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A Facile Route to Catalytic Degradation of Polystyrene over Zeolite and Barium Carbonate

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The present work investigated the catalytic degradation of polystyrene over zeolite and barium carbonate catalysts under heat reflux. The spectroscopic analysis confirmed the formation of alkyl benzene on degradation of polystyrene over both zeolite and $BaCO_3$ catalysts. The thermal stability of the polystyrene products after degradation was found to be lower compared to the pure polystyrene. The crystallinity and morphology of the polystyrene products before and after degradation were also examined using X-ray diffraction and scanning electron microscopy. In addition, a plausible mechanism of the degradation pathway of polystyrene over zeolite and $BaCO_3$ was elucidated. The degradation of polystyrene over zeolite produced ethyl benzene and cumene with a high selectivity while styrene monomer was obtained in degradation of polystyrene catalyzed by $BaCO_3$.

Keywords: Polystyrene, Catalytic degradation, Zeolite, Barium carbonate, Degradation mechanism.

INTRODUCTION

With the ever-increasing demand of convenient plastic products in our modern life such as toys, packaging, containers, home appliances, utensils, electronics, personal products, constructions, agricultural goods and so on, the production of plastics is increasing constantly [1,2]. Beginning at 1950s, worldwide plastic production flourished commercially from nearly 1.5 million metric tons to about 359 million metric tons today [3,4]. In developing countries, especially in Bangladesh, the per capita plastic consumption has expanded drastically from 2.07 kg (in year 2005) to 4.5 kg (in year 2014) [5,6]. At present, various plastic materials such as low density polyethylene (LDPE), high density polyethylene (HDPE), polypropylene (PP), polyvinyl chloride (PVC), polyethylene terephthalate (PET), polystyrene (PS), *etc.* are being manufactured in the industries globally. Among the available commercial plastic materials, the production and usage of polystyrene have expanded significantly owing to its cost-efficiency, excellent chemical and thermal stability, lightweight, resilience and longevity [7].

Although, polystyrene and other plastics greatly facilitate the industrial production of consumer goods, the disposal of plastic wastes causes serious environmental and ecological pollution due to its non-degradable nature [8]. In Bangladesh, around 3000 tons of plastic waste are generated per day, which is about 8% of the total volume of waste generated [9]. The hazard of disposing of plastic waste recognized as “white pollution” has become a serious global concern in recent years. The presence of plastics and microplastics in the environment, lands and coastal areas is reported to be increasing alarmingly in developing countries like Bangladesh, despite producing less plastic waste than in developed countries [8]. Thus, recycling of plastic wastes into environmentally friendly materials is essential for saving the eco-system.

To date, numerous scientific and commercial efforts have been made to recycle the polystyrene waste into eco-friendly materials like fuel oils using various degradation techniques *e.g.* thermal, chemical, photodegradation and catalytic degradation methods [10-14]. Among the various methods, at present, catalytic degradation of polystyrene under acidic or alkaline conditions has received an intense attention by the researchers due to its simple implications compared to the thermal degradation which requires high operational temperature upto 900 ºC and sophisticated equipment [1]. Until now, several solid materials which are typically known to have ability to cleave C-C bonds, including metal, metal oxides, for instances, Al_2O_3, SiO_2, Mg, MgO, K_2O, CaO, BaO, HgO, ZnO, MnO_2, CuO/Al_2O_3, TiO_2, Fe–K/Al_2O_3, zeolites (HNZ, ZSM-5, FCC, Al-MCM-41), mesoporous materials (K_2O/Si-MCM-41, K_2O-BaO/MCM-4, MCM-41) and clays have been used as catalyst for polystyrene degradation [2,15-20].

