TGA, SEM, and EDX data showed the thermal stability, morphology, and elemental composition of TPA.

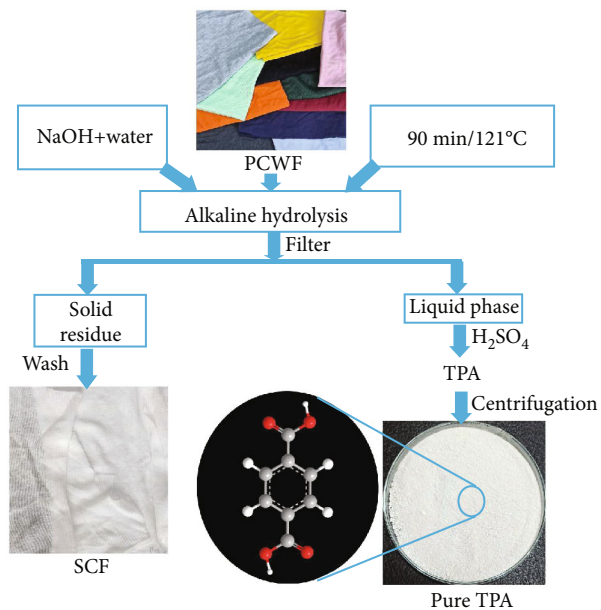
## 1. Introduction

Clothing is the crucial substitute for basic human needs. In this modern world, it has become a fundamental matter of personal taste, fashion, and style representing a person and his personality. This thought results in the overconsumption of clothing. According to the statistics, by 2017, the fiber used worldwide was almost 105 million tons, where synthetic fibers were reported 64.2%. Most of these fibers were approximately polyester 80% and cotton 24.1% [1]. An estimation suggests that by 2025, total fiber production will roughly surpass 100 million tons, and the consumption will be based on the increase of world population [2].

In Bangladesh, the garment sector has become an essential part of the economy. More than 6500 garment industries play a significant role in the average growth rate of Bangla-

desh. But when it comes to the environment, these industries have been recognized as major solid waste producers [3]. These industries generate about 120 to 125 tons of waste [4]. This enormous amount of waste causes environmental pollution and a waste of valuable resources. It was calculated that about 7 million tons of textile waste were thrown away in landfills [5]. The extensive amount of textile waste is generally blended fibers. Blending is a common term in textile industries because blending extends the performance and aesthetics, and most importantly, reduces the cost of production compared to monofibers [6]. Polyester-cotton, which is known as polycotton, is the most common fiber [7].

Cotton is a natural linear cellulosic fiber that contributes to softness and high-water absorbency [8]. The demand for cotton is increasing day by day, but the production of virgin cotton is not increased as much [9]. Thus, scientists are now



SCHEME 1: Synthesis of TPA and recycle of SCF from PCWF.

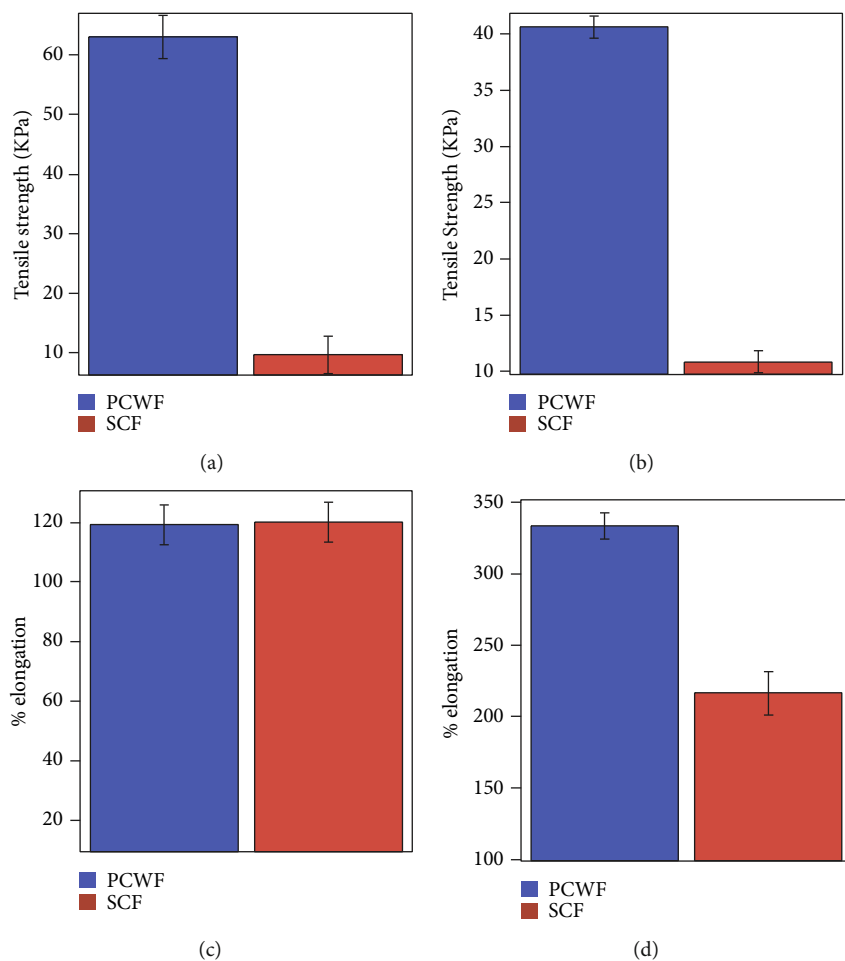


FIGURE 1: Tensile strength of PCWF and SCF towards warp (a) and weft (b), and % elongation of PCWF and SCF towards warp (c) and weft (d).

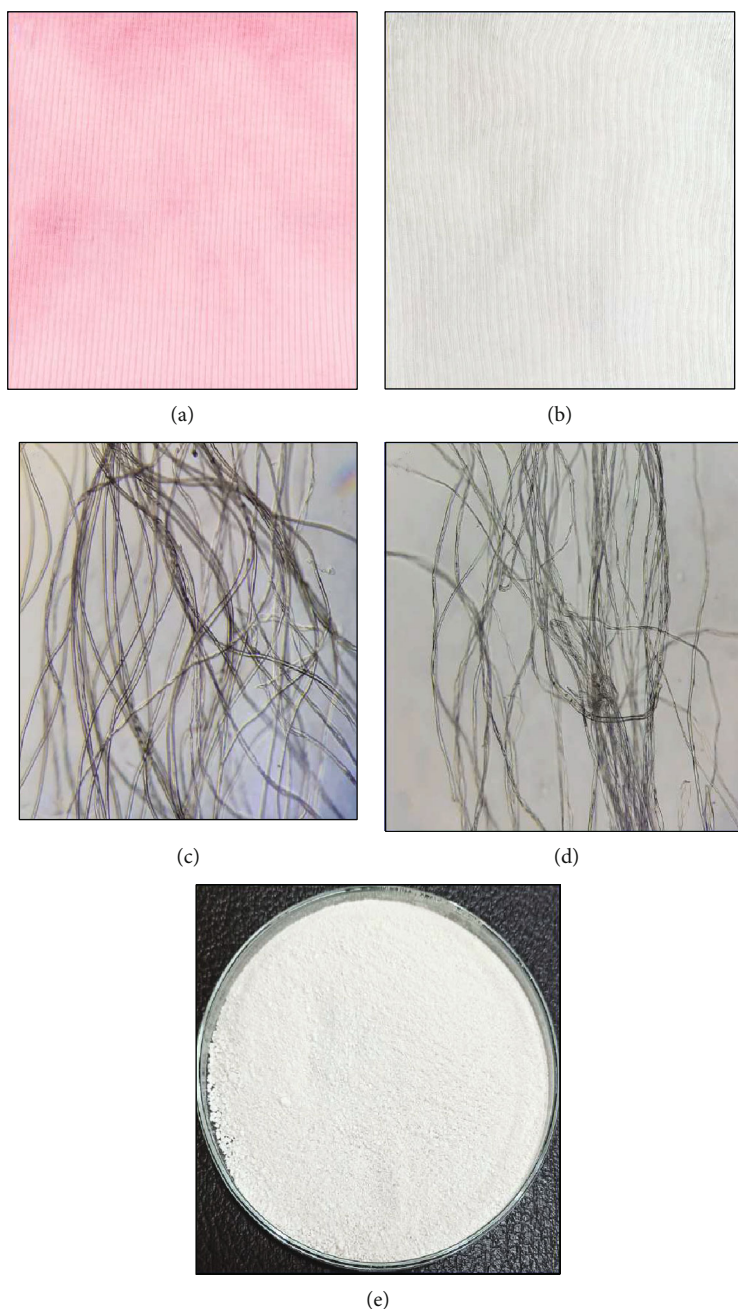


FIGURE 2: (a) PCWF before hydrolysis. (b) SCF after hydrolysis. (c) Microscopic view of PCWF before hydrolysis. (d) Microscopic view of SCF after hydrolysis and (e) TPA.

