ABSTRACT: Both lignin and waste plastic are refractory polymers whose oxidation can produce feedstocks for the manufacture of chemicals and fuels. This brief review explores how renewably generated electricity could provide energy needed to selectively activate the endothermic depolymerization reactions, which might assist the production of hydrogen. We identify mediated electrochemistry as a particularly suitable approach to contending with these refractory, sparingly soluble materials.

INTRODUCTION

Both lignin, the underutilized byproduct from the manufacture of paper and cellulosic ethanol,1 and man-made plastics, whose deliberate disposal and inadvertent release have created a growing environmental burden,2 are plentiful, refractory, organic polymers. Their potential economic value lies, at first glance, more in chemical products derived from their component monomers than in their value as fuels (Table 1). The listed economic values represent unrealizable upper limits: they do not account for real yields, processing costs, or market elasticities. Even so, they can be considered as gross comparators of the lines along which the process chemistry could be directed.

Both lignin and waste plastic are expensive to transport and thus are accumulated at geographically distributed sites, in quantities that are small on the scale of fuel production.10 Their conversion must, therefore, accommodate decentralized operation, that is, not rely heavily on an infrastructure of feedstocks (e.g., hydrogen) or utilities for sourcing and sinking heat (except, perhaps, electricity or natural gas). Preferably, the processes should result in liquid or solid products that are easily transported.

Candidate conversion processes include thermolysis,11 biological degradation,12 and even high energy radiolysis.13 Those processes have been directed primarily at producing fuels, where they lead to broad mixtures of gaseous, liquid, and solid products.

Here, we consider electrolytic depolymerization. Both electroreductive14 and electro-oxidative15 depolymerizations are possible. We emphasize electro-oxidation because, whereas both approaches can provide a chemical co-product or the elimination of waste, oxidation can also, in principle, provide a route to lower costs by decreasing the input energy (cell voltage) required for a concurrent, endergonic process such as hydrogenation. Converting the anolyte to carbon dioxide and water (“mineralization”) should provide the highest energy savings; however, mineralization of materials as refractory as lignin and plastics typically requires generating a potent oxidant, whose energetics may be decoupled from and, therefore, not offset by the exergonic mineralization. Instead of merely mineralizing the anolyte, producing a valuable product at both the anode and the cathode might offer a larger economic benefit.

Because neither lignin nor every waste plastic, such as polypropylene and polystyrene, is soluble in typical electrolytes, we commence with a brief review of the electolysis of suspensions of solids and then discuss the electrochemistry of
Table 1. Energy and Potential Economic Value of the Feedstocks and Products Potentially Derivable from Them
d
| resource          | availability in the U.S./Mt year⁻¹ | energy content/EJ year⁻¹ | value/G$ year⁻¹ if all the feed were converted to the indicated product |
|-------------------|------------------------------------|--------------------------|------------------------------------------------------------------------|
| waste plastic     | 35.4                               | 1.6                      | distillate fuel 10                                                      |
| lignin            | 13                                 | 0.31                     | BTX 23                                                                |
| petrofuel         | 658                                | 30                       | acetaldehyde 64                                                        |
| csi               | 13                                 | 25.3                     | distillate fuel 2                                                      |
| distillate fuel   | 10                                 | 0.21                     | phenol 13                                                             |
| coal              | 116                                | 12.2                     | vanillin 91                                                           |
| transportation fuel| 370                                | 11.1                     |                                                                  |
| BTX               | 23                                 | 0.7                      |                                                                  |

“The data are presented for the U.S. because the information is readily available. Roughly, they can be extrapolated to global figures by multiplying by 5. "Amounts from Geyer et al. Energy content estimated at 44 MJ/kg; product prices from IHS. Amount from FAO. Energy content estimated at 23.5 MJ/kg; product prices from IHS. Vanillin at 35% yield equal to the concentration of coniferol in nonwood lignin and a market price of 20 $/kg. "Amount from EIA. Energy content estimated at 45 MJ/kg, at a market price of $40/bbl with a netback of 1.5, which was estimated from examples in Kaiser et al."