Ukei *et al.* [14] demonstrated the catalytic degradation of polystyrene into styrene (both monomer and dimer) over BaO catalysts. Later on, Tiwary & Guria [15] used eight different metal oxides, CaO, MnO_2, TiO_2, PbO, Fe_2O_3, HgO, ZnO, CrO_3 as catalysts for the degradation of polystyrene in benzene solution at an elevated temperature. Among the various oxides, MnO_2 showed remarkable catalytic ability in polystyrene degradation. Meanwhile, Shah *et al.* [19] reported the catalytic degradation of polystyrene into liquid products and styrene monomer using Mg, MgO and $MgCO_3$ catalysts. It was found that Mg exhibits the best catalytic performance toward polystyrene degradation. Recently, Singh [20] successfully conducted the degradation of virgin polystyrene into green aromatic derivatives *via* pyro-lysis using $BaCO_3$ nanomaterials. Besides metal oxides and carbonates, zeolites are now being considered as a potential catalyst for the plastic degradation [13,21,22]. Lee *et al.* [21] investigated the degradation of polystyrene over natural zeolite (HNZ) catalyst at 400 ºC. The major product after the poly-styrene degradation was found to be styrene along with minor products of oils of C_5-C_{12}. In another study, Chumbhale *et al.* [22] reported the catalytic degradation of polystyrene waste into styrene, styrene dimer and ethylbenzene using mordenite zeolite catalyst.

Thus, it is evident that metal carbonates and zeolite could be used as catalysts to convert polystyrene into various products. However, a systematic approach to clearly understand the mechanism of degradation of polystyrene in acids or bases clearly is yet to be explored. In present work, the catalytic degradation of polystyrene over zeolite and $BaCO_3$ was investigated and also compared their catalytic activity. Furthermore, a plausible mechanism of polystyrene degradation over zeolite and $BaCO_3$ is also elucidated.

EXPERIMENTAL

Solid crystalline pellets of atactic polystyrene (*m.w.* 125,000-250,000) was purchased from Alfa Aesar (A Johnson Matthey Company, U.K.). The technical grade crystalline zeolite was procured form Spectrum (USA) and used without further purification. The $BaCO_3$ (Merck, Germany) and tetrahydrofuran (Merck, Germany) were used as received.

Catalytic degradation of polystyrene: Pure polystyrene pellets (0.20 g) were dissolved in 20 mL of distilled THF in a two-necked flask using an ultrasonic bath. Then, the catalysts, $BaCO_3$ powder (0.077 g) or zeolite (0.018 g) dissolved in 1 mL 3 M HCl were added to polystyrene and THF mixture. The reaction mixture was heated under reflux at 66-78 ºC for 94 h. After refluxing, the reaction mixture was cooled at room temperature. The product obtained after the degradation of polystyrene was separated by solvent extraction. In this connection, 50 mL of water and 100 mL of diethyl ether were added to the reaction mixture. The mixture was then placed in a separating funnel and shaken thoroughly until two distinct layers (aqueous and organic) appeared. About 50 mL of diethyl ether was added into the aqueous layer again and shaken thoroughly. The by-products such as salts were preferably dissolved in water, while the diethyl ether uptook the organic components. This process was repeated three times in order to separate the organic product. Finally, the solvent was evaporated in a rotatory evaporator to get the solid oxidized polystyrene products. The obtained product was rinsed with water to remove trace of remaining acid or salts and dried in an oven at 70 ºC for 94 h.

Characterization: The degradation process and products were assessed using several techniques like Fourier transform infrared (FT-IR) spectroscopy, scanning electron microscopy (SEM), X-ray diffraction (XRD) and thermogravimetric analyses (TGA). FT-IR spectra were recorded on a FT-IR spectrometer (Perkin-Elmer) in the range of 4000-400 cm^{-1} wavenumber to observe the change of functional groups in polystyrene in the catalytic degradation process. The thermal stability of polystyrene before and after degradation was investigated using TG analysis under nitrogen environment with a thermogravimetric analyzer (Hitachi TG/DTA 7200). The sample was heated from 35 to 500 ºC with heating rate of 10 ºC/min using aluminum pans. The crystallinity of the product was examined using X-ray diffraction (PANalytical– X 'Pert' Pro) analyzer. The morphology and microstructure of the pure polystyrene and polystyrene after degradation were analyzed using scanning electron microscopy (ZEISS, EVO 18).