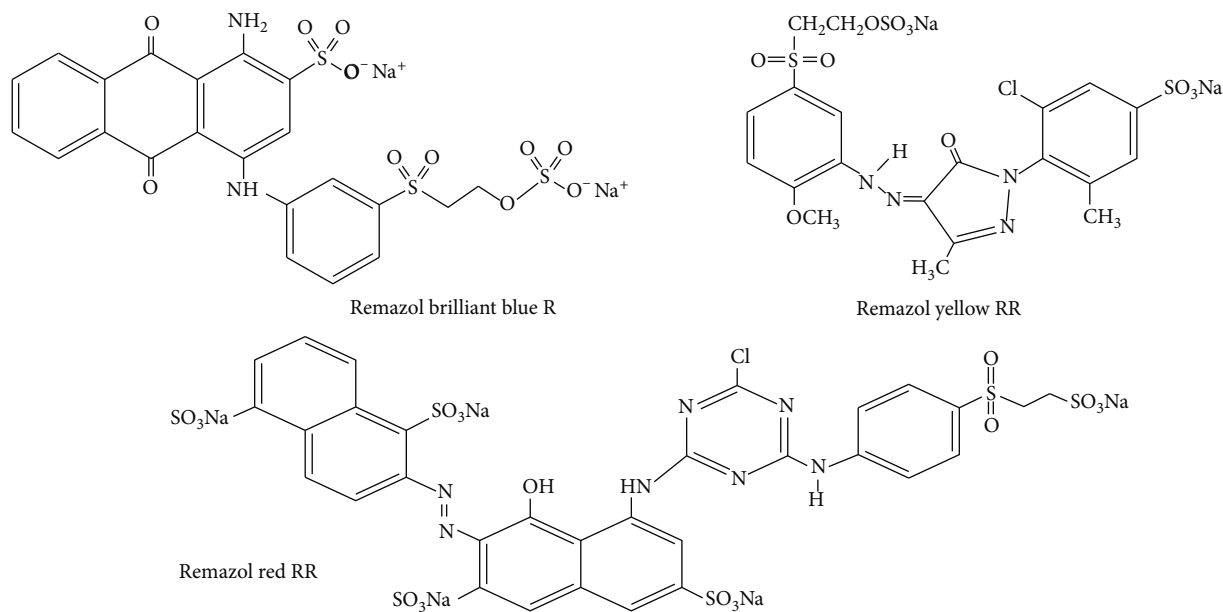
focusing more on recycling cotton. It has so many uses, such as different types of cellulosic fiber-based yarn like lyocell, viscose, rayon [10], CMC [11], and composite for false ceiling-based application [12].

On the other hand, polyester is an artificial synthetic fiber. It has advanced thermal stability, good chemical resistance, and tensile strength so that it provides long life, low cost, and good strength [13]; because of these advantages, it is used widely, and these uncontrolled uses make a threat to the clean environment. Polycotton is not like natural fibers. That is why it is not easily degraded on the earth. As per statistics, throw-

ing landfills can degrade only 20% of polyester [14]. Hence, recycling polyester became very important to save the environment and the resources of petroleum [15].

Polyester or polyethylene terephthalate (PET) is a long-chained semicrystalline, the thermoplastic polymer produced from petroleum-based chemicals. It is referred to as PET in packaging industries and textiles as polyester [16]. It has numerous uses for its lightweight and mechanical strength, e.g., textile fibers, X-ray and video films, and tire cords [17].

There are four prime polymer-recycling classifications: polyester-primary recycling or preconsumer industrial



SCHEME 2: Chemical structure of dyes.

scrap, secondary or mechanical recycling, tertiary or chemical recycling, and quaternary or energy recovery recycling. Among these recycling processes, the most acceptable and sustainable procedure for polyester is chemical recycling. In the chemical recycling process, some substances are used to split the ester groups of polyester [18]. These reagents differ from the procedure like glycols for glycolysis, alcohol for alcoholysis, aqueous solution for hydrolysis, and amines for aminolysis. In every chemical recycling process, one part is standard; when depolymerizing or dissolving one of the blended portions, others will be maintained [8].

It is so much easier to recycle polyester and cotton separately. But when blended, they create a rigid integration. As a result, it becomes formidable. Researchers always search for easy and significant methods—hydrolysis is one of them [6]. Hydrolysis (acid, neutral, and alkaline hydrolysis) of polyester is one of the chemical-recycling methods where terephthalic acid (TPA) and ethylene glycol (EG) were produced [19]. In this process, ester bonds of polyester are broken down by the nucleophilic attack of hydroxide ions and produce disodium terephthalate (Na-TPA) and EG. Both are soluble in water. But when the pH of the solution turned around 2-3 with sulfuric acid, the yield formed as the precipitation of TPA [20]. Generally, a phase-transfer catalyst can speed up the reaction [21]. Sun et al. hydrolyzed polyester with glycol and sodium bicarbonate as catalysts, and Li et al. used self-made SO<sub>4</sub><sup>2-</sup>/TiO<sub>2</sub> solid superacid catalyst [22, 23]. However, in this research work, no catalyst was used.

TPA with the chemical formula C<sub>8</sub>H<sub>6</sub>O<sub>4</sub> is a monomer of polyester. It was first synthesized in 1847 from oxidation, but this process was very challenging [24]. Due to this problem before the 1940s, dimethyl terephthalate (DMT) was used to synthesize polyester. After synthesizing polyester successfully from terephthalic acid and ethylene glycol

(EG) in the 1940s, TPA was used in the textile industry for fiber production [25]. In 1960, for the first time, TPA was available in the market; and since then, the polycondensation reaction between TPA and EG has played a significant role in polyester synthesis [26]. TPA has other uses in manufacturing industries, e.g., polyester fibers (used in apparel, carpet yarns, fill fibers, etc.), polyester films (used in photography, magnetic tapes, etc.), resins (used in automobile parts), and industrial filaments [27].

Polyester was separated from waste textile polycotton-blended fabric using previous techniques. It was worked with power ultrasound to separate the polycotton-blended fabric [28]. Another polycotton-blended fabric separation work done by Hou et al. and Ling et al. used hydrochloric acid, phosphotungstic acid, and catalysts which cause extra cost [29, 30].

Besides, to recrystallize the recovered TPA for further purification, various organic solvents such as pyridine and DMF are used. These organic solvents can generate byproducts that are harmful to the environment. As a result, these processes are technically and economically not suitable. On the other hand, it is critical to recovering-purified TPA using the activated carbon adsorption method [31]. Even without any pretreatment of raw materials or any catalyst, it is tough to reduce reaction time e. g., Das et al. and Sun et al. maintained their reaction time in 100 and 60-75 minutes, respectively [20, 22].

