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Recycling of waste PET for functionalised textile finishing

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The glycolysis of waste bottles made of polyethylene terephthalate (PET) has been carried out at 190°C for 8 h with sodium acetate catalyst. Products of reaction are then characterised by spectroscopy, thermogravimetry and calorimetry. The bis-hydroxyethyl terephthalate (BHET) obtained from the reaction is formulated using an emulsifying and dispersing agent and further utilised as an antistatic finishing chemical for imparting functional effect on polyester textile. The application has been performed by padding polyester fabric with a solution containing antistatic agents of different concertations (10, 20 and 40 g/L), followed by drying at 110° C for 60 s and then thermoset at 170°C for 40 s. The treated fabric is compared with a commercial antistatic agent for functional performance and then evaluated in terms of absorbency, wicking height, charge decay time, and hand feel. The results show that the fabric finished with 20 g/L BHET based formulation performs better than the commercial antistatic agent finished fabric, and the waste PET bottles can be effectively utilised as a textile finishing chemical.

Keywords: Antistatic effect, Auxiliary formulation, Functional finishing, PET valorisation, Polyethylene terephthalate, Polyester fabric, Waste treatment

1 Introduction

PET (polyethylene terephthalate) plastic is widely used for food and beverages packaging because of its properties like strength, lightweight, ease of handling, and cost economy[1]. The conversion of PET polymer to bottle is a simple and cheapest method compared to glass and aluminium bottle making. The bottles are made from PET polymer by the process of extrusion, solidification (cooling) and cutting as per the requirement of packing size[2,3].

Globally, around 500 million tons of plastic polymer is consumed as a single-use product and out of it, only 9 % goes for recycling. In contrast, the remaining 91 % is thrown as solid waste and goes for landfills or dumped in water bodies. Available data indicates that about 100 billion plastic bottles are sold only in the USA. Plastics dumped in the ocean is the biggest threat to aquatic life; as per the study, 100 % of marine animal muscles have contended microplastic[4]. In 2020, the consumption of PET bottles was 1.1 million tonnes and 0.99 million tonnes, respectively. The estimated annual value of India's organised PET recycling sector is between Rs 3,000 and Rs 4,000 crore. In India, organised players recycle 60 and 70 percent of PET bottles that are discarded. It should be noted that this estimate disregards PET used to create straps, sheets, and non-food containers as well as PET recycled by the unorganised sector. The overall PET recycling rate will be higher if the aforementioned categories are considered.

Recycling PET plastic waste into valuable products is considered the most prominent approach for reducing harmful environmental impact. Many researchers have worked on the valorisation of polyester waste and its utilisation in various end-use applications, such as making fibres for textile and clothing, using it as a matrix for reinforced composite and making paver blocks, packaging film, resin, plasticisers, etc[5-9]. The recycling of PET bottles is carried out by two methods, namely mechanical processing, and chemical synthesis. Mechanical recycling involves sorting, cleaning, shredding, melting, and extrusion for reforming plastic chips, while chemical recycling involves depolymerisation of plastic waste, its purification, and then re-polymerization for using it as a building block for chemical additives[10]. Valorisation of waste PET bottles by utilisation it as an auxiliary chemical for imparting functional properties on textile material is a novel concept and has not been extensively researched to the best of our knowledge. The possibility of converting waste PET into a finishing

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chemical for functionalising apparel clothing can pave the way for a large-scale reduction in solid waste management.

As per the report of precedence research, the textile auxiliaries' market was valued at US 7.2 $ billion in 2019 and is expected to grow at a CAGR of 4.2% and reach US $ 10 billion by 2027[11]. Such huge consumption would impact an increase in the pollution load of textile auxiliaries manufacturing industries. Considering these two significant problems, namely polyester waste and textile auxiliary manufacture pollution, the valorisation of PET waste into the textile auxiliaries would be considered a possible and sustainable solution.