RESULTS AND DISCUSSION

FT-IR studies: Fig. 1 shows the FT-IR spectra of polystyrene and degraded products of polystyrene by the catalysts zeolite and $BaCO_3$. Several distinct bands at 3026, 2920.7, 2849.3, 1600.7, 1491.7, 1450.3, 754.6 and 698.2 cm^{-1} were observed in each of the polystyrene samples. The FT-IR data of polystyrene sample is consistent with the reported results [14]. The sharp bands in the range of 3100-3000 cm^{-1} indicated the presence of C-H stretching vibration of aromatic ring. The bands in the region of 2925-2917 and 2853-2849.3 cm^{-1} are evident of the presence of C-H asymmetric stretching and symmetric stretching vibration of methylene ($-CH_2-$) group, respectively [14]. Three peaks in the range of 1600.7-1450.3 cm^{-1} corresponded to the C=C stretching vibration of aromatic ring. The four small bands in the region of 2000-1650 cm^{-1} are overtone and combination bands for typical aromatic rings and intense peaks in the region of 803.5-698.2 cm^{-1} confirmed the presence of C-H out-of-plane bending vibration of aromatic ring indicating the presence of a benzene ring. A new band observed at *ca.* 2962 cm^{-1} presumably attributed to the asymmetric stretching vibration of methyl ($-CH_3$) group suggesting formation of alkyl benzenes after catalytic degradation. The significant enhancement in peak intensity at transmittance band *ca.* 2962 cm^{-1} for the polystyrene product obtained by zeolite catalyzed degradation, suggests that zeolite facilitated the production of alkyl

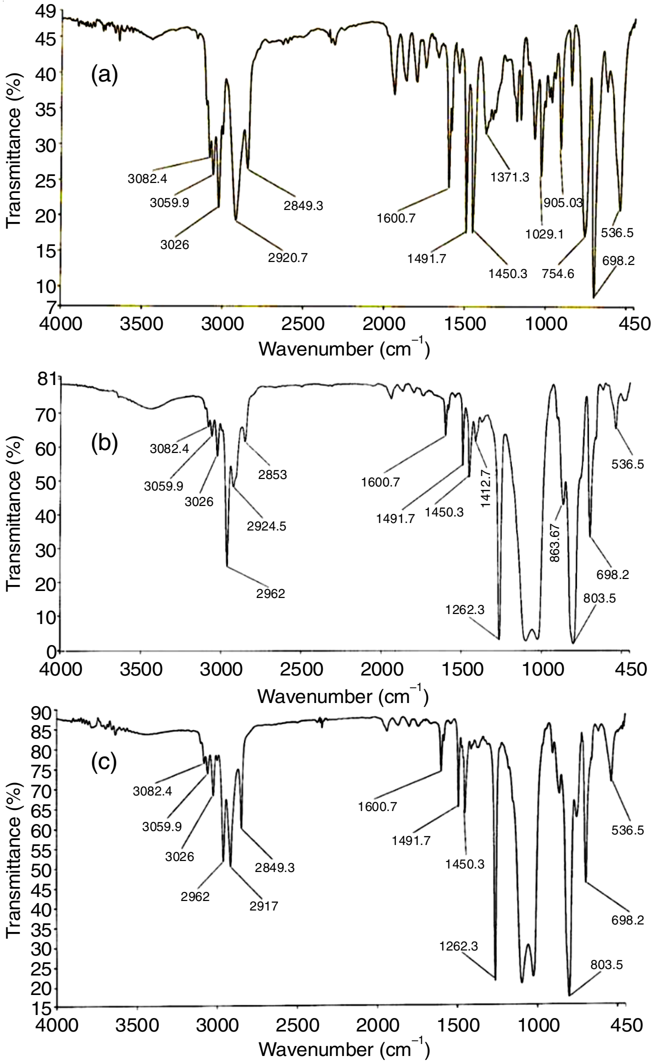


Fig. 1. FTIR spectra of (a) pure polystyrene, (b) polystyrene degraded by zeolite and (c) polystyrene degraded by $BaCO_3$