Color is very significant for the textile industry. There are more than 10,000 colors commercially obtainable all over the world [32]. In this point of view, it is not ascribable to supply textile items into the market only in one color. So, decolorization is a very important matter while recycling the fibers [33]. Due to omitting the color from blended waste fabric, it produces the foremost fibers with greyish, brownish, or its original white state, which is easy to dye with any other color or

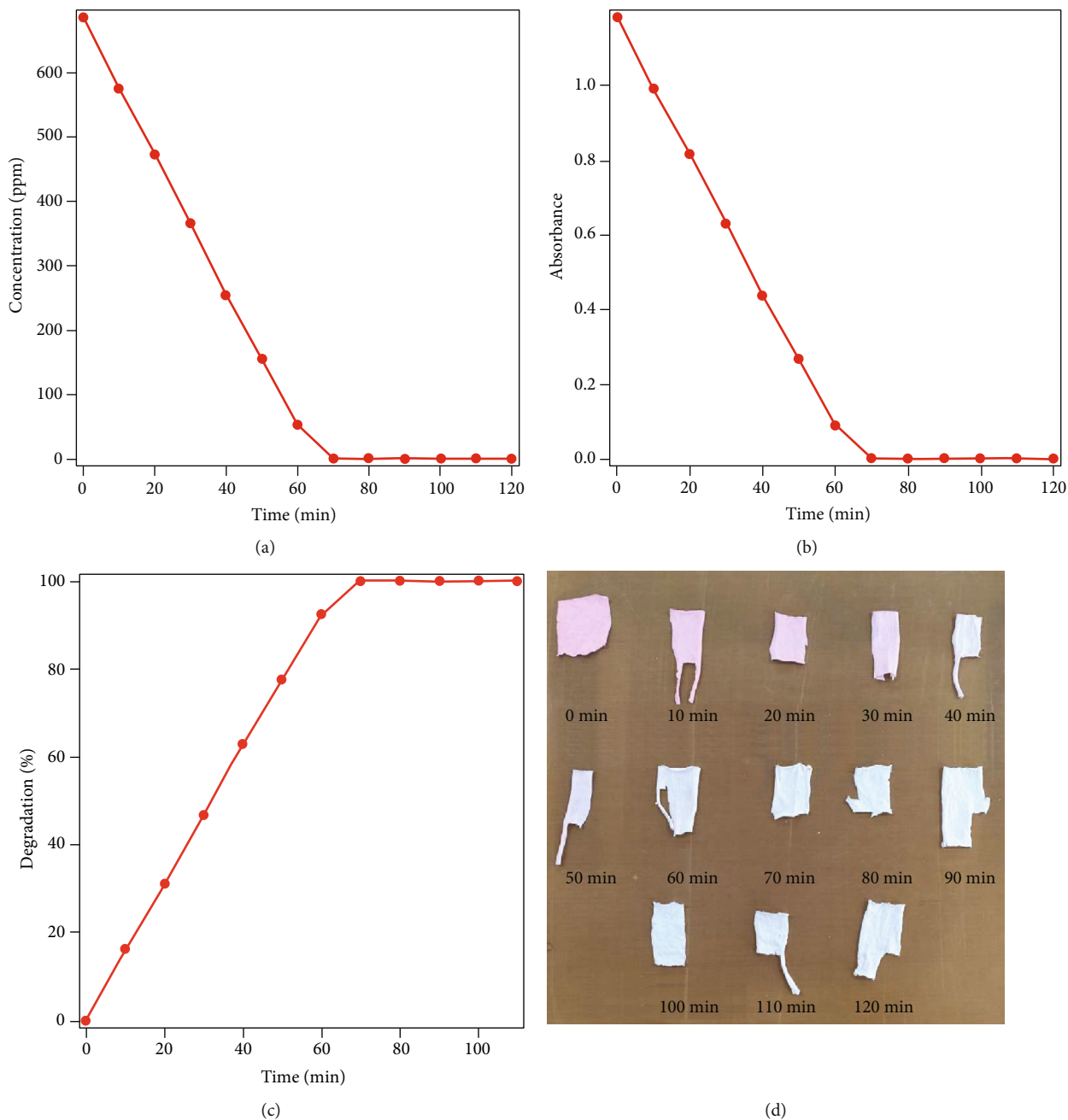


FIGURE 3: Graph of (a) concentration vs. time plot of PCWF before and after alkali treatment, (b) absorbance vs. time plot of PCWF before and after alkali treatment, (c) dye degradation percentage over time of PCWF before and after alkali treatment, and (d) image of the color change of PCWF at different times of alkaline treatment.

make yarn after trashing [34]. Direct dye removal with the separation process is crucial not only in terms of color management but also in terms of the circular economy.

This separation method is only effective for reactive-dyed fabrics; despite the limitation, it is a feasible process in many other respects. No pretreatment was given to any raw materials added here, and no catalyst was added to accelerate the reaction. Furthermore, in this method, not only PCWF but also polyester blends with other fabrics such as viscose; linen can also be successfully separated. For this article, only PCWF has been chosen. However, the textile

industry is currently turning to recycled-cotton yarn instead of conventional cotton yarn to combat the threat of global climate change. Accordingly, in 2020, recycled yarn market was valued at 3868.3 million dollars and is expected to reach 5960.8 million dollars by the end of 2026 with a CAGR (compound annual growth rate) of 6.3% during 2021-2026 [35].

Along with the increase in industrialization, the textile and packaging industries have also increased demand for TPA. In 2021, this demand was worth 54.8 billion dollars. By 2028, it will be 78.1 billion dollars; and between 2022

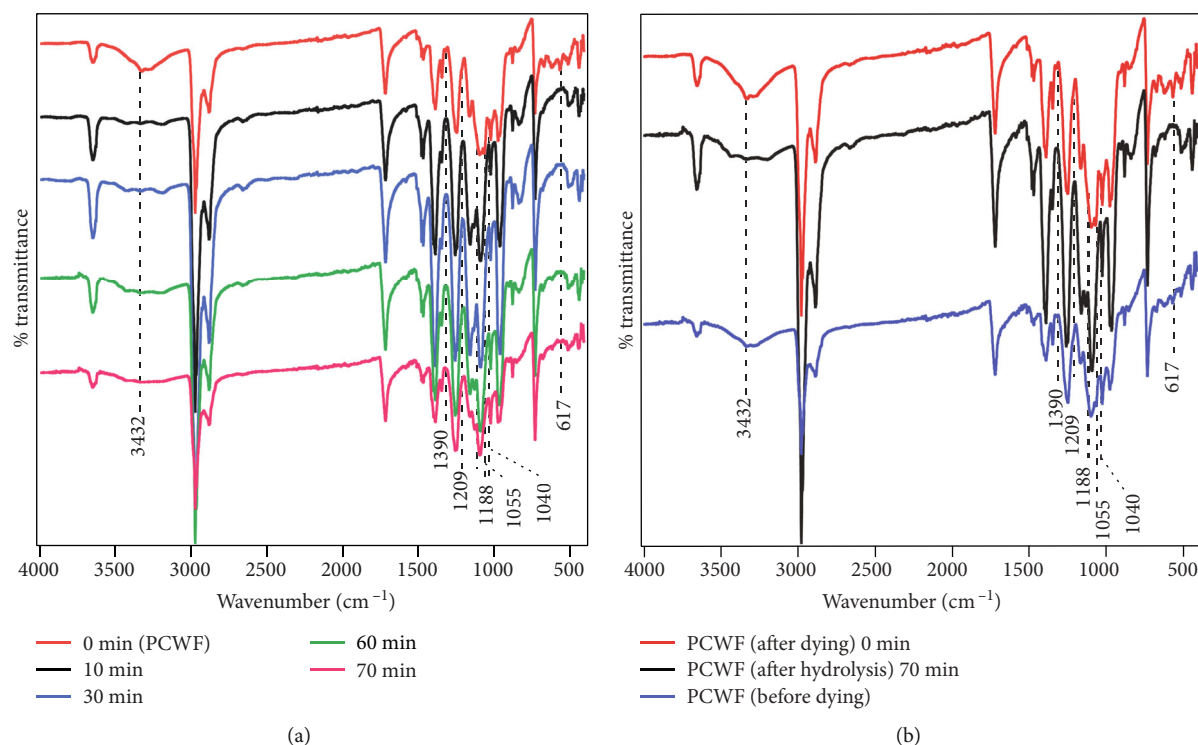


FIGURE 4: FTIR spectrum of (a) PCWF at 0 min before hydrolysis and 10 min, 30 min, 60 min, and 70 min after hydrolysis, and (b) PCWF (after dyeing at 0 min before hydrolysis), PCWF (after hydrolysis at 70 min), and PCWF (before dyeing).