Polyester fibre is hydrophobic in nature and tends to accumulate static charge which makes it uncomfortable to wear as the fabric clings to the body. For neutralising this static charge, it is necessary to deposite a compound on the fibre surface, which acts as a medium for charge dissipation. Application of antistatic agent on the polyester fabric helps to improve moisture retention and thereby reduce the static charge accumulation[12]. BHET is a product obtained by depolymerisation of PET, and it has the properties to hold moisture from the atmosphere[13]. Therefore, applying BHET in an adequately emulsified form as an antistatic agent on the polyester fabric could impart desired functionality.

Present research work is focused on the chemical recycling of waste PET bottles and converting them into BHET. Further, the process includes formulating the BHET with a suitable emulsifier and dispersing agent for developing a finishing chemical and then applying it on polyester fabric for imparting hydrophilic, antistatic effect and improving the static charge dissipation properties.

2 Materials and Methods

PET waste bottles were collected from local soft beverages shop in Matunga, Mumbai. 100 % polyester woven fabric was obtained from Sanjay Shah and Association, Mumbai. Ethylene glycol (EG), sodium acetate, Tween 80 (Sorbitan Monooleate - emulsifying agent) and acetic acid were purchased from SD Fine Chemical Ltd, Mumbai. DISPA WS Liquid (Naphthalene sulphonate - dispersing agent) was procured from Rossari Biotech Ltd, Mumbai and a commercial antistatic agent from a leading Indian textile chemical manufacturer.

2.1 Glycolysis of PET

The washed cut pieces of PET bottles were glycolyzed using EG by maintaining the molar ratio of PET repeating unit to EG at 1:6 and using 1% (w/w) sodium acetate as a catalyst. The mixture was poured into a three-necked flask, equipped with a reflux condenser, stirrer, and thermometer, and the reaction was run for 8 h at 190°C under stirring (Fig. 1). Then, the reaction mixture was filtered for separation of BHET, un-reacted EG and water-soluble oligomers. Further, the filtrate was concentrated, first by boiling and then by cooling, to get the white crystals of BHET. White crystalline powder of BHET was purified by repeated crystallisation from water and dried in an oven at 80°C for 4 h to remove the excess water. Then, the purified BHET was used for formulating the textile auxiliary chemical.

2.2 Preparation of Antistatic Agent

The BHET obtained from glycolysis was emulsified by mixing 10 % BHET, 1 % Tween 80 and 1 % Dispa WS at 1000 rpm. The homogenisation was performed for 60 min to get a stable emulsion. The formulated BHET was further used as an antistatic agent.

2.3 Application of Antistatic Agent on Polyester Fabric

100% Polyester fabric was treated with formulated BHET and a commercial antistatic agent at varying concentrations by the pad-dry-cure process. The fabric was dipped in a bath containing 10, 20 and 40 g/L of the finishing agent. The *p*H of the finishing bath was maintained in the range of 5 - 5.5 by the

