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A Rapid, Economical, and Eco-Friendly Method to Recycle Terephthalic Acid from Waste Poly (Ethylene Terphthalate) Bottles

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A method has been proposed to rapidly degrade waste PET (polyethylene terphthalate) bottles and recover terephthalic acid (TPA) in good yield. The method involves fusion of PET bottle chips with caustic soda in a china-clay crucible placed on the silicon carbide (SiC) slab by exposure to microwaves in a domestic oven. The PET waste was converted to water-soluble sodium salt of TPA in just 4 minutes. Afterwards, the solution was acidified with HCl to recover TPA in high yield. The method can be used to design environmentally benign and fast industrial prototypes to recycle terephthalic acid from waste PET bottles.

Keywords depolymerization, microwaves, PET waste, recycling, terephthalic acid

1. INTRODUCTION

Poly (ethylene terephthalate), commonly known as PET, is often used in manufacturing of soft drink bottles, audio and video films, thermoformed articles, as well as textile fibers [1]. Because of its light weight, transparency, easy moldability, and good strength, PET is considered one of the best packing plastics [1]. Around the world, used PET articles and bottles are not properly recycled/degraded and usually end as landfill refuse. It is necessary to recycle

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PET bottles and articles to reuse the material out of which they are made and to reduce the landfill loads. In many countries, PET plastics have resin identification code number "1" inside the universal recycling symbol, usually found on the bottom of the container. In recent years, PET waste recycling has gained significant attention because of increasing environmental concerns. PET can be depolymerized by hydrolysis, alcoholysis, trans-esterification, and glycolysis [2–6]. Depolymerization of PET usually gives terephthalic acid (TPA), dimethyl terephthalate, and ethylene glycol. Scientists around the world are trying to develop a quick and efficient method to recycle/degrade PET [7–12]. Accordingly, performance of various catalysts [13], different solvent systems [14,15], and chemicals [1,9,12,16] has been tested to evaluate their ability to degrade PET.

In our previous work [1], it was found that when PET chips were heated to dryness in the presence of concentrated aqueous solution of sodium hydroxide, terephthalic acid (TPA) has been recovered up to 99% in nearly 10 minutes. In the present work, the same reaction was carried out in a china clay crucible placed on a silicon carbide (SiC) slab that was heated with 2.45 GHz microwaves in a domestic microwave oven. The reaction time has been reduced to 4 min without lowering the yield. The method can be used to design environmentally benign industrial prototypes for quick recycling of terephthalic acid from waste PET bottles.

2. EXPERIMENTAL

Laboratory-grade sodium hydroxide, hydrochloric acid, deionized water, and AnalaR grade terephthalic acid (TPA) were used. High-quality TPA was employed as reference during IR analysis to compare its spectra with the spectra of recovered TPA. Used PET bottles (colorless) were washed thoroughly with distilled water, dried in an oven, and cut into small chips of square shape having a length of 0.5 inch. PET chips (4.0 g) and sodium hydroxide (10.0 g) were taken in a china clay crucible. Afterwards, the crucible was placed on a SiC slab of square shape having 4-inch length and 0.5-inch height. The set of crucible and slab was positioned in front of a magnetron of a domestic microwave oven working at full power and giving 2.45 GHz microwaves. As the glass tray of the oven cannot bear high temperature, it has been removed and the set was placed on a refractory brick. Within two minutes, the SiC slab got red-hot and all the mass in the crucible became melted. Fusion was carried out for a further two minutes and then the contents of the crucible (after cooling at room temperature for 5 min) were poured in deionized water (100 ml). Concentrated hydrochloric acid was added drop-wise until the pH of the solution become slightly acidic. White crystals of TPA were precipitated out and were filtered and dried. Melting/sublimation point, solubility, and IR analysis were employed to characterize recycled TPA. The experiment was repeated three times to check the reproducibility.