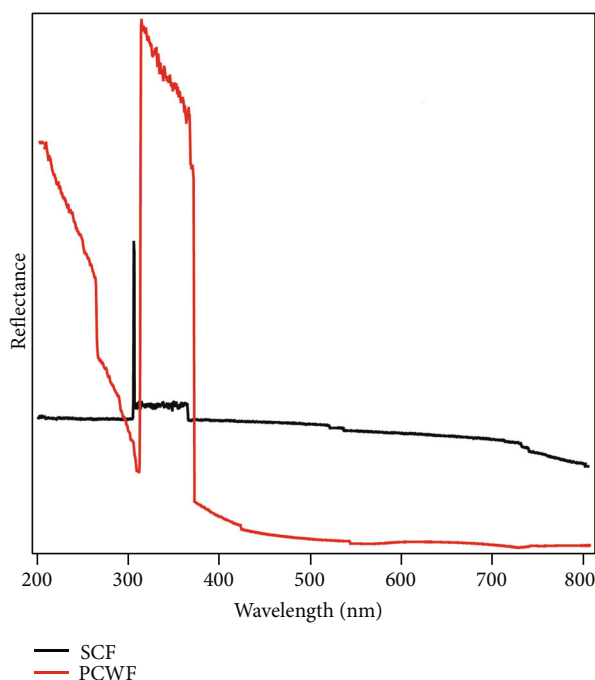


FIGURE 5: UV-visible spectrophotometric data of SCF (black) and PCWF (red).

and 2028, the CAGR (compound annual growth rate) will be 5.6% [36]. As a result, both SCF and TPA obtained using this method have positive economic implications.

This study mainly focused on reducing textile fabric waste (PCWF) using modified alkaline hydrolysis without producing secondary waste. TPA production and SCF decolorization with the direct use of PCWF without using any catalyst and minimizing the reaction time were the aims of this research work, which would be comprehensive examples of the circular economy concept.

## 2. Experimental

**2.1. Materials.** This research used PCWF as a raw material, where the polycotton ratio was 70 : 30. Echotex Ltd provided the raw material for this study. Sodium hydroxide (NaOH) (Merck, Germany) and sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), acetic acid (Sigma-Aldrich, Germany), pyridine (Merck, India), and phenolphthalein (Merck, India) were used in this analysis. All the chemicals used in this experiment were reagent grade and were not altered before use.

**2.2. Extraction of TPA from Polyester Cotton-Blended Colored Fabric.** The decolorization of PCWF and extraction of TPA is shown in Scheme 1. TPA and cotton's separation process from PCWF has been carried out in a round bottom (RB) flask. NaOH was first taken in the RB flask and dissolved in water. The PCWF to NaOH ratio was 5 : 10. The reaction was carried out in an Autoclave (model: Biomedic-65 L, Daihan Labtech Co., Ltd, South Korea) whereas reaction time were 60, 90, and 120 minutes with an autonomous pressure, which depends on the temperature. It was 103.42 KPa with a temperature of 121°C. After the reaction

TABLE 1: Shade (%), K/S value, and reflectance (%) of PCWF and SCF.

Sample name	Shade (%)	K/S value	Reflectance (%)
PCWF	0.0858	37.06	76.1
SCF	0	0.035	1.302

TABLE 2: Yield (%) of TPA in various reaction time.

Reaction time (min)	Yield (%)
120 min	98.78 ± 0.58%
90 min	97.66 ± 1.94%
60 min	75.37 ± 1.03%

TABLE 3: TPA, ash, and moisture content.

TPA content	Ash content	Moisture content
99.21 ± 0.35%	1.75 ± 0.05%	0.51 ± 0.04%

period, the sample was collected from the autoclave and filtered to separate the solid and liquid phases. The liquid phase was taken in a beaker and sedimented with 98%  $H_2SO_4$  at pH 3; then, the sediment was separated by filtration and centrifuged until the pH reached 7. Finally, the sample was dried at 105°C and stored for subsequent analysis. On the other hand, the solid residue soaked for 10 minutes in a 2% aqueous acetic acid solution. Then the residue was washed with water until neutral and dried in an oven at 70°C. For this solid sample, the conventional chemical dissolving technique was used to assess the purity of SCF under ISO 1833-11 : 2017.

**2.3. Determination of Mechanical and Physical Properties of Fabrics.** After polyester separation, mechanical properties (tensile strength, elongation, etc.) of PCWF and SCF were tested using the ASTM D5035 method at a speed of 300 mm/min and a gauge length of 100 mm with a Titan universal strength tester machine (1410 Titan 5, UK). Microsized fabrics were observed using MICROS optical microscope (MCX100) followed by EN ISO 1833-11 : 2017. Paraffin oil was used to disperse the samples.

**2.4. Determination of Dye Degradation.** The degradation study of PCWF was carried out to examine how time affected the process of degrading dye [37]. At first, a combined dye solution was first taken to make the standard curve using a UV-visible spectrophotometer (UV2401PC, Shimadzu, Japan). It was measured at 520 nm, and concentrations were 25, 50, 75, 100, 125, 150, 175, 200, 225, and 250 ppm. Then, the PCWF of before and after alkaline hydrolysis treatment at 121°C was taken to measure the color concentration and absorbance at different times (10, 20, 30, 40, 50, 60, 70, 80, 90, 100, 110, and 120). The percentage of dye degradation of PCWF was

calculated by using the following

$$\text{Dye degradation} = \frac{A_0 - A}{A_0} \times 100. \quad (1)$$

Here,  $A_0$  refers to the concentration of PCWF before alkali hydrolysis;  $A$  refers to the concentration of PCWF after alkali hydrolysis.

**2.5. Determination of K/S Value.** In colorimetry, color strength or K/S value is used to determine the depth of the fabric's color; and this value is measured by using reflectance (%). The reflectance (%) of untreated and alkali-treated fabric was measured by UV spectrophotometer (UV2401PC, Shimadzu, Japan). Then the following equation from Kubelka-Munk theory was used to find out the value of K/S [38].

$$\frac{K}{S} = \frac{(1 - R)^2}{R}. \quad (2)$$

In Equation (2),  $K$  is the absorption coefficient,  $S$  is the scattering coefficient, and  $R$  is reflectance.

**2.6. Measurement of TPA Yield.** The yield percentage of dried TPA was calculated using [39].

$$\text{TPA Yield}(\%) = \frac{\text{Actual yield of TPA (g)}}{\text{Theoretical yield of TPA (g)}} \times 100. \quad (3)$$

**2.7. TPA Content.** Around 1 g of dry TPA was placed in a conical flask, and 25 mL of pyridine was applied to dissolve the TPA sample. The suspension was heated using a reflux condenser. After the suspension had converted into a liquid solution, the condenser was cleaned with another 5 mL of pyridine. Finally, a 0.5 N NaOH solution was titrated against a phenolphthalein endpoint to determine purity [19].