aromatic compounds. In contrast, for $BaCO_3$ catalyzed polystyrene degradation product, the peak intensities corresponding to the aromatic C–H bond vibrations, aromatic C=C stretching in the region of 3100-3000 and 1600.7-1450.3 cm^{-1}, respectively, were decreased compared to the zeolite catalyzed product. The decreasing trends in peak intensities for $BaCO_3$ catalyzed polystyrene product indicated decrease in the production of some aromatic compounds suggesting in the presence of $BaCO_3$. The zeolite exhibits superior performance for recovering alkyl benzenes possibly due to the formation of carbocations that consequently promote the production of ethyl benzene, toluene and cumene derivatives during degradation of polystyrene [14,23].

XRD studies: The XRD data are recorded to analyze the crystallinity of the polystyrene degraded product. Fig. 2 displays the XRD patterns of pure polystyrene and polystyrene degraded by zeolite and $BaCO_3$, which indicate that all of them have amorphous nature. No characteristic peak for crystalline polystyrene was observed in the measured 2θ region. The peak obtained at 2θ value of 18.36º corresponded to the amorphous nature of polystyrene. Previously, the similar XRD pattern of pure polystyrene having amorphous nature has also been reported by Chaudhary *et al.* [24] and Chen *et al.* [25]. The XRD patterns of polystyrene samples degraded by zeolite and $BaCO_3$ are almost identical to the pure polystyrene. However, a dwindling trend in the peak intensity at 2θ = 18.36º was observed for both zeolite and $BaCO_3$ degraded polystyrene products. There was no obvious change in XRD pattern in terms of appearance of new peaks and/or peak shift in the presence of catalysts. The crystallinity index (Cr. I%) values of polystyrene before and after degradation products by zeolite and $BaCO_3$ were estimated from the ratio of diffraction from a crystalline region to the total diffraction of the samples [26]. The Cr. I% values were calculated to be *ca.* 25.47, 19.03 and 20.96% for pure polystyrene, polystyrene-zeolite and polystyrene-$BaCO_3$, respectively. The decrease in the Cr. I% values for both polystyrene-zeolite and polystyrene-$BaCO_3$ confirmed the degree of deterioration of the original compound, pure polystyrene after degradation. The amorphous phase of polystyrene grows significantly after degradation by both zeolite and $BaCO_3$ catalysts. In addition, polystyrene-zeolite exhibited lower Cr. I% value indicating the zeolite catalyst has a significant impact on the crystallinity in the degraded polystyrene products. The FTIR data (*vide supra*) also confirmed the remarkable influence of zeolite on the degradation of polystyrene. To understand the effect of catalysts, the further morphological characterization of the polystyrene degraded products was carried out using SEM (*vide infra*).

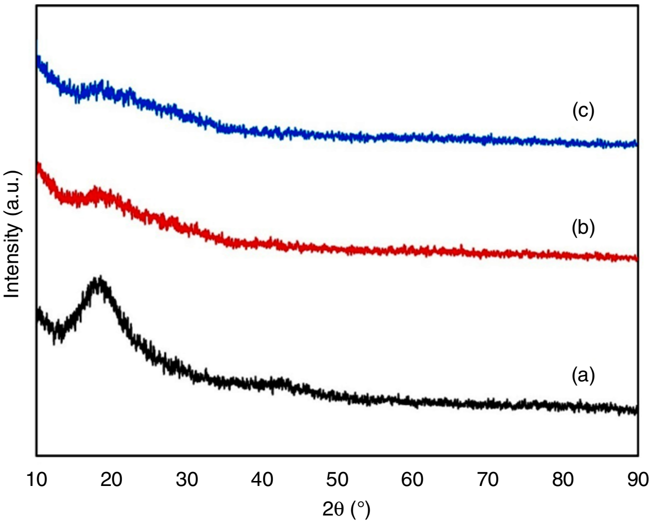


Fig. 2. XRD patterns of (a) pure polystyrene, (b) polystyrene degraded by zeolite, and (c) polystyrene degraded by $BaCO_3$