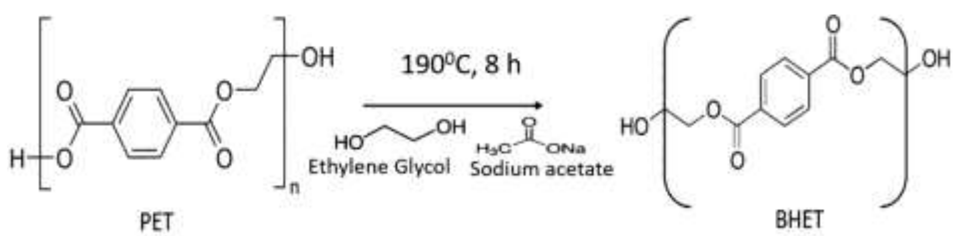

Fig. 1 — Glycolysis of PET

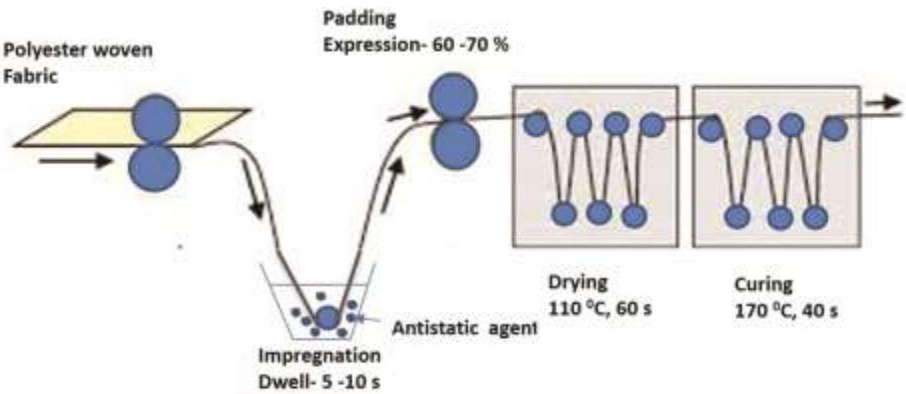

Fig. 2 — Application of antistatic agent on polyester fabric

addition of acetic acid. The percentage of the chemical pick-up was maintained at about 70 %. After padding, the fabric was dried in a hot air oven at 110°C for 60 s and cured on stenter at 170°C for 40 s. The schematic diagram is illustrated in Fig. 2.

2.4 Evaluation and Characterization

Purified BHET was analysed by Fourier Transform-Infrared (ATR-FTIR) using a pike miracle ATR module with diamond/ Zn Se crystal on FTIR-8400S (Bruker India Scientific Private Limited) using 4 cm^{-1} resolution and 45 consecutive scans in a wavenumber range of 400 - 3750 cm^{-1}. Differential scanning calorimetry (DSC) scan of the product was obtained using Shimadzu 60 at the heating rate of 10°C/min from 20-300°C in a nitrogen atmosphere. The thermogravimetric analysis (TGA) was performed using DTG-60H (SHIMADZU, Japan) to measure the product’s weight loss in a nitrogen atmosphere during a temperature range from 25 - 500°C, and at a heating rate of 10°C/min. The water absorbency of the fabric was measured by AATCC Drop Test -Method 79-2005. The wicking performance of fabric was evaluated by AATCC Test Methods 197-2011. The static charge decay time of polyester fabric was estimated by ASTM D 4238 method using the TREK model 156A charged plate monitor system. In all tests, average of 5 readings was reported in results. The handfeel was tested by a subjective evaluation method, which is generally used in industries where ratting is given by experienced people from textile backgrounds.

3 Results and Discussion

3.1 FTIR, DSC and TGA Studies of Recycled BHET

Figure 3 shows the FTIR spectra of the BHET obtained from PET glycolysis. The spectra of

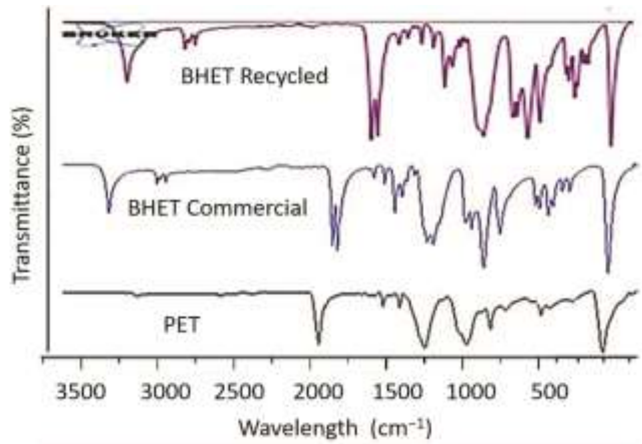

Fig. 3 — FTIR of recycled BHET

commercial BHET and PET in the figure confirm the conversion of PET to BHET. Compared with the FTIR curves of commercial BHET, no new peak was observed in the spectra of recycled BHET, indicating that no new chemical bonds are formed. In the curve of recycled BHET, the absorption peaks at 1689-1712 cm^{-1} and 700–800 cm^{-1} demonstrate benzene ring. The two peaks at 1282 cm^{-1} are attributed to the vibration of the C–O–C bond. The 1712 cm^{-1} peak is due to the C=O bond. The sharp peaks at 3446 cm^{-1} indicate the alcoholic [O-H] group. The two attached peaks at 2962 and 2877 cm^{-1} show the aliphatic carbon [C-H] group. The peak at 3446 cm^{-1} is much sharper than in PET curve. The main reason for this, is the increasing O–H bond in HOCH group of recycled and commercial BHET[14].

