Catalytic degradation of high density polyethylene over mesoporous and microporous catalysts in a fluidised-bed reactor

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Abstract

High density polyethylene (HDPE) was pyrolysed over various catalysts using a laboratory fluidised-bed reactor operating isothermally at ambient pressure. HZSM-5 catalysed degradation resulted in much larger amounts of volatile hydrocarbons compared with degradation over non-zeolitic catalysts (MCM-41 and SAHA). When an HZSM-5 was used as a cracking additive in combination with a non-zeolitic catalyst (MCM-41 and SiO 2-Al 2 O 3 ), the solid mixed catalysts produced less gas with a lower loss of gasoline than HZSM-5. MCM-41 with large mesopores and SAHA with weaker acid sites resulted in a highly olefinic product and gave rise to the broadest carbon range of C 3-C 7. Both SAHA and MCM-41 materials allow bulky reactions to occur leading to the generation of coke and subsequently deactivation of the catalyst. This paper presents the conversion of polymers to useful hydrocarbons using various cracking catalysts and attempts to provide a basis for optimising the potential benefit of catalytic polymer recycling.

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Keywords: Catalytic degradation; Polymers; Catalysts; Fluidised-bed reactor; Selectivity

1. Introduction

The destruction of polymer wastes by incineration is prevalent, but this practice is expensive and often generates problems with unacceptable emissions. Decreasing availability and increasing cost of landfill makes polymer recycling more favourable. Conversion of waste polymers to useful chemical feedstocks or fuels may be promising as a solution to their growing environmental problem and as a viable alternative to fossil fuels [1,2]. Possible technologies for the conversion of waste to useful products have attracted research in the area of thermal degradation [3–5]. However, the thermal degradation of polymers to low molecular weight materials requires high temperatures typically more than 500 °C and even up to 900 °C, and has a major drawback in that a very broad product range is obtained. Catalytic pyrolysis provides a means to address these problems [6–11]. Our previous studies on the degradation of polymers in a fluidised-bed noted that, when catalysts are used, degradation takes place at much lower temperatures, yielding large amounts of volatile products [12–14].

Catalytic reactions of polymer degradation for producing valuable hydrocarbons have been carried out with emphasis on the effect of zeolitic catalysts [15–18]. Much less is known about performance of non-zeolitic catalyst in polymer degradation. Since the introduction of catalytic cracking, the initial catalysts were clays and amorphous silica–alumina and evolved with time to zeolites, particularly the use of REY and, finally, to the USY, REUSY and multi-zeolite catalysts used today. In this sense, HZSM-5 has been used in recent years as an
additive to boost gasoline octane and to increase the yield of propylene at the expense of gasoline. Recently, a new family of mesoporous aluminosilicates with a regular array of uniform pores of 2–20 nm has been discovered [19]. These MCM materials can be synthesised over a large range of framework Si/Al ratios and can develop mild acidity. The extra-large pore material containing aluminium offers interesting possibilities while considering heavier feedstocks, such as “bottoms cracking” [20]. The presence of these very large uniform pores combined with acidic properties may open alternative possibilities in the field of solid acid catalysis. Potential concepts of the MCM-41 catalysts have been investigated using thermal analysis to estimate the amount of catalytic degradation of the polymer and to predict kinetic behaviors [21,22].

Most studies refer to temperatures in excess of 450 °C and utilise batch or fixed-bed reactors [23–26]. Although catalysis has been used, this often involves thermal cracking of the polymer followed by catalytic conversion of the degradation products. However, the configuration of the pyrolysis-reforming reactors poses serious engineering and economics constraints. Problems associated with blockage and limited polymer/catalyst contact within the reactor make continuous processing difficult in fixed-bed reactors. Without good contact the formation of large amounts of residue are likely, and scale-up to industrial scale is not feasible. The objective of the research outlined in this paper is using a laboratory catalytic fluidised-bed reactor to study: (i) the potential application of MCM-41 and mixed catalysts for polymer cracking, and (ii) the activity of these catalysts and their effect on product distribution and selectivity, and to enhance the potential benefit of catalytic polymer recycling.

