Reconstruction of cotton-polyester blended fabric

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Abstract. Through this article, as the procedures of added glycol (EG) catalyzer in reaction system; After depolymerize the polyester fibre (PET) of the waste cotton-polyester blended fabric to alcoholysis as bis-hydroxyethyl terephthalate (BHET), and then separate it with solid cotton and liquid BHET through the Solid-liquid separation device. To added separation product (BHET) and catalyzer in the Polycondensation reaction system to polycondensation BHET as reconstituted Polyethylene terephthalate (PET). The experimental results indicated that separated cotton and reconstituted PET meet the requirement of recycling.

1. Introduction
Currently, most of the waste textiles are mainly take the measures by physical, e.g. in processes of machining preliminarily, incineration, burying, etc. Therefore, greatly restrict the recycling value and renewable economic benefits of waste textiles. Also, in the aspect of recycling by chemical, most of the blend fabric can’t be separated or recycled directly because of textiles are not existing with a single component thus greatly affected the recycling rate as well.

In this paper, through the way of ethylene glycol alcoholysis to conduct chemical alcoholysis with waste cotton-polyester blended fabric, meanwhile, to realize the polycondensation and reconstitution with alcoholysis products.

2. Experimental part

2.1 Alcoholysis separation process

The waste polyester-cotton blended fabric is chopped into an area of about 4-25cm², wash it with water then dry it and sample it with 20g. According to a certain quality ratio to add the waste fabric, glycol and catalyst zinc acetate to a high temperature reactor with dasher for alcoholysis. After the completion of the reaction, under the protection of nitrogen to lower the temperature of them until about 150°C, then filter and separate quickly to obtain solid cotton 1st being washed by glycol with the temperature of 150°C, 2nd being washed by water then dry it to obtain pure cotton fibre as “sample A”, then conduct the analysis for the surface with electron microscopy and analysis of tensile properties of “sample A”.

2.2 Polycondensation and reconstitution process

Make use of the separated liquid and catalyst ethylene glycol antimony to added into the polycondensation reactor for polycondensation with the mass ratio as 1:0.1%[1]. through the pre-polycondensation and final-polycondensation to obtain the polyethylene glycol terephthalate (PET) as the “sample B”. the-period of pre-polycondensation creation of vacuum is 0.5h. the siring speed is
6r/min \[^{[2]}\], the temperature is 265-275 °C, vacuum degree is 0.9kPa, the period of final-polycondensation creation of vacuum is 0.5h-3h, Siring speed 4.5r/min, the temperature maintain as 285°C \[^{[3]}\], the vacuum degree maintain as 10-40Pa. To conduct DSC differential thermal analysis, IR infrared analysis, Intrinsic viscosity analysis with sample B.

### 3. Results and discussion

#### 3.1 electron microscope analysis of cotton fiber

By using of QUANTA200 scanning electron microscope manufactured by FEI Company to conduct analysis for the before and after experiment surface of cotton fibre. The surface electron microscope pictures as the Figure 1 shown is the cotton fibre are being magnified with 6K(left), 10K(right) before experiment. As the Figure 2 shown the cotton fibre are being magnified with 6K(left), 10K(right) after experiment.

In comparison can be seen that there’s a certain differences between before and after experiment of cotton fibre. There are Some natural impurities on the surface of the cotton fiber before the experiment, the impurities disappeared after the processes of experiment to make the surface of the fibre become smooth. Through the analysis can be reached is that for part of the cotton fiber surface impurities mixed into EG, BHET Mixed solution in processes of experiment may cause Irregular peaks of test of the PET (sample B) was being obtained from polycondensation experiment.