The DSC curves of the recycled BHET, commercial BHET and PET material are illustrated in Fig. 4. The DSC analysis is important to check the thermal characteristics of BHET obtained after glycolysis of PET waste and compared with commercial BHET. Its results show identical thermal behaviors, which means the chemical recycling does not alter the thermal behaviour of BHET. The DSC curve of PET shows a glass transition temperature at around 80°C, an exothermic event where heat is evolved at 100° -150°C, which causes cold crystallisation of PET, and a sharp endothermic pick at 250° - 260^{0}C shows the melting of PET[15]. The DSC curve of all three recycled BHET shows the same trend; there is a sharp endothermic peak at 105° C. Furthermore, the melting onset and the peak temperatures of BHET are 98^{0}C and 105°C respectively, which is very close with the information available in the literature[16] (105°C and 109°C).

The TGA curve of the PET material, recycled BHET and commercial BHET are illustrated in Fig. 5. The TGA curve of PET indicates significant weight loss at around 400°C, which is due to the thermal decomposition of PET. The TGA curve of all BHET products shows the same trend, where two apparent weight losses are exhibited. The first is approximately 30- 35% at an onset temperature of 230°C due to the thermal decomposition of BHET. After the first weight loss, heating in TGA causes re-polymerization of BHET to PET. The other weight loss is approximately 55-60% at an onset temperature of 400°C. The second weight loss is a thermal decomposition of PET produced by BHET thermal polymerisation during TGA[17].

3.2 Effect of Formulated BHET on Functional Properties of Polyester

Table 1 illustrates polyester fabric performance finished with a commercial antistatic agent and the recycled polyester BHET formulated product. Polyester fabric treated with an antistatic agent shows improvement in absorbency as compared to unfinished fabric. As compared with the commercial antistatic agent, BHET formulated product imparts excellent absorbency at the same concentration of 20 g/L, and a similar effect is observed in the case of wicking performance (Fig. 6). The effect on the antistatic performance of the BHET formulated product is much better than the commercial product based on the time taken for the voltage dissipation, which is less than 2 s. The full decay time means the time taken for the charge dissipation from 1000 volts to 0 volts, and the half decay time is for the charge dissipation from 1000 volts to 500 volts. The results indicate that the time required for the decay of charge

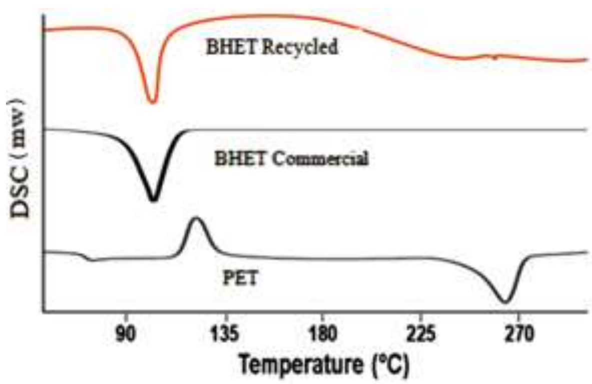

Fig. 4 — DSC of recycled BHET

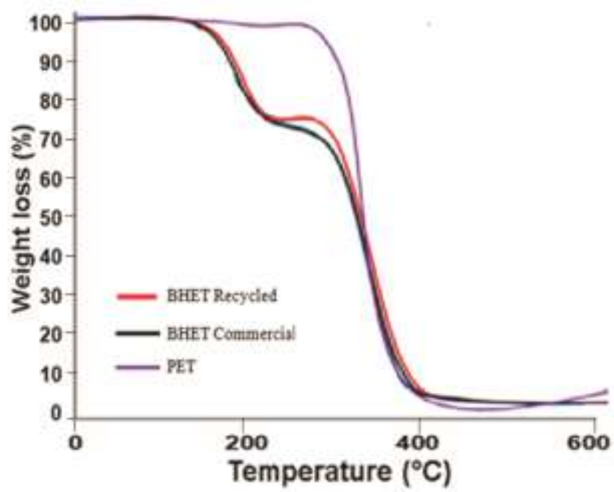

Fig. 5 — TGA of recycled BHET

Table 1 — Properties of polyester fabric finished with a commercial antistatic agent and BHET