2. Experimental

2.1. Materials and reaction preparation

The polymer used in this study was pure high density polyethylene (unstabilised, MW ≈ 75,000, ρ = 960.3 kg m⁻³, BASF). HDPE is a structurally regular chain material with very few small branch points and hence the polymer packs efficiently resulting in a highly crystalline material with correspondingly high density. HDPE was pyrolysed over various catalysts (HZSM-5, SiO₂–Al₂O₃ (SAHA) and MCM-41 containing aluminium (MCM-41(Al))), and mixture of MCM-41/HZSM-5 and SAHA/HZSM-5 which were prepared using a ratio of 40 wt% non-zeolitic catalyst of SiO₂–Al₂O₃ and MCM-41, 40 wt% HZSM-5 and 20 wt% ludox to act as a bonding agent and a diluent. The catalysts employed are described in Table 1. Prior to use, all the catalysts were pelleted, crushed and sieved to give particle sizes ranging from 75 to 180 µm. The catalyst (0.25–0.3 g) was then activated by heating in the reactor in flowing nitrogen (50 ml min⁻¹) to 120 °C at 60 °C h⁻¹. After 2 h the temperature was increased to 520 °C at a rate of 120 °C h⁻¹. After 5 h at 520 °C the reactor was cooled to the desired reaction temperature.

High purity nitrogen was used as the fluidising gas and the flow was controlled by a needle valve. The feed system was designed to allow HDPE particles, purged under nitrogen, to enter the top of the reactor and to drop freely into the fluidised bed. The particle sizes of both catalyst (75–180 µm) and polymer (75–250 µm) were chosen as being large enough to avoid entrainment. The catalyst is small enough to be adequately fluidised. The polymer feed system was designed to allow HDPE particles, purged under nitrogen, to enter the top of the reactor and to drop freely into the fluidised bed at t = 0 min. On addition of the polymer, the fluidised bed remains fluidised. The added polymer melts, wets the catalyst surface and is pulled into the catalyst macro pores by capillary action [14]. At sufficiently low poly mer/catalyst ratios the outside of the catalyst particles are not wet with polymer, so the catalyst particles move freely.

Table 1

<table>
<thead>
<tr>
<th>Catalyst</th>
<th>Micropore size (nm)</th>
<th>BET area (cm²/g)</th>
<th>Si/Al ratio</th>
<th>Commercial name</th>
</tr>
</thead>
<tbody>
<tr>
<td>MCM-41(Al)</td>
<td>4.2–5.2b</td>
<td>845</td>
<td>17.5</td>
<td>—c</td>
</tr>
<tr>
<td>SAHA</td>
<td>3.15b</td>
<td>274</td>
<td>2.6</td>
<td>Synlyst 25d (Silica–alumina)</td>
</tr>
<tr>
<td>HZSM-5</td>
<td>0.55 × 0.51</td>
<td>391</td>
<td>17</td>
<td>—d</td>
</tr>
</tbody>
</table>

a Mixture of 40 wt% MCM-41{Al} (or SiO₂–Al₂O₃ (SAHA)) with 40 wt% HZSM-5 and 20 wt% ludox.

b Single point BET determination.
c Synthesised by procedure outlined by Beck [19].
d Crosfield Chemicals, Warrington, UK.
e BP Chemicals, Sunbury-on-Thames, UK.

2.2. Experimental procedures and product analysis

A detailed description of the experimental system is given schematically in Fig. 1. The reactor consists of a 400 mm long pyrex glass tube with a sintered distributor (10 mm i.d.) in the middle section. The tube had an inverted bell shape and was divided into three parts—an upper section (170 mm × 20 mm i.d.), a middle section (30 mm × 10 mm i.d.) and a lower section (200 mm × 10 mm i.d.). A three-zone heating furnace with digital controllers was used and the temperatures of the furnace in its upper, middle and bottom zones were measured using three thermocouples. High purity nitrogen was used as the fluidising gas and the flow was controlled by a needle valve. Volatile products
leaving the reactor were passed through a glass-fibre filter to capture catalyst fines, followed by an ice-water condenser to collect any condensable liquid product. A three-way valve was used after the condenser to route product either into a sample gas bag or to an automated sample valve system with 16 loops. Tedlar bags, 15 l capacity, were used to collect time-averaged gaseous samples. The bags were replaced at intervals of 10 min throughout the course of reaction. The multiport sampling valve allowed frequent and very rapid sampling of the product stream at 0.5 and 1 min intervals.