![Figure 1](image1.png)

**Figure 1.** The surface electron microscopy pictures of the cotton fibre was being magnified with 6K (left), 10K (right) before the experiment

![Figure 2](image2.png)

**Figure 2.** The surface electron microscopy pictures of the cotton fibre was being magnified with 6K (left), 10K (right) after the experiment

#### 3.2 the analysis for the Tensile properties of cotton fibre

By using of the model of YGO01A fiber electronic strength meter manufactured by Taicang Textile Instrument Factory to perform the Tensile properties test of cotton fibre after the experiment,
tensile speed 15mm/min; pretension 150mg; Stretching distance 10mm, for sampling 50 group of fibre to conduct measure then take the average value. The tensile property performance of the cotton fibre as the Table 1 shown below:

| Experimental productD | Ordinary cotton fiber\cite{4} |
|-----------------------|--------------------------------|
| Breaking strength (/cN·dtex\(^{-1}\)) | 2.19 | 1.8-3.1 |
| elongation at break (/%) | 7.91 | 7.0-12.0 |
| Initial modulus (/cN·dtex\(^{-1}\)) | 68.31 | 60.0-82.0 |

As can been seen from the date of test that after the experiment the Single cotton fiber breaking strength, elongation at break and fiber modulus may remain within the range of spinnable cotton fiber. So It can be seen the high temperature alcoholysis experiment in short time could not be a serious damage for the cotton fiber.

3.3 DSC analysis

By using of DSC Differential scanning calorimetry scanner manufactured by Germany NETZSCH company to conduct measurement in Nitrogen, with the heating rate of 20\(^°\)C/min to heat the temperature from 30\(^°\)C to 280\(^°\)C then suspend with 5mins, and get start to Cooling down with the cooling rate of 20 \(^°\)C / min from 280 \(^°\)C down to 30 \(^°\)C. The DSC differential thermal analysis. The Sample E is the PET slices made by Waste fabric which without Zn(AC)\(_2\) alcoholysis catalyst, Sample F is a PET slice produced by Sinopec Yizheng Chemical Fibre co., LTD. The Sample G is the bubble material produced by Juyou Chemical. The DSC Differential calorimetry analysis as the Table 2 shown:

| Glass transition temperature Tg/\(^°\)C | Crystalline peak (Tc)/\(^°\)C | Enthalpy of crystallization J/g | Melting peak (Tm)/\(^°\)C | Enthalpy of fusion J/g | Exothermic crystallization peak (Tc\(\prime\))/\(^°\)C | degree of super-cooling (\(\Delta\)Tc)/\(^°\)C |
|---------------------------------------|-----------------------------|-------------------------------|--------------------------|-----------------------|---------------------------------------------|-----------------------------|
| Sample B | 74.504 | 137.919 | 31.899 | 238.228 | 36.316 | 173.740 | 64.548 |
| Sample E | 70.885 | 133.902 | 32.195 | 229.241 | 33.712 | 167.423 | 61.818 |
| Sample F | 80.663 | 152.621 | 33.684 | 252.882 | 37.405 | 175.079 | 77.803 |
| Sample G | 80.793 | 152.942 | 31.550 | 248.212 | 33.415 | 174.413 | 73.799 |

Glass-transition temperature provides the information of Amorphous phase chain segment motion, PET can cause Physical, especially rapid changes of mechanical performance when it get changing. Therefore, the glass transition temperature is often a very important parameter to be considered in pet processing or application. As shown in table 2 The glass transition temperature of sample B and sample E is lower and Its physical processing performance will also decline accordingly because of the main reason is that the sample B and sample e are partially degraded during the process of Recycling.

Degree of super-cooling (\(\Delta\)Tc)\(\Delta\)Tc=Tm-Tc\(\prime\), degree of super-cooling is mainly determined by the driving force and temperature of the Phase transition process. In the process of phase transition, there should be a "too cold", and then the phase transition will be took place spontaneously. And the more faster undercooling (the less degree of super-cooling) is, the more likely it is to crystallize. Therefore,
the more smaller the degree of super-cooling is, to indicated that the crystallization ability of PET is more stronger. As can be seen from the calculations above, the sample B is more easily crystallized than the sample F.