**2.8. Moisture Content and Ash Content of TPA.** The moisture content of TPA was determined using a KERN moisture analyzer (model: DBS 60-3, Germany) and the ash content was determined using a muffle furnace (model: L51/S, Naber, Germany) with a crucible containing 5 g of TPA sample and a temperature of 800°C. The following was used to determine the ash content of TPA [31].

$$\text{Ash content of TPA} (\%) = \frac{\text{Weight of ash (g)}}{\text{weight of TPA (g)}} \times 100. \quad (4)$$

**2.9. Solubility of TPA.** The solubility of TPA was examined in water and ethanol. Two different TPA samples were taken to two separate beakers. The beakers were then filled with water and ethanol. The samples were filtered, dried, and weighed after an hour. In estimating the solubility of TPA in water and ethanol, the following was applied.

$$\text{TPA Solubility} = \frac{\text{Weight of TPA after test (g)}}{\text{weight of TPA before test (g)}} \times 100. \quad (5)$$

TABLE 4: Physiological parameters of TPA.

Form	Melting point	Solubility in water	Solubility in ethanol
Solid powder	295°C	Poorly soluble ( $0.0016 \pm 0.0002$ g/100 mL)	Poorly soluble ( $0.21 \pm 0.06$ g/100 mL)

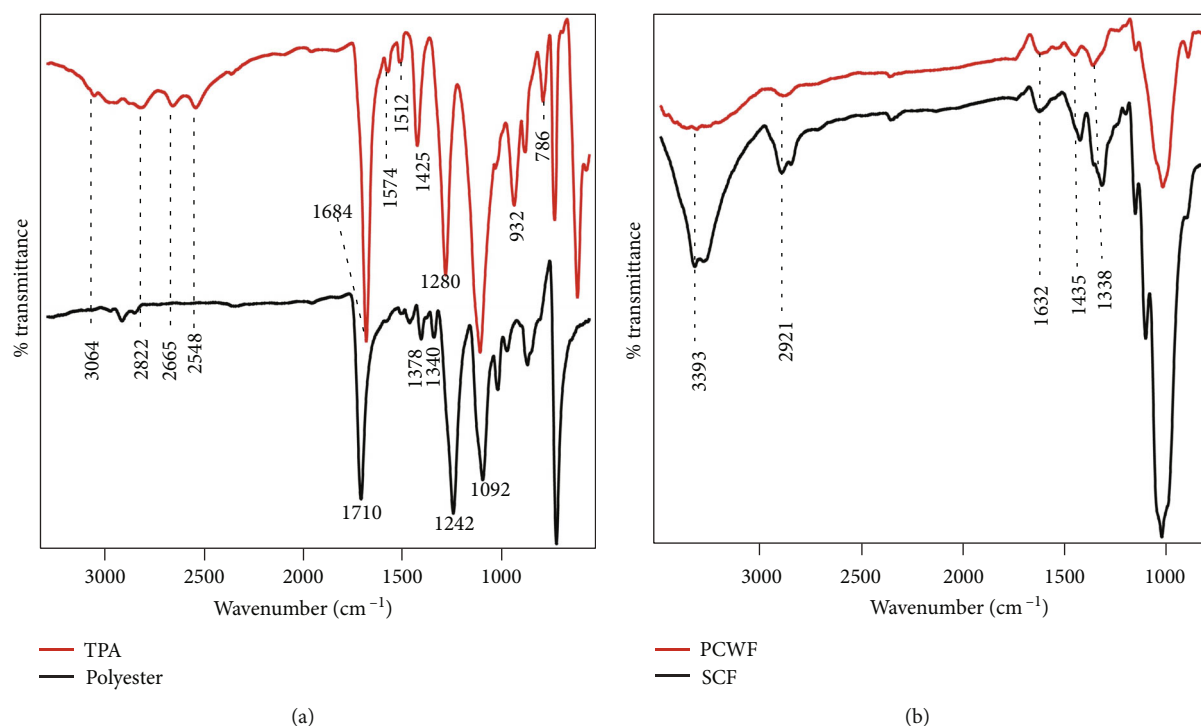


FIGURE 6: FTIR spectra of (a) TPA (red) and polyester (black) and (b) PCWF (red) and SCF (black).

**2.10. FTIR Spectroscopy.** A Fourier transform infrared spectrometer (model: Prestige-21, Shimadzu, Japan) was used to examine the functional groups of pure polyester, recycled TPA, PCWF, and SCF. All samples were dried in an oven to remove the unexpected moisture. Then 2 g KBr was mixed with 0.2 g of each powdered and dried sample and compressed into pellets. FTIR spectrum measurement range was  $4000\text{ cm}^{-1}$  to  $400\text{ cm}^{-1}$ .

**2.11. X-Ray Diffraction (XRD).** X-ray diffraction was used to determine the crystal structure and purity of TPA, pure polyester (collected from EchoTex Ltd.), SCF, and PCWF. An X-ray diffractometer (model: SmartLab SE, Rigaku, Australia) was used to conduct this experiment at room temperature. The scanning rate was  $0.1^\circ\text{S}^{-1}$ , and the Cu-K $\alpha$  radiation was used with a range of  $2\theta = 1^\circ$  to  $50^\circ$ .

**2.12. Thermogravimetric Analysis.** The thermal behavior of PCWF, SCF, TPA, and pure polyester (collected from EchoTex Ltd.) was investigated using a thermogravimetric analyzer (TGA) (model: STA 449 F3 Jupiter, NETZSCH, Germany). The samples were heated at a steady heating rate of  $1.50^\circ\text{C}/\text{min}$  up to  $600^\circ\text{C}$  in a nitrogen atmosphere.

**2.13. Scanning Electron Microscopy and Energy Dispersive X-Ray.** The dried TPA and pure polyester (collected from

EchoTex Ltd.) samples were examined and photographed using a scanning electron microscope (model: PV25MK, Edwards, England) that had been coated with gold and observed at 15 (kV) accelerating voltage. The elemental constituents of polyester and TPA powder were studied using Energy-Dispersive X-ray (EDX).

### 3. Results and Discussion

**3.1. Mechanical and Physical Properties of Fabrics.** Figure 1 illustrates the tensile strength and elongation at break, obtained from the stress-strain graph. In Figure 1(a), tensile strength measure is taken towards the warp for both samples. For PCWF, 62 KPa force had been applied to overcome the stress tolerance limit, and the SCF had been deformed at 9 KPa force. Similarly, in Figure 1(b), tensile strength-measuring was taken towards the weft for both samples. PCWF overcame its stress tolerance limit at 41 KPa, and SCF deformed at 12 KPa. In both cases, for warp and weft, PCWF's tensile strength was more than SCF.

Besides, in Figure 1(c), PCWF and SCF were elongated 119% and 120%, respectively, towards the warp before exceeding the stress limit. In Figure 1(d), towards the weft, the elongation was 335% and 220%, respectively. In this case, for both PCWF and SCF, elongation was more towards weft than towards warp.