3. RESULTS AND DISCUSSION

Microwave heating has taken an incontestable place in analytical and organic laboratories as an effective and non-polluting method of activation. Silicon carbide is a good microwave susceptor. It can absorb microwaves and turn them into heat like food items but the rate of heating- and high-temperature-withstanding capacity of SiC is much greater than that of later and other ordinary materials [17]. Silicon carbide ceramics have a high dielectric loss at room temperature. Microwave muffle furnaces are available based on oven linings made of SiC. SiC rapidly heats up the oven's cavity to fusion temperatures. Microwave heating increases the rate of certain chemical reactions by 10 to 1,000 times when compared to conventional heating. This is because of its ability to substantially increase the temperature of a reaction. SiC heated with microwaves is a clean and environmentally benign source of energy because no greenhouse gas or other side-products are produced. The use of solvents in the chemical reaction can also be reduced or removed during microwave heating. Reactions with microwave heating are more reproducible because of better control of process parameters. In the present work, silicon carbide slab, exposed to microwaves, has been employed as an energy source to carry out fusion of waste PET to recover terephthalic acid.

In our previous work [1], it was found that alkaline hydrolysis of the PET leads to better results in comparison to acidic hydrolysis. In the present work, caustic soda was melted in a china clay crucible by heating on a SiC slab that was heated with microwaves of 2.45 GHz frequency. PET chips were fused with molten caustic soda and then the fused mass was dissolved in water. Later, TPA was recovered from the water solution by acidifying it with concentrated hydrochloric acid. Yield was found to be $96 \pm 2\%$ (repeated three times). Recovered terephthalic acid (TPA) has a crystal-white appearance which sublimed in the temperature range 297–301°C. When heated in a sealed tube,

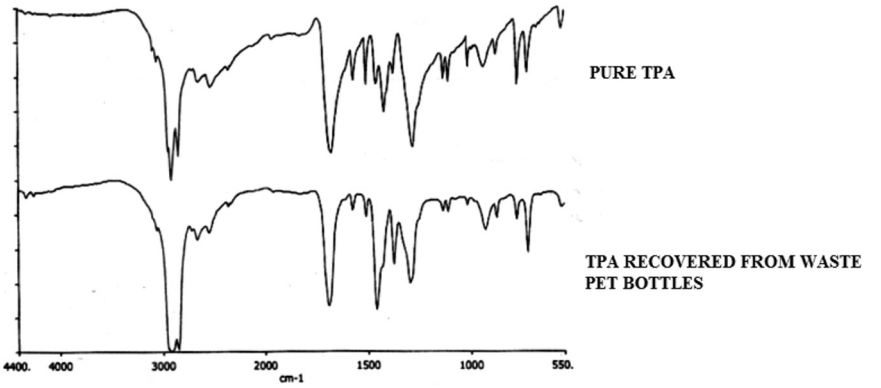

Figure 1: Comparison of IR spectra of recycled terephthalic acid (TPA) with that of pure TPA.

it melted at 428°C. The product had good solubility in sulfuric acid, pyridine, and dimethylformamide while it was insoluble in acetone, ether, and water. FTIR analysis was carried out on a PerkinElmer BX model with ATR system of sample evaluation. Figure 1 represents the comparison of the IR scan of recovered TPA with that of the pure. The figure evidently shows the recovered product was pure terephthalic acid. In addition, CHN analysis was carried out on a CHNS Elemental Analyzer (Gerhard; Helium and Oxygen) to confirm the product that suggested the product had 56-58% carbon, 4-6% hydrogen, and 36–40% oxygen.

4. SUMMARY

A quick, environmentally friendly and efficient method has been proposed to degrade waste PET (polyethylene terphthalate) bottles and to recycle valuable terephthalic acid in good yield. Fusion of PET bottle chips with caustic soda was carried out in a china clay crucible placed on silicon carbide, SiC slab, and exposed to microwaves in a domestic oven. The PET bottles were converted to water-soluble sodium salt of terephthalic acid in just 4 minutes. Afterwards, the solution was acidified with HCl to recover terephthalic acid in high yield. Melting/sublimation point, solubility, IR, and CHN analysis have confirmed the high purity of recycled TPA. The method can be used to design environmentally benign industrial prototypes for recycling of terephthalic acid from waste PET bottles.

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