SEM study: The SEM images of polystyrene before and after degradation are depicted in Fig. 3 a-c. From Fig. 3a, the pure polystyrene samples show a flakes of micrometer sized polystyrene particles distributed randomly. The samples after degradation show an obvious morphological variation in the polystyrene surface. For polystyrene degradation products, significant cracks and pores were appeared (Fig. 3b-c). Moreover, a nucleation growth in the particles was observed which resulted in a coarse structure of the particles on the polystyrene surface. The roughness of the polystyrene surface was also increased after degradation by zeolite and $BaCO_3$ catalysts. The increased particle size could be associated with the pheno-

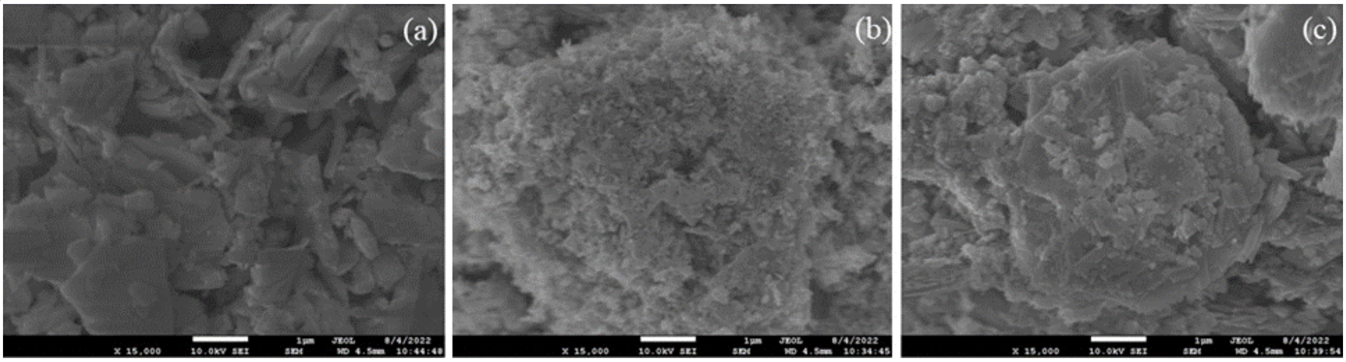


Fig. 3. SEM images of (a) pure polystyrene, (b) polystyrene degraded by zeolite, and (c) polystyrene degraded by $BaCO_3$

menon of coalescence that occurred more easily on the polystyrene after degradation [27].

Thermal analysis: The thermal analysis was employed to determine the content of any residual solvent or compounds in the polystyrene degradation products and the obtained TGA curves are shown in Fig. 4. Pure polystyrene shows a single stage weight loss process between 314-500 ºC and the maximum weight loss occurred at 442 ºC with loss of 96.43% of its initial weight. The single step weight loss is related to the presence of a carbon-carbon bond, which encourages the random scission at elevated temperature [28,29]. From Fig. 4, the polystyrene materials started to decompose at 314 ºC and the remaining weight of about 3.57% left unchanged in the temperature region of 442-506 ºC. In case of degradation catalyzed by zeolite, three stages of thermal degradation were observed and the temperature at which solid degradation products begin to decompose was about 140 ºC. The second weight loss step occurred in the temperature range of 200-400 ºC and consequently reached to final maximum transition temperature at 506 ºC. The residual weight of the degraded product was about 18.86% at 506 ºC. Similarly, for $BaCO_3$ catalyzed polystyrene degraded product, decomposition of solid products began at about 140.19 ºC and final weight loss was observed in the programmed temperature range of 431-506 ºC. The weight loss and residual weight of the polystyrene-$BaCO_3$ were estimated to be 79% and 21%, respectively. According to TG analysis results, the degraded products show a lower initial degradation temperature compared to the pure polystyrene sample revealing that the thermal stability of polystyrene sample decreases profoundly after degradation. The reduced thermal stability might be attributed to the changes in crystallinity, molecular weight and polymer chain length [30]. The decrease in thermal stability of polystyrene might be corresponded to changes and breaking of bonds caused by the catalytic activity of zeolite and $BaCO_3$.