Table 2. Comparison of Conditions Used for Electrocoagulation and Electrochemical Conversion

| substrate | pH range used for flocculation | pH range used for anodic oxidation |
|-----------|--------------------------------|-----------------------------------|
| plastics  | 7.5₁⁵                            | 7.5−14₂¹₂³                        |
| lignin    | 7.6₁⁵                            | <0                                |

Table 3. Nominal Compositions and Thermodynamic Properties of C-Based Substrates

| substrate | C/wt % | H/wt % | O/wt % | ΔH_cal/molC⁻¹ | S°/J molC⁻¹ K⁻¹ |
|-----------|--------|--------|--------|----------------|-----------------|
| lignin    | 63.5   | 5.71   | 30.8   | −453           | 20              |
| waste plastic | 46.6 | 7.6   | 36.9   | −502           | 20              |
| coal      | 60     | 6      | 34     | −490           | 20              |

Electrochemistry of suspensions has been studied for many years as a way to improve the performance of an electrode and as a way to process solids, for example, coal slurries. Experiments with carbon black show that electrochemical currents can arise from:

1. Physical processes, e.g., charging of the double layers adjacent to the high surface areas of the suspended particles; transfer of metal ions from the particles to the electrode
2. Chemical processes, e.g., oxidation of the suspended solids

Chemical conversion can occur interfacially in the strong electric field present in the double layer of the electrode. In that case, the rate of the reaction depends strongly on the adhesion of the particles to the working electrode rather than to the frequency of collisions. Alternately, the solid could react with a homogeneous electron transfer mediator, a species that is oxidized/reduced at the electrode and then reduced/oxidized by interacting with the solid. Oxidation mediators include metal centers, such as oxidized polyoxometalates, or simple ions, e.g., Fe⁺ⁿ produced at the anode, hyponitrite, radicals (e.g., TEMPO), or other potent oxidants such as hydroxyl radicals, peroxyacetic acid, or peroxydisulfates produced at doped diamond electrodes.

In principle, electrocoagulation of suspensions of lignin and finely divided plastics could compete with the electrochemical conversion of the materials because the lower exposure (surface/volume ratio) of flocs would make the substrates less accessible. However, the coagulation typically operates near neutral pH, whereas the conversions proceed under very acidic or very basic conditions (Table 2).

Electrochemistry of suspensions and emulsions of solid substrates

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Total electro-oxidation of lignin and plastic waste

To exemplify the mineralization of those materials, we assume nominal compositions for lignin, waste plastic, and, as an extension, coal (Table 3). We estimated the heats of combustion corresponding to those compositions using a modified Dulong relation:

\[ \text{HHV/kj kg}^{-1} = 349.1^\circ\text{C} + 1178.3^\circ\text{H} - 103.4^\circ\text{O} \]

where C, H, and O are the wt % of each element in the material. We then used Hess’s Law to calculate a heat of formation of each substrate. The entropies of each substrate per mol of carbon were all set at 20 J mol⁻¹ K⁻¹, which is a value representative of several man-made polymers (plastics) and lignin. The entropy of polymerization per carbon for polyolefins are about 4 times higher and depend, of course, on the ordering and crystallinity of the polymer.