Gaseous products were analysed using a gas chromatograph (VARIAN 3400) equipped with: (i) a thermal conductivity detector (TCD) fitted with a 1.5 m × 0.2 mm i.d. Molecular Sieve 13X packed column, and (ii) a flame ionisation detector (FID) fitted with a 50 m × 0.32 mm i.d. PLOT Al2O3/KCl capillary column. The solid remaining deposited on the catalyst after the catalytic degradation of the polymer were deemed “residues” and contained involatile products and coke. The amount and nature of the residues were determined by thermogravimetric analysis (TA Instruments, SDT 2960 Simultaneous DTA-TGA). Data analysis was carried out using a Microsoft Excel spreadsheet. The reactor and various units of the collection system were weighted before and after the runs to determine the mass balance.

3. Results and discussion

Catalytic pyrolysis products (P) are grouped together as hydrocarbon gases (<C3), gasoline up to C9 (C5–C9), liquids (condensate in condenser and filter) and residues (coke and products, involatile at reaction temperature, deposited on catalyst) to enable the overall pyrolysis processes to be described more easily. The term “yield” as used in this paper is defined by the relationship

$$\text{Yield (wt\%)} = \left(\frac{P(\text{g})}{100}\right) = \frac{\text{polymer Fed (g)}}{\text{polymer Fed (g)}}$$

3.1. Degradation of HDPE over various catalysts

A number of runs were repeated in order to check their reproducibility. Due to the high nitrogen flow rates used in this study, it is difficult to completely recover all the lower molecular weight material, and this results in some loss in the mass balance. The reaction yield (based on the feed) of HDPE degradation for each catalyst is summarised in Table 2. The bulk of the products observed were in the gas phase (> 85 wt%) with less than 4 wt% liquid collected. The yield of volatile hydrocarbons for zeolitic cracking catalysts (HZSM-5 > MCM-41/HZSM-5 > SAHA/HZSM-5) was higher and the highest yield (nearly 94 wt%) was obtained for HZSM-5. The highest level of unconverted polymer was observed with non-zeolitic catalysts (SAHA > MCM-41), whilst the highest BTX was observed with HZSM-5. Some similarities were observed between MCM-41 and SAHA with C1–C4 and C5–C9 yields, which were approximately 26–31 wt% and 54–61 wt%, respectively. However, with MCM-41/HZSM-5 and SAHA/HZSM-5 the C1–C4 and C5–C9 yields were approximately 57–59 wt% and 29–32 wt%, respectively.

Differences shown in Fig. 2 between the catalysts can be seen clearly with SAHA/HZSM-5 generating > 55 wt% C3+C4 alkenes compared with HZSM-5 producing > 45 wt%, and with SAHA producing > 45 wt% of C4+C5 alkenes. MCM-41 produces
comparable total amounts of alkenes to SAHA but with higher overall amounts of C₆ and C₇ alkenes. Paraffin cracking is well known to proceed by carbocation mechanisms where the initially formed ions undergo chain reactions via processes such as isomerisation and β-scission (monomolecular) and hydrogen transfer and oligomerisation (bimolecular), to yield typically C₃–C₆ paraffin and olefins [14].

Table 2: Summary of the main products of HDPE degradation at reaction temperature of 360 °C over various catalysts

<table>
<thead>
<tr>
<th>Degradation results</th>
<th>Catalyst type</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>MCM-41</td>
</tr>
<tr>
<td>Yield (wt% feed)</td>
<td></td>
</tr>
<tr>
<td>Gaseous</td>
<td>87.88</td>
</tr>
<tr>
<td>Liquid</td>
<td>3.04</td>
</tr>
<tr>
<td>Residue</td>
<td>9.08</td>
</tr>
<tr>
<td>Involatile residue</td>
<td>6.91</td>
</tr>
<tr>
<td>Coke</td>
<td>2.17</td>
</tr>
<tr>
<td>Mass balance (%)</td>
<td>92.83</td>
</tr>
<tr>
<td>Distribution of gaseous products (wt% feed)</td>
<td></td>
</tr>
<tr>
<td>Hydrocarbon gases $(\sum C_1–C_4)$</td>
<td>26.43</td>
</tr>
<tr>
<td>Gasoline $(\sum C_5–C_9)$</td>
<td>61.37</td>
</tr>
<tr>
<td>BTX $^c$</td>
<td>0.08</td>
</tr>
</tbody>
</table>

Fluidising N₂ rate = 570 ml min⁻¹, catalyst particle size = 75–180 μm and polymer to catalyst ratio = 40 wt%.

