RECOVERY OF TEREPTHALIC ACID MONOMER FROM WASTE OF POLYCOTTON MATERIAL USING GREENER METHOD

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ABSTRACT:
Polycotton fibre is combination of cotton and polyester. This fibre is very durable, wrinkle free, soft and cheaper than cotton, therefore bedsheets, towel, curtains and fashionable clothes of affordable price are made by this fibre. Due to huge population and modern lifestyle, consumption of Polycotton fabric is growing rapidly and with it, its waste is also increasing tremendously. Separation of both fibre and recovery of terephthalic acid from PET is possible by depolymerization by alkaline hydrolysis. Use of PTC catalyst tetra butyl ammonium bromide in alkaline hydrolysis serves the aim of Green chemistry. At only 100°C and in reflux time of 120 minutes recovery of TPA from (poly(ethylene terephthalate)) or 96% PET is possible.

KEYWORDS: Polycotton Waste, Depolymerization, Terephthalic Acid, Green Chemistry, Phase Transfer Catalyst

INTRODUCTION:
There has been rapid growth in the population of the countries like India and to meet the clothing requirement for its people, there has been rapid growth in textile industry as well. In the material that is used it is observed that in developing countries cost of finished product plays an important role. To cater the demand of clothing for large population only natural fibers like cotton cannot fulfill the demand, therefore manufacturing of synthetic fibre like polyester becomes very important. These synthetic fibres are easy to use and wash thus being cost effective, these have long life too but do not provide comfort like cotton clothes. The blending of fibres of cotton and polyester provides a solution here. It can serve the purpose of meeting the increased demand at lower cost to fulfill the demand of fashion industry. Cotton is a natural fibre having 88 to 96% cellulose, clothing of cotton gives best comfort to human body, it is breathable and good absorbent, it provides cooling effects in warm climate. Manufacturing of clothing from cotton is being done since ancient history, but the shelf life of these fibre is not very long. Due to fading of shades and cost cotton clothing is becoming unaffordable for common men. For production of cotton large amount of water and hazardous chemical in the form of insecticides are used as well. Polyester is a synthetic fibre made by terephthalic acid and glycol by condensation polymerization. It has ester bond due to carbon-carbon covalent bond attached with benzene ring structure. It is a petroleum derived product, Average shelf life of the fibre made from polyester is more than 100 years, these are very cheap, easy to dye and require less maintenance but do not give comfort like cotton. Combination of two fibres made by polyester and cotton is named as polycotton, generally 65% of cotton and 35% of polyester fibre blending gives best comfort.Having low cost and long shelf life, easy to wash and dry, gives it additional advantage. Poly cotton fibres are used in manufacturing of bed sheets, towels, curtains and low-cost
clothes which are very economical. Due to lower cost and with explosive increase in population and changing fashion trends its demand is growing very rapidly. Polycotton is named as PC yarn in industry. Abnormal demand also creates huge waste and best use of waste is very important to create wealth and to save environment. Separation and reuse of both the fibres is the need of the hour.

Many different methods are known to separate the fibres but in most of the case either one or both can lose their original nature.

**ROLE OF GREEN OR SUSTAINABLE CHEMISTRY**

There are 12 basic principles of green chemistry. Out of these, dependence on petroleum products must be discouraged, and use of selective catalyst which helps to reduce requirements and to make reaction more efficient must be used as much as it can be. Now only those catalysts are preferred which have little toxicity and are recyclable.

Another important use of green chemistry is to use waste to produce valuable products to save our environment. Polycotton is a combination of Polyester synthetic fibre and cotton natural fibre, to separate both of them and to reuse them has become very important for sustainable development. In order to apply these principles effectively, softer and cost-effective method is to use chemicals of lower concentration and at lower temperature. The behavior of cotton and polyester with different solvent was studied. It was reported that mild alkali does not alter the structure of the cotton fibre. It is also reported that when concentrated solution of alkali is used cotton swells. At moderate temperature polyesters have very low resistance, therefore polyesters can be depolymerized at moderately high temp.

Cotton is readily depolymerized in acidic environment and at lower concentration of alkali it does not lose its texture but at higher concentration its crystalline structure may change, it is due to oxidation of end groups of cellulose to carboxylic acids.