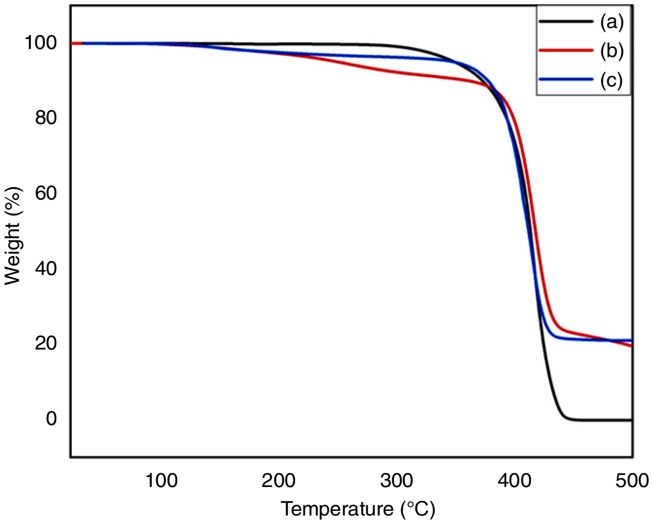
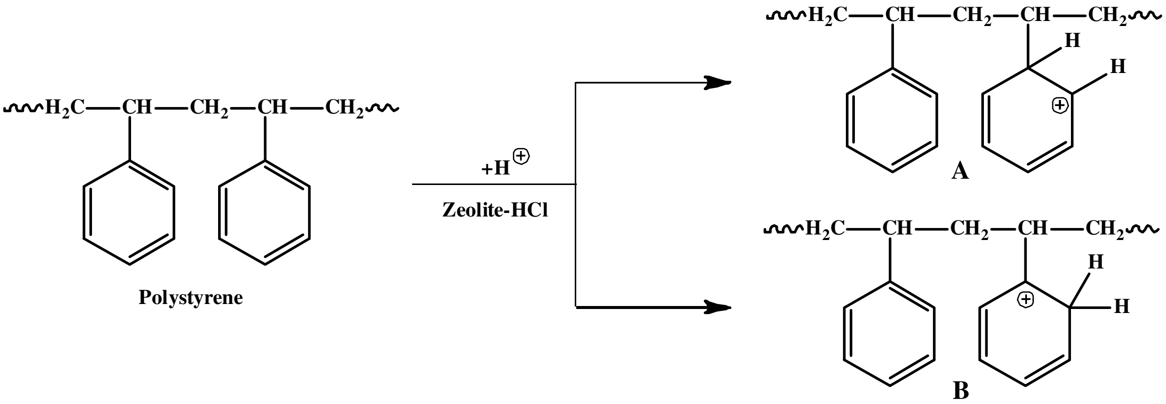