$^a$ Condensate in condenser and captured in filter, unidentified.

$^b$ Coke and involatile products.

$^c$ Benzene, toluene and xylene.

catalysts shows less total gases, as well as less dry gases, with a lower amount of gasoline than HZSM-5. The results shown in Fig. 3 illustrate that more isoalkanes are produced with MCM-41/HZSM-5 and SAHA/ HZSM-5 catalysts compared with MCM-41 and SAHA used alone. In the case of MCM-41/HZSM-5 mixtures can also provide an improvement on product selectivity with much more isoalkanes in the volatile products compared to SAHA/HZSM-5.

3.2. Product stream variation with reaction temperature and catalyst type

The main product distributions for HDPE degradation over MCM-41 at temperatures in the range of 290–430 °C are illustrated in Table 3. Overall, the liquid and residue fractions decreased with increasing reaction temperature. The remaining solid deposited on the catalyst after the polymer degradation contained involatile products and coke. The amount and nature of the residues was determined by TGA [27]. First the post-reaction catalyst and residue were heated in nitrogen (to determine the amount of involatile products). The sample was then allowed to cool before switching to air and heating to burn off carbonaceous deposits (coke) on the catalyst. The yield of smaller cracked products increased with temperature as did the yields of coke and BTX. Equilibrium ratios of i-butane/n-butane and i-butene/Σbutenes were predicted using Gibbs free energy minimisation on the PRO/II flowsheets package for the temperatures used experimentally and are presented alongside the corresponding experimental results in Table 4. The i-butane/n-butane ratio reflects the involvement of tertiary C₄ carbenium ions in
bimolecular hydrogen transfer reactions and since tertiary carbenium ions are more stable than primary ions, a higher yield of isobutane would be expected [28,29]. Further evidence of the increase in secondary reactions was seen in the lowering of the \(i\)-butane/\(n\)-butane and \(\sum\text{olefins}/\sum\text{paraffins}\) ratios with temperature.

The observed \(i\)-butane/\(n\)-butane ratios are well above calculated equilibrium values and the high ratio is consistent with the cracking of branched oligomers to yield isobutane. The \(i\)-butene/\(\sum\text{butenes}\) ratio is very close to the predicted equilibrium values and thus the reactions involved in the production and interconversion of butenes are very fast over various catalysts, and their ratio is primarily equilibrium controlled. SAHA with weaker acid sites and MCM-41 with large mesopores resulted in a highly olefinic product and gave rise to the broadest carbon range of \(\text{C}_3\text{e}_{\text{C}_7}\). Differences of product distributions were observed in the alkene product within the narrow distribution of \(\text{C}_3\text{e}_{\text{C}_5}\) olefins and more isoalkanes were produced when HZSM-5 was used as a co-additive (Figs. 2 and 3). On the other hand, the selectivity could further be influenced by changes in reaction conditions; in particular, valuable olefins and \(i\)-olefins were produced for mixed catalysts such as, SAHA/HZSM-5 (\(i\)-butane/\(n\)-butane = 9.35; \(\sum\text{olefin}/\sum\text{paraffin} = 8.79\)) and MCM-41/HZSM-5 (\(i\)-butane/\(n\)-butane = 7.45; \(\sum\text{olefin}/\sum\text{paraffin} = 6.58\)), compared to HZSM-5 (\(i\)-butane/\(n\)-butane = 4.55; \(\sum\text{olefin}/\sum\text{paraffin} = 4.63\)) used in HDPE cracking (Table 4).  