![Cottons Natural Fiber Molecular Structure of Repeating Unit](image)

**Polyester Fiber Composed of Linear Macromolecules of An Ester of adipol And Terephthalic Acid**

**Use of PTC for a greener approach**

Greener approach towards depolymerization of PET can be achieved by using phase transfer catalyst (PTC) because major advantages of PTC is that they work at lower temperature and without
any expensive solvent. PTCs can be used in presence of mild bases like aqueous alkali, metal hydroxides. They produce higher yields by suppression of side reactions.

The basic function of a PTC is to transfer reagents from one phase to another. Most popular of the PTCs are quaternary onium salts (ammonium or phosphonium salts). Essential requirements for PTC are catalyst must be cationic and should have enough organic structure, the stability of catalyst under reaction condition must be moderate, the effective cation and anion bonding should be loose enough to allow higher anion reactivity, and easy recovery of catalyst.

For our experiment quaternary ammonium halides (Q⁺X⁻) is dissolved in aqueous phase. This undergoes anion exchange with the anion of reactants dissolved in the aqueous solution. The ion pair thus formed can cross liquid-liquid interface due to its lipophilic nature and it diffuses from inter phase into organic phase. This step is the “phase transfer” step. In the organic phase, the anion of the ion pair being quite nucleophilic, undergoes a nucleophilic substitution reaction with the organic reagent forming the desired R⁻Y⁻, the catalyst is then returned to aqueous phase and the cycle continues.

In our experiment, tetra-n-butyl-bromide (TBAB) is chosen as PTC catalyst. It has bromide counterion used as PTC

**Experimental**

Three neck round bottom borosil flask of 1000 ml is selected to reflux well cut, well washed and dried polycotton waste. 50 gm NaOH in 500 ml distilled water was taken in the flask. On one of the necks of flask thermometer was fixed, on second neck reflux condenser while on third neck thermostat was fixed. Content of the flask was heated at different temperatures for 120 minutes. After heating the content was filtered to separate cotton and polyester precipitate. Every time filtrate was cooled and concentrated HCl was added very slowly with constant stirring until solution became acidic. The white precipitate of TPA thus collected was again filtered and washed many times and dried in oven at 100°C. After each iteration weight was observed. The process of heating, separation, collection of precipitate, drying and weighing was performed again and again at different temperatures.
Table No. 1: Conversion of polyester waste to Terephthalic acid with variation of temperature for 120 minutes

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<tr>
<th>Sl No</th>
<th>Temperature in °C</th>
<th>Percentage Conversion</th>
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<tr>
<td>1</td>
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<td>7</td>
<td>160</td>
<td>88</td>
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In the same process when 1 gm of phase transfer catalyst TBAB is added as catalyst the conversion of polyester into terephthalic acid was observed to be faster and at even lower temperature.

Table No 2: Conversion of polyester into terephthalic acid with variation of temperature for 120 minutes in presence of PTC TBAB

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<thead>
<tr>
<th>Sl No</th>
<th>Temperature in °C</th>
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CONCLUSION

When polycotton contents were heated with dilute alkali, polyester part of fibre was found to be partially or fully dissolved in NaOH and it came out as filtrate from which terephthalic acid (TPA) was recovered. Dilute alkali was used for separation and depolymerization of polyethylene terephthalate (PET). When polycotton waste was heated with dilute alkali reasonable conversion was observed at more than 120°C. When PTC (TBAB C₁₆H₃₆BrN) was used as catalyst, the reaction was carried out at less than 100°C as the melting point for TBAB is 100°C. This catalyst acts very similar to action of surfactants. It acts as bridge between organic waste (solid PET) and inorganic phase (dilute alkali solution). PTC is hydrophilic to get dissolved in aqueous phase yet lipophilic enough to get attached to solid particles of PET.

In absence of catalyst, recovery of TPA was observed to be 88% when the content was heated at 160°C for 120 minutes, however when 1 gm of PTC was added as catalyst, at 100°C itself 96% of TPA was recovered from the polyester part of polycotton fabric. Therefore, it can be deemed to be a good way to recover maximum TPA monomer from polycotton fabric.

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