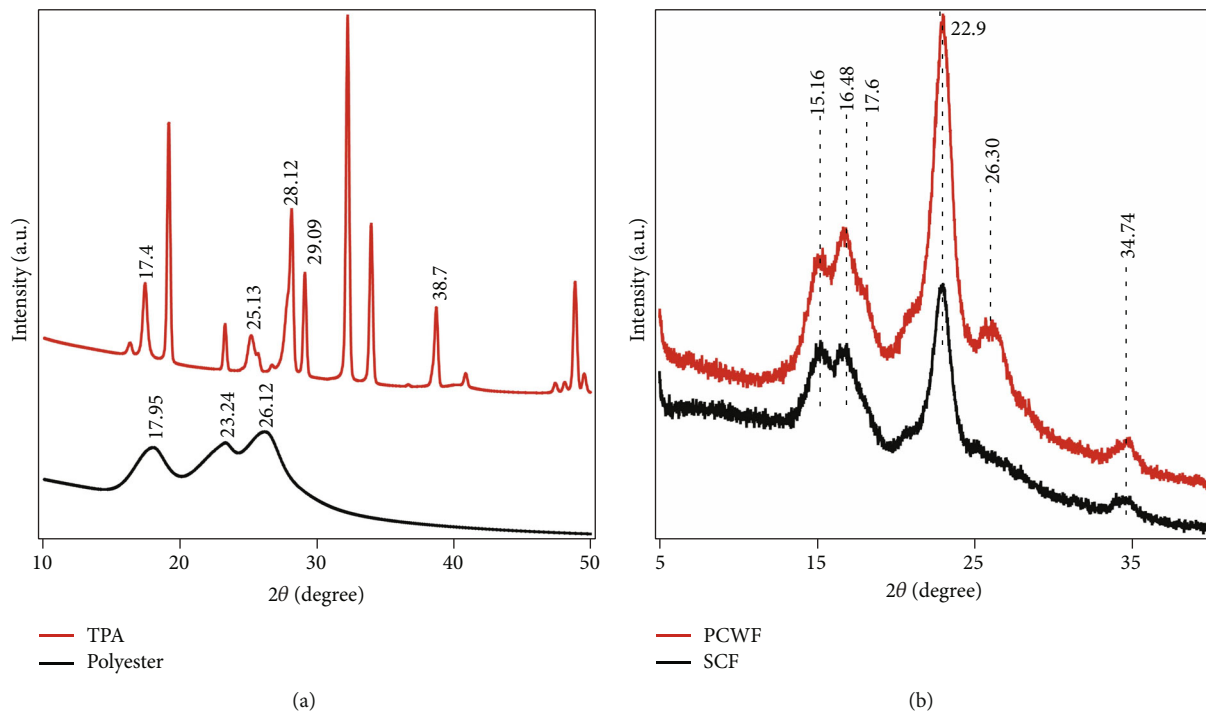


FIGURE 7: XRD spectra of (a) TPA (red) and polyester (black) and (b) PCWF (red) and SCF (black).

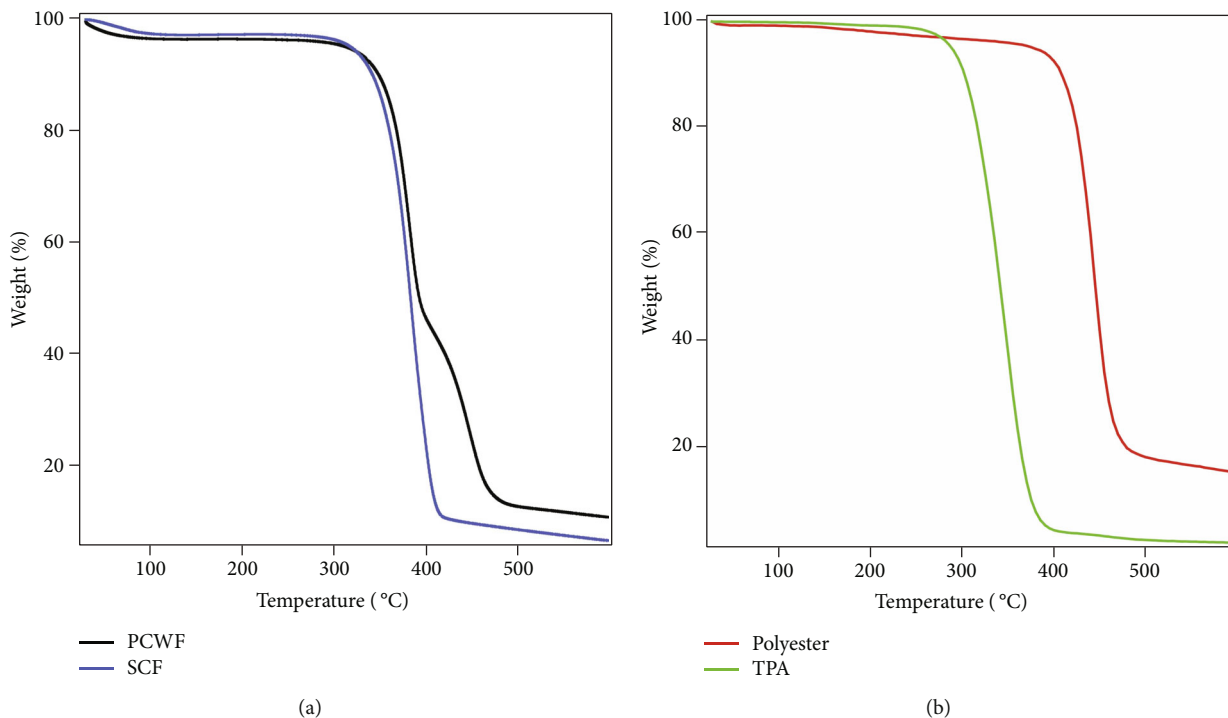


FIGURE 8: TG curve of (a) PCWF (black) and SFC (blue) and (b) polyester (red) and TPA (green).

A microscopic image is an easy way to distinguish between fibers. Figure 2 shows the longitudinal microscopic image of PCWF where structureless, uniform diameter, rod-like appearance is found with mature, flat, ribbon-like appearance. Besides, Figure 2(d) shows the longitudinal microscopic image of SCF where a thin, almost transparent,

mercerized smooth, and cylindrical structure is observed, and due to the hydrolysis process, rod-like appearance has wholly disappeared from the SCF [40].

3.2. *Dye Degradation Study.* When a dye has a reactive group which can form covalent bonds by chemical reaction with