Test parameter	Unfinished fabric	Commercial antistatic agent	BHET formulated antistatic agent		
		20 g/L	10 g/L	20 g/L	40 g/L
Absorbency, s	295	9	7	5	3
Wicking height, cm					
1 min	0	3.0	4.0	3.5	3.0
5 min	0.1	7.3	7.5	7.3	7.2
10 min	0.2	12.2	12.8	12.4	12.0
Decay time, s					
Half decay	463	13	9	0.9	0.66
Full decay	725	37	24	1.74	1.24
Hand feel (subjective)[a]	2	3.5	3.5	3.5	4.0

[a]Rating 1-5.

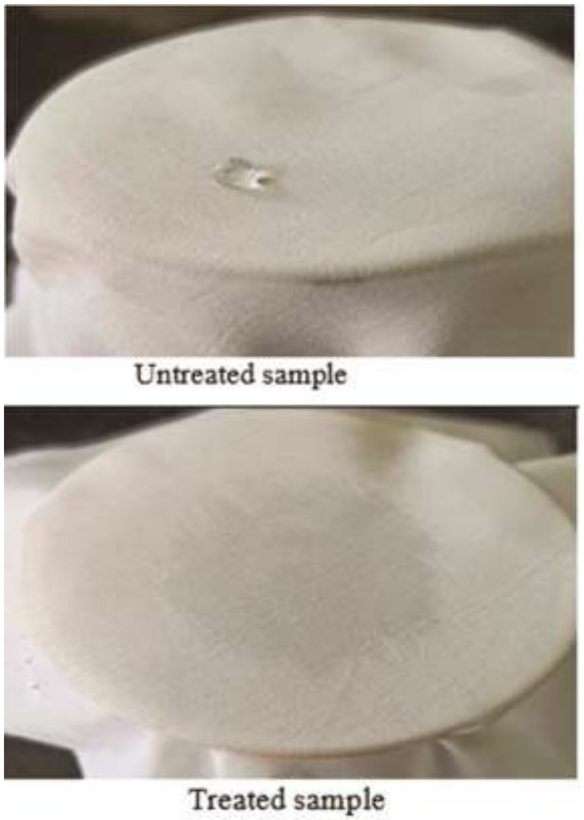

Fig. 6 — Effect of BHET on absorbency of polyester

is very high for the unfinished polyester fabric, which means that the untreated fabric does not have any tendency of charge dissipation due to its hydrophobic nature. The polyester fabric treated with a commercial antistatic agent of concentration 20 g/L shows 13 s for half decay and 37 s for full decay, which is very high as compared to 20 g/L for BHET formulated product treated fabric. This is mainly attributed to the enhanced moisture retention and water absorption properties, and hence charge dissipation is fast. Improvement in softness or hand feel of the antistatic agent treated cloth is also almost comparable. Thus, the overall performance of 20 g/L BHET formulated product treated fabric is better than the commercial antistatic agent.

4 Conclusion

The results indicate the viability and applicability of recycled BHET from PET bottle waste for its use as an antistatic agent in the polyester fabric finishing process. The comparative performance of the BHET formulated product (10 % BHET, 1 % Tween 80 and 1 % Dispa WS) against the commercially used antistatic agent in the textile processing industry shows that the hydrophilicity, static charge decay and softness are well within the acceptable limits and meet the desired functional performance parameters. Therefore, the recycling of waste PET bottles by the glycolysis route using sodium acetate as a catalyst and formulating the resultant BHET as an auxiliary chemical for textile finishing would not only reduce the solid waste accumulation problem but also pave the way for developing a value-added product.

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