### Table 3
Product distributions shown from MCM-41 catalysed pyrolysis of HDPE at different reaction temperatures

<table>
<thead>
<tr>
<th>Degradation results</th>
<th>Temperature (°C)</th>
<th>290</th>
<th>330</th>
<th>360</th>
<th>390</th>
<th>430</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield (wt% feed)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Collection time (min)</td>
<td></td>
<td>30</td>
<td>20</td>
<td>15</td>
<td>15</td>
<td>15</td>
</tr>
<tr>
<td>Gaseous</td>
<td></td>
<td>84.72</td>
<td>86.53</td>
<td>87.80</td>
<td>90.37</td>
<td>92.25</td>
</tr>
<tr>
<td>Liquid&lt;sup&gt;a&lt;/sup&gt;</td>
<td></td>
<td>4.73</td>
<td>4.16</td>
<td>3.12</td>
<td>2.74</td>
<td>2.37</td>
</tr>
<tr>
<td>Residue&lt;sup&gt;b&lt;/sup&gt;</td>
<td></td>
<td>10.55</td>
<td>9.31</td>
<td>9.08</td>
<td>6.89</td>
<td>5.38</td>
</tr>
<tr>
<td>Involatile residue</td>
<td></td>
<td>8.73</td>
<td>7.35</td>
<td>6.91</td>
<td>4.53</td>
<td>2.85</td>
</tr>
<tr>
<td>Coke</td>
<td></td>
<td>1.82</td>
<td>1.96</td>
<td>2.17</td>
<td>2.36</td>
<td>2.53</td>
</tr>
<tr>
<td>Mass balance (%)</td>
<td></td>
<td>86.62</td>
<td>89.21</td>
<td>92.83</td>
<td>91.37</td>
<td>93.48</td>
</tr>
<tr>
<td>Distribution of gaseous products (wt% feed)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hydrocarbon gases ((\sum\text{C}<em>3\text{e}</em>{\text{C}_4}))</td>
<td></td>
<td>25.48</td>
<td>26.13</td>
<td>26.43</td>
<td>27.41</td>
<td>27.54</td>
</tr>
<tr>
<td>Gasoline ((\sum\text{C}<em>3\text{e}</em>{\text{C}_0}))</td>
<td></td>
<td>59.24</td>
<td>60.37</td>
<td>61.37</td>
<td>62.58</td>
<td>63.95</td>
</tr>
<tr>
<td>BTX&lt;sup&gt;c&lt;/sup&gt;</td>
<td></td>
<td>-d</td>
<td>0.03</td>
<td>0.08</td>
<td>0.38</td>
<td>0.76</td>
</tr>
</tbody>
</table>

<sup>a</sup> Condensate in condenser and captured in filter, unidentified.

<sup>b</sup> Coke and involatile products.

<sup>c</sup> Benzene, toluene and xylene.

<sup>d</sup> Less than 0.01 wt%.

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Fig. 3. Comparison of alkane products for the catalytic degradation of HDPE at 360 °C over different catalysts (polymer to catalyst ratio = 40% wt/wt, rate of fluidisation gas = 570 ml min<sup>-1</sup>).
the polymer being degraded over 15 min. The rate of gaseous hydrocarbon evolution further highlights the slower rate of degradation over SAHA/HZSM-5 as shown in Fig. 5 when comparing all catalysts under identical conditions at 360 °C. It seems that mixed catalysts (SAHA/HZSM-5 and MCM-41/HZSM-5) are less likely to form a polymer/catalyst complex and consequently to decrease the scission reaction further to generate the volatile products [14]. The maximum rate of generation was observed after 1 min with HZSM-5 (56 wt% min⁻¹/C₂₅₅), whereas the maximum was observed after 2 min with MCM-41 (34 wt% min⁻¹/C₂₅₅) and SAHA (28 wt% min⁻¹/C₂₅₅). This can be explained in terms of the nature of the acid sites with SAHA being made up predominantly of weaker sites compared to the predominantly more acidic sites found in zeolite catalysts. In the case of the MCM-41 containing aluminium with activity coupled with large pores, it may lead the heavier hydrocarbons (such as, partially depolymerised HDPE) to diffuse to the majority of the active sites, although the differences in the nature of the sites may also be relevant [30]. Next paper will discuss mechanistic aspects, in relation to the structure of catalysts and their acid density, in more detail.