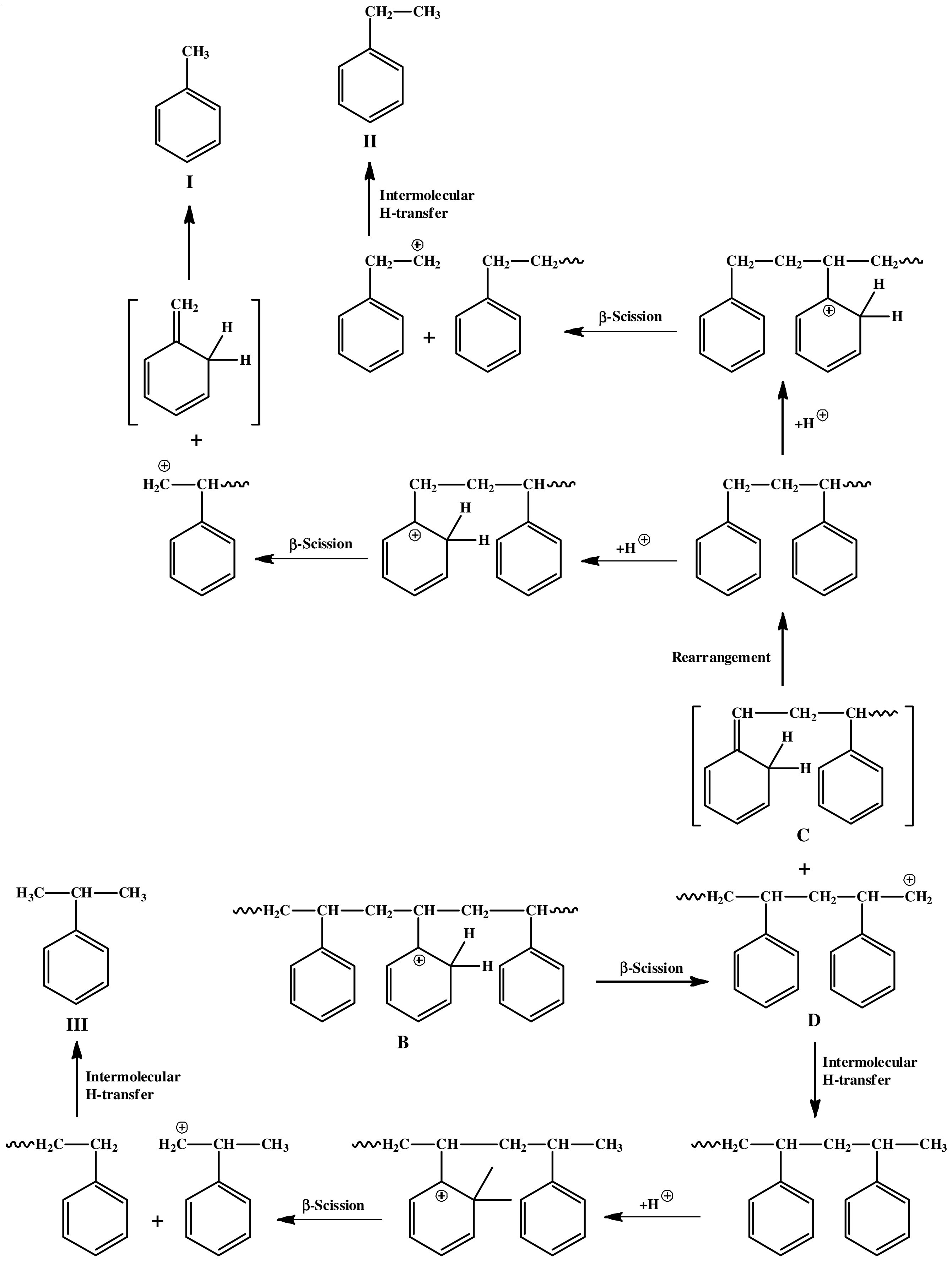