However, because of there’re varying degrees of degradation of Sample B, E in the process of Polyester alcoholysis, so there is the phenomenon of uneven distribution of molecular weight In the process of polycondensation, moreover, Sample B, E were used of Metal ion catalyst in process of alcoholysis and polycondensation to cause varying degrees effect of reconstituted polyester slice molecule. These are the main reasons for the differences between reconstituted polyester and Yizheng polyester chips. By the comparison of sample B and sample E that can be seen the data of sample B is more closer to sample F, Mainly because the sample E wasn’t be added with depolymerization catalyst to make the duration of alcoholysis became longer thus cause excessive degradation of depolymerization product, so finally. The performance of reconstituted polyester was affected. From this it is able to see that the using of depolymerization catalyst played a positive role for the reconstituted polyester.

3.4 IR analysis

By using of the vector 22 infrared spectrometer produced by Germany Bruker to test sample B, IR infrared analysis of the sample B as the Figure 3 shown.

From the figure 3 can be seen that the peak at 3055cm⁻¹ is C-H stretching vibration of benzene ring, the 2970cm⁻¹ and 2908cm⁻¹ are caused by saturated methylene CH₂ stretching vibrations, the 1960cm⁻¹ is a pan frequency peak, the 1730cm⁻¹ is the C=O stretching vibration absorption peak, 3430cm⁻¹ is the frequency doubling peak of 1730cm⁻¹ peak, The peaks of 1283cm⁻¹ and 1132cm⁻¹ are generated by the C-O-C stretching vibration; 1021cm⁻¹ and 972cm⁻¹ belong to the absorption peaks of -OCH₂CH₂O- ;730cm⁻¹ is the oscillation absorption peak in the benzene torus of PET. Thus, sample B has all the basic characteristics of PET. However in Figure 3, there is a carboxyl group of antisymmetric stretching vibration absorption peaks near 1538cm⁻¹, which indicates that besides -COOH there is carboxylate [5] in the sample B. The reason is that zinc acetate is added to the depolymerization reaction and ethylene glycol antimony is added as the catalyst in the polycondensation reaction. In contrast can be seen by figure 3, there are more mixed peaks in 2000cm⁻¹ to 4000cm⁻¹ in samples B, the main reason is that the addition of catalyst, the mixture of residual unknown object on waste fabric, and the generation of oligomers during polycondensation.

![Figure 3. IR analysis](image-url)
3.5 Intrinsic viscosity analysis

With the mass ratio of 1:1 to phenol four chloride mixed solvent, the sample B polyester PET tested as sample is cut into small particles (about LMM * 1mm) and put into the weighing bottle, then put the bottle in a drying box for drying with the temperature of 90°C, after 20-25min with the evaporation of the water in the sample, and take it out to put into a desiccator for cooling until reach room temperature. Weigh the sample with the amount of b0.125g after drying (accurate to 0.0001g), placed into a dry 25ml volumetric flask. Be adding 15-20ml prepared phenol four chloride solvent by straw to heating and dissolving in the oven with the temperature of 80-90°C. When all the samples are dissolved, cool down to room temperature. Then place the measuring bottle into the constant temperature water tank with the temperature of 25°C±0.1°C for 15mins. Then next measure the flowing time of the determination of solution with the Ubbelohde viscosity agent under the temperature of 25°C. Then conduct the conversion to obtain the intrinsic viscosity of sample B is \([\eta]=0.6892\text{dL/g}\). John Shais pointed out that the best intrinsic viscosity of polyester chip for spinning is between \([\eta]=0.62-0.68\ \text{dL/g}\). Therefore, the sample B has reached the requirement of preparing polyester fiber.

4. Conclusion

1. The performance of the cotton fiber wasn’t be affected too much during the alcoholysis process, it is remain meet the using requirements after the separation.
2. Reconstruction performance of pet has decreased, but still meets the recycling requirements.

References
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