The composition of waste plastic is highly variable and includes heteroatoms, notably nitrogen (polyamides) and chlorine (polyvinyl chloride), which are not included in the table above. Each type merits its own discussion, but those complications need to be deferred to a review longer than this one, which seeks to compare the processing of lignin and waste plastic rather than focus on either. Total oxidations of lignin and man-made polymers are exergonic reactions (\( \Delta G < 0 \)). Therefore, their oxidation at the anode of an electrolysis cell could be used to decrease the overall cell potential for an uphill reaction occurring at the cathode, say electrolysis of water to make hydrogen. IUPAC now deprecates the use of the term “depolarization” for such a
decrease in cell voltage. We have coined the term “anode boosting” to emphasize the benefit of coupling a thermodynamically spontaneous oxidation with an endergonic reduction.26

The estimates in Table 4 suggest that the oxidations could generate sufficient free energy to decrease the thermodynamic cell potential for generating hydrogen to about 0 V, i.e., an anode boosting of more than 1 V (Table 4). However, the thermodynamic potentials bear only weakly on the over-potentials needed to achieve practicable rates, so the anode boosting is found to be much less than indicated in the table.

When the total oxidations involve mediators, say ozone, hydrogen peroxide, hydroxyl radical, or an oxidized transition metal ion, then the anode potential will correspond not to the free energy of oxidation of the feedstock but rather to the energy required to generate (oxidize) the mediator (Table 5). Thus, if the mediator were the OH radical then, instead of having to add practically zero energy to oxidize lignin, as shown in Table 4, the electrolyzer would require adding 460 kJ/mol of generated H2 (or per mol of oxidized C).

Even if the anode reaction does not decrease the cell potential of a coupled reduction (e.g., protons to make H2, hydrogenation, or hydrodeoxygenation of an organic intermediate), it might serve to purify an organic-laden process stream.

**SELECTIVE ELECTRO-OXIDATION OF LIGNIN AND POLYMERS**

**Lignin.** Selective oxidation of the refractory polymers could also produce a product whose sale could improve the overall economics of the process.6,30 In the case of lignin, electrochemical oxidation primarily attacks the −O− ether linkages, which are both abundant and thermally and oxidatively labile compared to the C−C linkages also present in lignin.

The products of breaking the −O− bonds are oxygenated aromatics (Figure 1), with an industrial emphasis on vanillin because of its ready market. Mass yields are typically <2 wt %, and there is no one catalyst or set of process conditions that confer extraordinary activity or selectivity. The simultaneous separation of vanillin to prevent its over-oxidation looks particularly promising.23

**Figure 1.** Illustrative electrochemical oxidation of a notional tetralignol to products in mass yields described by Stecker et al.31

Judicious use of mediators, which provide a way to enable excellent contacting of lignin polymers with electrochemically activated species would seem to be an approach that could offer both activity and selectivity. We note that polyoxometalates, with their large, tunable range of oxidation states, have been used as mediators for delignification and bleaching but primarily through thermally activated catalysis.32 We speculate that there could be an opportunity to employ them as electrochemical mediators.

**Plastics.** Man-made polymers, except polyesters and amides, contain few fragile linkages (Figure 2). Therefore, electrochemical oxidation of them has required very potent

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Table 4. Estimated Anodic Boosting by the C-Based Substrates at 298 K

| reaction | ΔH/kJ mol⁻¹ | ΔS/J mol⁻¹ K⁻¹ | ΔG/kJ mol⁻¹ | E_{cell}^0 /V |
|----------|-------------|----------------|-------------|-------------|
| H₂O → ¹/₂O₂ + H₂ | 285.3 | 163 | 237 | −1.23 |
| lignin + H₂O → CO₂ + H₂ | 168 | 364 | 60 | −0.14 |
| plastic + H₂O → CO₂ + H₂ | 178 | 407 | 57 | −0.12 |
| coal + H₂O → CO₂ + H₂ | 130 | 368 | 21 | −0.05 |