Fig. 4. TG curves of (a) pure polystyrene, (b) polystyrene degraded by zeolite, and (c) polystyrene degraded by $BaCO_3$

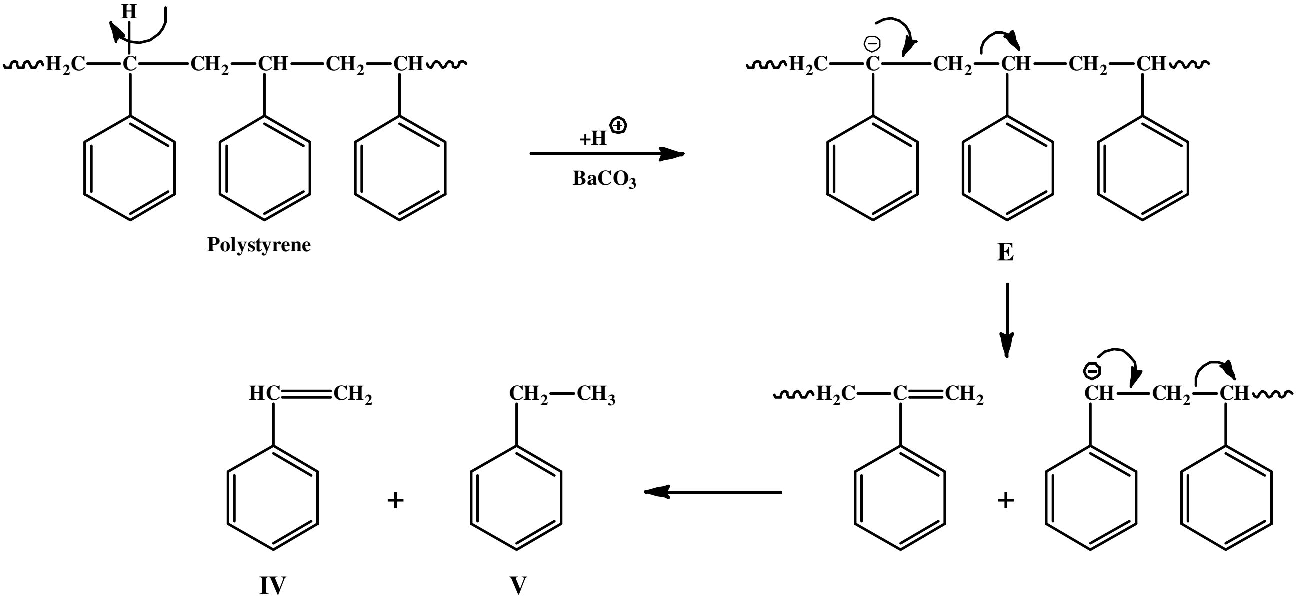
The present work, proposed a plausible mechanism for the degradation of polystyrene in the attendance of zeolite and $BaCO_3$ catalysts. Zeolite is considered to exhibit a high proton donor strength which is associated with AlO_4 tetrahedra having the smallest number of Al atoms. Thus, it can act as a Brønsted



Scheme-I: Protonation of aromatic ring in polystyrene, (A) C-1 protonation (B) C-2 protonation



Scheme-II: Possible degradative pathways of polystyrene using zeolite catalyst



Scheme-III: Possible degradative pathways of polystyrene using $BaCO_3$ as catalyst

acid. Protons may preferentially attack the C-1 and C-2 ring positions because the aliphatic polymer backbone is electron releasing group for the aromatic rings as presented in **Scheme-I**.

An electrophilic attack of proton to the aromatic ring may give rise to the two ions (A) and (B). Benzene is produced from ion (A) together with a polycation, as a result of β-scission. Polycation (B) may undergo a β-scission to give a polymer ion with the charge on the last carbon atom of the chain (D) and a cyclodiene substituted polymer (C), which in the reaction conditions, may rearrange to an aromatic ring (**Scheme-II**). Further protonation of (C) may lead to the formation of toluene (I) and ethylbenzene (II) and intermolecular hydrogen transfer on (D) can regulate the formation of isopropyl benzene (cumene) (III) [23].

When $BaCO_3$ is introduced to polystyrene as a catalyst, polyanions (E) may be generated by the elimination of hydrogen atoms from the polystyrene surface (**Scheme-III**). These polyanions may lead to the formation of styrene (IV) along with smaller polyanions (F). The eliminated hydrogens from polystyrene, absorbed on the base sites of $BaCO_3$, may dissociate on heating. These hydrogens may lead to the formation of small fragments of polystyrene (G) along with alkylbenzene moieties (V) as displayed in **Scheme-III** [14]. The formation of alkylbenzene moieties, especially ethyl benzene, has been developed by some kinetic models and measurement of energy involved in the transition state, however, exact routes are still to be explored [31].

Conclusion

The present work has demonstrated a facile and efficient chemical degradation method for polystyrene using zeolite and $BaCO_3$ as catalysts. The spectroscopic and thermal analysis results revealed that zeolite has superior catalytic activity compared to $BaCO_3$ toward the degradation of polystyrene. The polystyrene degradation is preferably selective to alkyl aromatics compounds in the presence of zeolite and $BaCO_3$. This study also demonstrates a promising route for the recycling and recovery of alkyl aromatics compounds, which could open routes for diverse applications in scientific research as well as in industry.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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