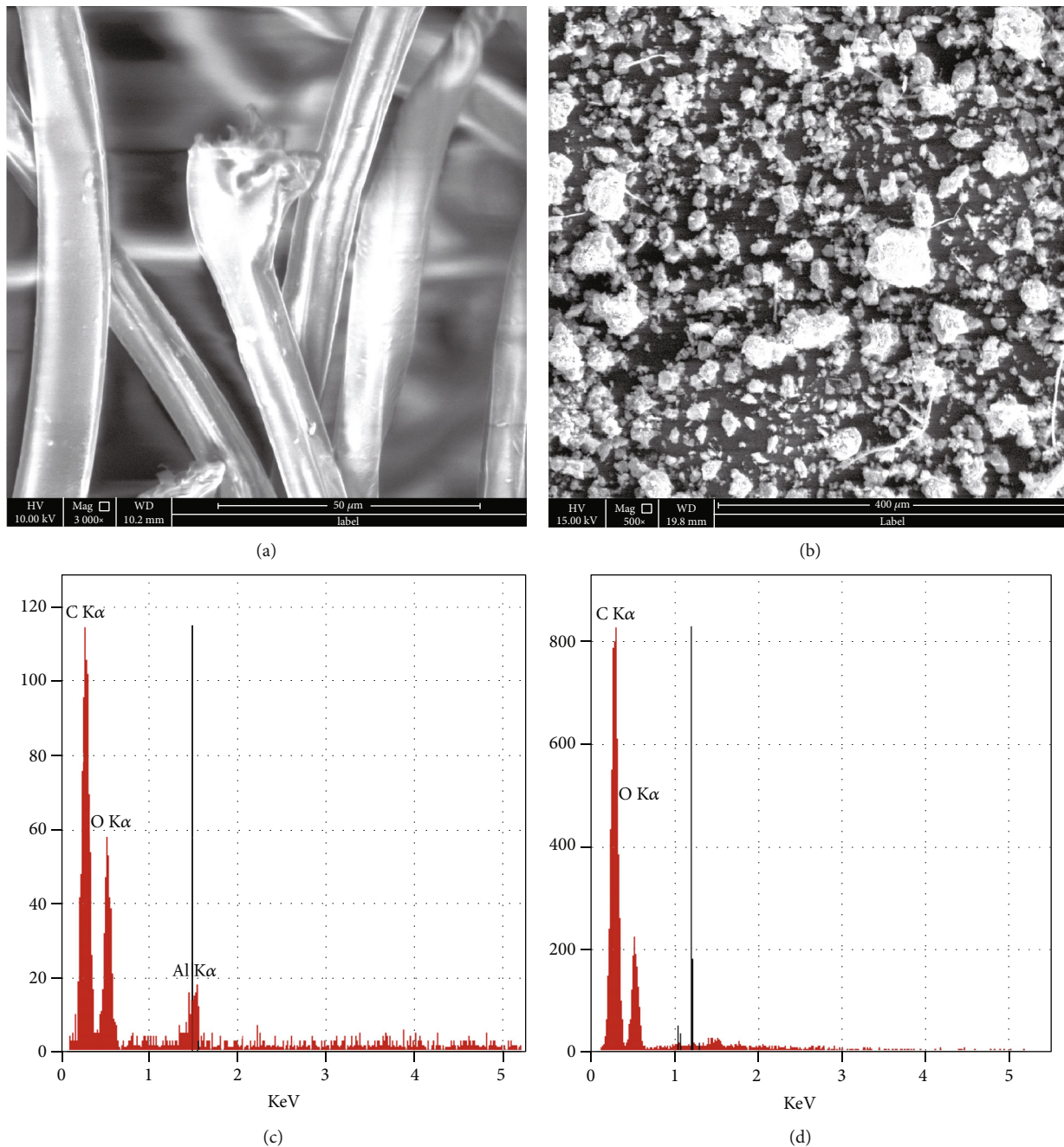


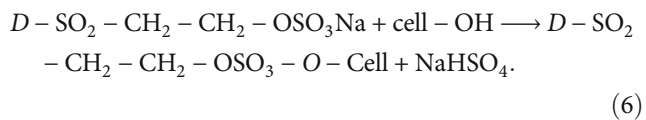
FIGURE 9: Polyester SEM (a) and EDX (c) and TPA SEM (b) and EDX (d).

the terminal -OH group of fiber, it is called reactive dye. This type of bond is much stronger than an ionic bond, hydrogen bond, and van der Waals force [41]. In this research, three types of reactive dyes were used, e.g., Remazol Brilliant Blue RR (RBB), Remazol Yellow RR (RY), and Remazol Red RR (RR), which structures were shown in Scheme 2. The combination of these three hues was used to dye the PCWF fabric.

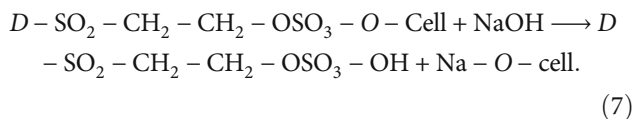
During dye degradation under alkaline circumstances, the surface tension of dyes gradually decreases over time, and the dye undergoes hydrolytic fission, resulting in the formation of non-reactive oxi-dye. The longer the time spent in an alkaline

solution, the dye concentration drops, which is evident from its fading, depending upon time. The hydroxyl group can accelerate dye ionization and degradation by blocking dye molecule contact. Therefore, dye and fiber cannot interact again, and dye loss is enhanced during hydrolysis [42].

At dye bath:



At Alkali Bath:



Here, D means dye and cell means cellulosic polymer.

Figures 3(a) and 3(b) demonstrates how the concentration and absorbance of PCWF change over time at 520 nm wavelength and in an alkali environment. It achieves an equilibrium state after 60 min. At 60 min, concentration and absorbance were 52.24 ppm and 0.09 gradually. The concentration and absorbance values of PCWF before alkali hydrolysis are 686.4 ppm and 1.184. Figure 3(c) also demonstrates how the dye degradation rate gradually increases. The degradation percentage did not significantly rise after 70 minutes. At equilibrium, the dye degradation of PCWF was 100%. Thus, this study suggests that alkali hydrolysis could be an alternate, more efficient method for degrading colors from PCWF.

Figure 4(a) depicts the FTIR spectrum data of PCWF at 0 min before hydrolysis and 10 min, 30 min, 60 min, and 70 min after hydrolysis. Six significant peaks can be seen in the PCWF at 0 min before alkaline hydrolysis: 3432  $\text{cm}^{-1}$  for the  $\text{NH}_2$  stretching vibration, 1390  $\text{cm}^{-1}$  for the S=O stretching of sulfite, 1209  $\text{cm}^{-1}$  as well as 1040  $\text{cm}^{-1}$  for the S=O stretching of sulphonic acid, 1188  $\text{cm}^{-1}$  for the symmetric vibration of  $-\text{SO}_3\text{H}$ , 1055  $\text{cm}^{-1}$  for the C-N stretching, and 617  $\text{cm}^{-1}$  for the C-S stretching of the sulfur group [43–45].

These six peaks specify the characteristics of three dyes—RR, RY, and RBB. At 10 min of alkaline treatment, the molecules imperceptible are 3432  $\text{cm}^{-1}$ , 1209  $\text{cm}^{-1}$ , and 617  $\text{cm}^{-1}$ . All bonds except symmetric vibration of  $-\text{SO}_3\text{H}$  at 1188  $\text{cm}^{-1}$  and S=O stretching of sulphonic acid at 1040  $\text{cm}^{-1}$  are broken when the alkaline treatment reaches 30 min. Only one peak left at 1040  $\text{cm}^{-1}$  for S=O stretching of sulphonic acid at 60 min of hydrolysis, a stage before equilibrium. Any dye peak is not observed at 70 min, indicating that dyes in PCWF have completely degraded by that point. This state demonstrates the sustainability of the degradation state.

The comparison among PCWF (after dyeing at 0 min before hydrolysis), PCWF (after hydrolysis at 70 min), and PCWF (before dyeing) is shown in Figure 4(b). PCWF (before dyeing) does not exhibit the typical peaks of the three colors. However, the distinctive color peaks appear in PCWF (after dyeing at 0 min before hydrolysis), and in PCWF (after hydrolysis at 70 min), those vanish once more.

**3.3. K/S Value.** In textile applications, the K/S value is essential to control the color process parameters in the dyeing and finishing of fabrics [46]. Furthermore, dyes' property indicates the capacity to transfer color to other materials [47]. The reflectance (%) of a fabric's surface can influence this value. The reflectance (%) of a fabric's surface indicates the ability to reverberate the energy transferred by electromagnetic radiation at a specified frequency.

The UV spectra of SCF (black) and PCWF (red) are illustrated in Figure 5. PCWF's K/S value is 37.06, and the

reflectance (%) is 76.1. The SCF's K/S value is 0.035, and the reflectance (%) is 1.302. On the other hand, shade (%) refers to the color's depth, which is influenced by the K/S value. As the K/S value rises, the shade (%) becomes lower. When the K/S value is 37.06 in PCWF, the shade (%) reduces to 0.0858. As a result, the K/S value and reflectance (%) are inversely proportional to shade (%). Table 1 shows the relation between shade (%), K/S value, and reflectance (%).

**3.4. Yield (%) of TPA.** In this experiment, the depolymerization of polyester without catalyst was carried out at 121°C, 103.42 KPa, and the same amount of alkali solution but varying with the reaction times. The first test was conducted at 60 minutes then gradually increased the reaction time. The influence of reaction time on the degradation is shown in Table 2. The degradation of polyester was almost half at 60 minutes. Carrying out the reaction for 90 minutes led to very high polyester degradation, and the TPA recovery yield was  $97.66 \pm 1.94\%$ . At 120 minutes, the polyester degradation was also nearly complete, and the TPA recovery rate was  $98.78 \pm 0.58\%$ . Thus, it can be predicted that it would be hard to make polyester depolymerized completely, even for a long enough time. The TPA yield (%) for different reaction times is shown in Table 2. According to the data, it optimized the 90 minutes reaction time for further procedures.

**3.5. TPA, Ash, and Moisture Content.** The ash content of recycled TPA was used to determine inorganic impurities such as sodium sulfate [31]. Table 3 shows the TPA, ash, and moisture content data.

**3.6. Physiological Parameters.** TPA, made from PCWF, has several physiological properties. TPA is a fine powder with a slightly acidic odor, low water, and alcohol solubility [48]. Table 4 summarizes the findings.