Table 5. Reduction Potentials of Possible Aqueous Oxidation Mediators (E^0 Referenced to SHE)27

| mediator generating reaction | ΔG/kJ mol⁻¹ | E_{red}^0 /V |
|-----------------------------|-------------|-------------|
| O₂ | O₂ + H₂O → O₃ + 2H⁺ + 2e⁻ | 401 | 2.08 |
| H₂O₂ | 2H₂O → H₂O₂ + 2H⁺ + 2e⁻ | 340 | 1.76 |
| OH⁺ | H₂O → OH⁺ + H⁺ + e⁻ | 230 | 2.38 |
| lignin peroxidase²⁸ | Fe(III) → Fe(IV)O⁺ + e⁻ | 138 | 1.43 |
| CH₃COOOH | CH₃COOH + H₂O → CH₃COOOH + 2H⁺ + 2e⁻ | 300 | 1.39 |
| H₃PMo₆V₆O₄₀ | [V₃V⁺^V⁺] → [V₃V⁺^V⁺] + e⁻ | 60 | 0.64 |
| manganese peroxidase²⁸ | Mn(II) → Mn(III) + e⁻ | 77 | 0.8 |
| laccases²⁹ | Cu(I) → Cu(II) + e⁻ | 39−77 | 0.4−0.8 |

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oxidation mediators, e.g., Ag^{2+} (E^0 = 1.98 V), Co^{3+} (E^0 = 1.9 V), and Ce^{4+} (E^0 = 1.7 V), just to effect surface treatments.\textsuperscript{33} Evidently, such strong oxidants do not offer much selectivity. Using smaller oligomers to investigate the electrochemical depolymerization of plastic cannot capture well the structuring of the electrolyte in the vicinity of the more challenging substrates, which are also highly hydrophobic (e.g., polyolefins). Therefore, we are not confident to extrapolate from the behavior of such surrogates.

Because they are so refractory, the electrochemical depolymerization of waste plastic may benefit from pretreatments that use modest amounts of energy or reagents to initiate the bond breaking. Plasma chemistry,\textsuperscript{34} sonochemistry,\textsuperscript{35} mechanochemistry,\textsuperscript{36} and, again, radiochemistry\textsuperscript{13} come to mind. The latter two approaches have been demonstrated on lignin but not yet, to our knowledge, on synthetic plastics. At issue would be the usual trade-offs from increasing capital expense for the additional reactor and operating expense for the additional energy input against the process effectiveness.

To venture again into speculation, we wonder if two known chemistries, selective oxidative dehydrogenation of alkanes combined with olefin metathesis, might be combined to depolymerize polyolefins (Figure 3).

The first reaction (Figure 3) has been demonstrated, albeit using photochemical activation and not for polymers.\textsuperscript{37} For small molecules (e.g., butane), the reaction proceeds at a noticeable rate when activated with photons whose wavelength is 322 nm (=371.5 kJ/mol, 3.85 V). That high a potential is incompatible with an aqueous solvent but could be compatible with an electrolyte based on ionic liquids whose operating potential window can be greater than 5 V\textsuperscript{38} or a polar but non-aqueous electrolyte such as sulfolane. The second reaction, ethenolysis (Figure 3), has been used to depolymerize natural rubber, polyisoprene, coincidentally in an ionic liquid as the reaction medium.\textsuperscript{39} Although that sequence of reactions is plausible, its implementation will likely require considerable research.

\section*{CONCLUSIONS}

Nonselective electrochemical oxidation of lignin or waste plastics, which also serves to remove organics from process water, could offer offset endergonic reactions at the cathode. Whereas there are examples using plastics and coal for such anode boosting, we do not have the ability to predict either the extent of the oxidation and the amount of boosting. Depolymerization, especially of lignin, to make chemically and economically interesting intermediates is also known. Not surprisingly, given the complexity of lignin, the reactions typically yield conversions of a few weight percent, distributed among several products. The use of mediated electrochemistry offers a route to the depolymerization of lignin and waste plastic for both generating energy or valuable chemicals.

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The manuscript was written through contributions of both authors.

Figure 2. Representation of an electrochemical oxidation of a polyolefin mediated by Ag^{2+}. After Brewis et al.\textsuperscript{13}

Figure 3. (a) Oxidative dehydrogenation of a polyolefin. (b) Ethenolysis of the dehydrogenated polyolefin.
Notes
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