The deactivation of the catalyst was examined by the transient change in the amount of gaseous compounds produced. Deactivation of MCM-41 and SAHA catalysts was observed (Fig. 6) when the spot samples, taken during the course of the reaction, were analysed. The deactivation is reflected in the decrease of the amount of isobutane (i-C₄) and isopentane (i-C₅) produced (product of bimolecular reaction) and the relative increase in olefins (product of monomolecular reaction), exemplified by, sum of butenes (ΣC₄) and pentenes (ΣC₅). The larger pore opening of MCM-41 and amorphous SAHA materials allows bulky reactions to occur, ultimately leading to the generation of coke and subsequently deactivation of the catalyst. In contrast, the pore structure of HZSM-5 restricts the formation of bulky intermediates and, consequently, the catalyst is resistant to coke formation explaining the product streams remain virtually unchanged throughout the degradation of HDPE. In addition, HZSM-5 is resistant to coking when coke builds up on outersurface and the

<table>
<thead>
<tr>
<th>Ratio</th>
<th>Catalyst type a</th>
<th>MCM-41 290 °C</th>
<th>MCM-41 360 °C</th>
<th>MCM-41 430 °C</th>
<th>SAHA 360 °C b</th>
<th>ZSM-5 360 °C</th>
<th>SAHA/ZSM-5 360 °C</th>
<th>MCM-41/ZSM-5 360 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>i-butane/n-butane</td>
<td>10.75</td>
<td>7.53</td>
<td>4.35</td>
<td>12.57</td>
<td>4.55</td>
<td>9.35</td>
<td>6.45</td>
<td></td>
</tr>
<tr>
<td>i-butane/Σbutenes</td>
<td>1.17</td>
<td>0.95</td>
<td>0.81</td>
<td>0.58</td>
<td>0.49</td>
<td>0.52</td>
<td>0.50</td>
<td></td>
</tr>
<tr>
<td>i-butene/Σbutenes</td>
<td>0.59</td>
<td>0.53</td>
<td>0.47</td>
<td>0.56</td>
<td>0.52</td>
<td>0.48</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Σolefin/Σparaffins</td>
<td>9.24</td>
<td>8.57</td>
<td>6.84</td>
<td>10.73</td>
<td>4.63</td>
<td>8.79</td>
<td>5.84</td>
<td></td>
</tr>
</tbody>
</table>

a Represents a series of runs where polymer to catalyst ratio = 40 wt% and fluidising N₂ = 570 ml min⁻¹.
b Represents a series of runs with various catalysts where reaction temperature = 360 °C.
c Predicted equilibrium data.
d Σolefins denotes the sum of all olefinic products; Σparaffins denotes the sum of all paraffinic products.
product stream remains essentially unchanged, whereas the mixed catalysts (SAHA/HZSM-5 and MCM-41/ HZSM-5) along with the increased tolerance to “coke” in mixture pore structures provide the most likely reasons for the lack of variation in the product stream over these catalysts.

4. Conclusions

The systematic experiments discussed in this paper show that the use of various catalysts improve the yield of volatile products and provide better selectivity in the product distributions. The catalytic degradation of polyethylene performed in fluidised-bed reactor was demonstrated to be a useful method for the production of potentially valuable hydrocarbons.

Both catalyst acidity and diffusion constraints within individual pores of each catalyst may play significant roles in the observed product distribution from HDPE cracking. HZSM-5 catalysed degradation resulted in much more amounts of volatile hydrocarbons compared with degradation over non-zeolitic catalysts (MCM-41 and SAHA). Both MCM-41 and SAHA yielded a saturate-rich stream with a wide carbon number distribution and substantial coke levels. Some valuable hydrocarbons of olefins and i-olefins were produced for mixed catalysts when HZSM-5 was used as a co-additive. The larger pore structure of the MCM-41 and SAHA catalysts showed deactivation in contrast to the more restrictive HZSM-5.

Chemical recycling, i.e., conversion of waste polymers into feedstock or fuels, has been recognized as an ideal approach and could significantly reduce the net cost of disposal. It is concluded that under appropriate reaction conditions and suitable catalysts can have the ability to control both the product yield and product distribution from polymer degradation, potentially leading to a cheaper process with more valuable products.

References

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