**3.7. FTIR Analysis.** FTIR spectra of terephthalic acid (TPA) were used to characterize and compare it with pure polyester (Figure 6(a)). The stretching vibration and out-of-plane ring bend of the C-H group in the benzene ring trigger the transmittance peak at 3064  $\text{cm}^{-1}$ . The -OH group stretches vibration in carboxylic groups described by absorption peaks in 2500–3000  $\text{cm}^{-1}$ , 2548  $\text{cm}^{-1}$ , 2665  $\text{cm}^{-1}$ , and 2822  $\text{cm}^{-1}$  are the bands in the 2500–3000  $\text{cm}^{-1}$  range. The absorption band at 1710  $\text{cm}^{-1}$  indicates the presence of a carbonyl group in an ester, which is a characteristic of pure polyester. When the bond energy decreases from 1700  $\text{cm}^{-1}$ , it shows the complete depolymerization of polyester, and 1684  $\text{cm}^{-1}$  indicates the formation of TPA monomer [39]. The skeletal vibration of the aromatic phenyl ring is shown by the 1574  $\text{cm}^{-1}$  and 1512  $\text{cm}^{-1}$  peaks. The frequency of C-O, which is present in pure TPA, is represented by the 1280  $\text{cm}^{-1}$  band. The O-H groups' flexural vibration peaks are 1425  $\text{cm}^{-1}$  and 932  $\text{cm}^{-1}$  [49]. The carboxyl groups in the phenyl ring are in the paraposition, as shown by the peak at 786  $\text{cm}^{-1}$  [39].

According to Figure 6(a), the FTIR spectrum reveals a typical signal at 1710  $\text{cm}^{-1}$  that corresponds to C=O carbonyl stretching due to acid groups and is consistent with perpendicular dichroism [50]. A band at 1242  $\text{cm}^{-1}$  relates to the C-

O stretching of ester groups, linked to parallel dichroism [49, 51]. C-O stretching of ester groups are also allocated to a band at  $1092\text{ cm}^{-1}$ . The aliphatic C-H bonds are responsible for the wavelengths of  $1340\text{ cm}^{-1}$  and  $1378\text{ cm}^{-1}$ . The purity of polyester is highlighted by the absorption bands mentioned above [27]. The FTIR spectra of TPA do not contain any of the mentioned similar peaks of pure polyester, ensuring the formation of TPA monomer [52].

The FTIR spectra of PCWF (collected from EchoTex Ltd.) and SCF are represented in Figure 6(b). The absorption band at  $3393\text{ cm}^{-1}$  from FTIR analysis is due to the intermolecular and intramolecular O-H stretching [29]. C-H stretching vibration is responsible for the band  $2921\text{ cm}^{-1}$ .  $1632\text{ cm}^{-1}$  is shown for the C=C stretching band. Besides,  $1435\text{ cm}^{-1}$  and  $1338\text{ cm}^{-1}$  bands are assigned to  $-\text{CH}_2$  scissoring and  $-\text{OH}$  bending vibrations, respectively.

**3.8. X-Ray Diffraction Analysis.** Figure 7(a) gradually depicts the characteristic crystalline peaks of polyester and TPA. The diffractogram exhibits the characteristic crystalline peaks of polyester at  $2\theta=17.95, 23.24,$  and  $26.12$ . These three sharp and narrow peaks demonstrate polyester's purity and crystallinity [53]. TPA's crystallinity peaks are observed at  $2\theta=17.4, 25.13, 28.12, 29.09,$  and  $38.7$ . According to Ren et al., these narrow and sharp diffraction peaks confirmed the high crystallinity and purity of TPA [54].

The XRD analysis at Figure 7(b) shows that PCWF has XRD peaks at  $2\theta=15.16, 16.48, 17.6, 22.9, 26.30,$  and  $34.34$ . But after alkaline hydrolysis, XRD peaks at  $2\theta=15.16, 16.48, 22.9,$  and  $34.74$ , representing SCF as cellulose I type, and the hydrolysis process does not change the cellulose structure of SCF [30]. According to Hou et al., these sharp and narrow peaks represent the crystalline phase, and the remaining peaks represent the amorphous phase of cotton [29].

**3.9. Thermogravimetric Analysis.** Thermogravimetric analysis under a nitrogen atmosphere can provide thermal stability information by studying the weight loss curves, as shown in Figure 8. The results obtained from the TGA analysis of the PCWF and SCF are shown in Figure 8(a). The rate of weight loss reflects the kinetics of thermal degradation. The decomposition of PCWF occurs in a slightly different way than SCF. Initially, the initial weight loss was executed for PCWF and SCF due to the vaporization of water [55]. The preliminary weight loss for PCWF and SCF occurred at  $100^\circ\text{C}$  to  $150^\circ\text{C}$ . The maximum decomposition of SCF at  $330^\circ\text{C}$  to  $410^\circ\text{C}$  is related to the degradation of the cellulose in the fiber [56]. After  $550^\circ\text{C}$ , the residue rate of SCF was 6.49%.

Besides, fractional thermal decomposition of some components of PCWF is observed in the range of  $250^\circ\text{C}$  to  $300^\circ\text{C}$ . The TG curve shows significant PCWF deterioration between  $340$  and  $480^\circ\text{C}$ . The highest PCWF thermal breakdown is attributed at  $550^\circ\text{C}$ , and the rate of weight loss is 89.33%. This decomposition of PCWF happened in three steps. Firstly, cotton serves as the initial combustion source and begins to degrade thermally at a temperature significantly lower than that needed to decay polyester. Therefore, the total decomposition of cotton and the partial decompo-

sition of polyester is the primary cause of the weight loss in the second step. In the third part, an additional temperature is needed for the combustion of polyester. [57].

Figure 8(b) depicts TG of polyester and TPA. A single-phase decomposition profile exhibits pure polyester [58]. Polyester is thermally stable under  $390^\circ\text{C}$ . It began to reduce weight with the increase in temperature. Once the temperature of  $476^\circ\text{C}$  was achieved, maximum decomposition of polyester occurred, and weight loss was 79.08%. But no weight loss was observed above  $700^\circ\text{C}$  [59].

On the other hand, TPA is thermally stable below  $290^\circ\text{C}$  and continues to sublimate as the temperature rises. TPA degraded at  $390^\circ\text{C}$  and achieved the maximum weight loss of 99.21%. Polyester's molecular structure and bond energy are both affected during conversion to TPA, resulting in TPA's slightly different thermal behavior. This complete thermal decomposition took place in an endothermic nature [60].

**3.10. Scanning Electron Microscopy and Energy-Dispersive X-Ray Analysis.** The visual morphological image of pure polyester and TPA after the hydrothermal process was investigated using SEM, depicts in Figures 9(a) and 9(b). The peripheries of polyesters are smoothly compatible with their synthetic origin [61]. Due to the hydrothermal reaction, the surface of TPA is not as smooth as those of polyester. It demonstrates that the polyester to TPA conversion was flawless and that the TPA contained no polyester oligomers. On the other hand, small TPA particles exhibit compatibility and irregularity [30].

Figures 9(c) and 9(d) corresponding to EDX show the percentage composition of the elements for polyester and TPA and EDX spectra. Only C and O elements were found in the EDX study of the polyester fiber and TPA. Because of the aluminum plate, there is a peak of  $\text{Al}^{3+}$  metal at both polyester and TPA. TPA's composition shows the absence of sodium and sulfur ion impurities.

## 4. Conclusion

This study successfully revealed a sustainable and practical way to SCF and convert polyester to TPA from PCWF. This research establishes a high-purity TPA preparation process ideal for industrial production. NaOH was used to decolorize PCWF, depolymerize polyester, and  $\text{H}_2\text{SO}_4$  to generate TPA. The optimum temperature, time, and pressure were  $121^\circ\text{C}$ , 90 minutes, and 103.42 KPa. No pretreatment for PCWF was proposed in this research, and no catalyst was used. This method makes it easier to separate polyester and removes the dye from the SCF, which can be used for yarn production. Ultimately, this developed method is a way to save the environment by diminishing textile solid waste and creating a circular raw material chain by recycling textile cutting waste.

## Data Availability

The data used to support the findings of this study are available within the article. Further data or information is available from the corresponding author upon request.

## Conflicts of Interest

The authors declare that they have no conflict of interest.

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