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The use of mechanical alloying for the preparation of palladized magnesium bimetallic particles for the remediation of PCBs

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ABSTRACT

The kinetic rate of dechlorination of a polychlorinated biphenyl (PCB-151) by mechanically alloyed Mg/Pd was studied for optimization of the bimetallic system. Bimetal production was first carried out in a small-scale environment using a SPEX 8000M high-energy ball mill with 4- μ m-magnesium and palladium impregnated on graphite, with optimized parameters including milling time and Pd-loading. A 5.57-g sample of bimetal containing 0.1257% Pd and ball milled for 3 min resulted in a degradation rate of 0.00176 min⁻¹ g⁻¹ catalyst as the most reactive bimetal. The process was then scaled-up, using a Red Devil 5400 Twin-Arm Paint Shaker, fitted with custom plates to hold milling canisters. Optimization parameters tested included milling time, number of ball bearings used, Pd-loading, and total bimetal mass milled. An 85-g sample of bimetal containing 0.1059% Pd and ball-milled for 23 min with 16 ball bearings yielded the most reactive bimetal with a degradation rate of 0.00122 min⁻¹ g⁻¹ catalyst. Further testing showed adsorption did not hinder extraction efficiency and that dechlorination products were only seen when using the bimetallic system, as opposed to any of its single components. The bimetallic system was also tested for its ability to degrade a second PCB congener, PCB-45, and a PCB mixture (Arochlor 1254); both contaminants were seen to degrade successfully.

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1. Introduction

Polychlorinated biphenyls (PCBs), a class of non-polar chlorinated aromatic molecules, were largely manufactured during the 1960s and early 1970s for use in a wide variety of commercial applications which took advantage of their chemical and physical properties such as low water solubility, low vapor pressure, and high thermodynamic stability [1]. Since their ban in the 1976 Toxic Substances Control Act (TSCA), due to evidence of toxicity in humans and animals, PCBs have remained an environmental threat due to these very properties which were exploited for industrial products including paint stabilizers, transformer oils, capacitors, printing inks, flame retardants, and pesticides [1,2].

Until recently, the only economical option for treatment of PCB contamination was incineration, though this method can cause the formation of highly toxic polychlorinated dibenzodioxins (PCDDs)

and polychlorinated dibenzofurans (PCDFs) which may be more detrimental to the environment than the initial PCB contamination itself. Furthermore, high cancer rates have been correlated with exposure to PCBs, dioxins, and proximity to chlorine-contaminated waste incinerators [3,4]. Other remediation techniques currently under investigation include dredging and landfilling, which do not treat the contaminant, and microbial degradation which suffers from low rate constants and incomplete degradation limiting its use at this time [5].

A plethora of recent literature studies have reported on the use of various chemical treatments shown to cause rapid, complete reductive dechlorination of PCBs using systems such as metallic calcium with a Rh/C catalyst [6], palladium coated iron (Fe/Pd) [7–12], magnesium (Mg/Pd) in aqueous media [13–16], or simply Pd/C [17,18]. The use of the mentioned bimetallic systems relies on the reduction potentials of the zero-valent metal coupled with the hydrodehalogenation catalytic activity of palladium.

This research focuses on the Mg/Pd bimetallic system due to its several advantages over the Fe/Pd system including the ability of the Mg/Pd to dechlorinate in the presence of oxygen which causes the complete corrosion of micro- and nano-scale iron. This is possible for magnesium because it forms thin, oxygen-impermeable

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but slightly water-soluble oxide layers [19]. Additionally, the magnesium or iron acts as a reductant (electron donor) during the dechlorination process of PCBs, and gives rise to another advantage of using Mg due to its greater thermodynamic driving force as demonstrated by a comparing their reduction potentials [13].

Standard reduction potentials for Mg and Fe (Reactions (1A) and (1B)):

$$Mg^{2+} + 2e^- \rightarrow Mg^0, \quad E^0 = -2.20 V \text{ vs.SHE}$$
 (1A)

$$Fe^{2+} + 2e^- \rightarrow Fe^0, \quad E^0 = 0.44 \text{ V vs.SHE}$$
 (1B)

The palladium acts as a hydrodehalogenation catalyst by dissociating hydrogen gas (in this case, formed from the reaction of Mg⁰ or Fe⁰ with a proton source) adsorbed onto the palladium surface to produce atomic hydrogen, which can then react via radical mechanisms with chlorine atoms attached to an organic molecule (in this case, the PCB). The following reaction scheme depicts the generally accepted dechlorination reaction for Mg/Pd.

Production of atomic hydrogen on Pd and replacement of Cl atom (Reactions (2A)–(2C)):

 $Mg^0 + 2H^+ \rightarrow Mg^{2+} + H_2 \tag{2A}$

 $H_2 \rightarrow 2H^{\bullet} (duetoPdcatalyst)$ (2B)

 $2RCl + 2H^{\bullet}$ (dissociatedoncatalystsurface) $\rightarrow 2RH + 2Cl^{-}$ (2C)

Thusly, the overall reaction for the complete dechlorination of a polychlorinated biphenyl is as follows.

Overall dechlorination reaction of PCBs to biphenyl (Reaction (3)):

$$C_{12}H_{(10-x)}Cl_{x(aq)} + xMg^{0}{}_{(s)} + xH^{+} \rightarrow C_{12}H_{10(aq)} + xMg^{2+}{}_{(s)} + xCl^{-}{}_{(aq)}$$
(3)

While the observed products are biphenyl and chloride ions as depicted, the overall mechanism for the reaction has not been completely elucidated. Agarwal et al. [20] reported on the reactivity of PCBs based on the pattern of chlorine substitution, showing a preferential trend of $p \rightarrow m \rightarrow o$ -chlorine elimination with Mg/Pd systems in EtOH/H₂O; it was also shown that this trend can be complicated by both inductive and steric effects in the molecules. Previously completed work on the system studied here has shown a similar step-wise dechlorination process with a proposed three possible hydrogen species as the intermediate reactant responsible for the abstraction of chlorine in the process [21] as well as a demonstration that the choice of solvent system has dramatic effects of the dechlorination pattern and possible degradation of the biphenyl ring itself [22].

Previous studies incorporating the use of Mg/Pd bimetallic systems focused on either an in situ preparation method using zero-valent Mg and K₂PdCl₆ which allows for the formation of the bimetallic particle during the dechlorination process [13-15], or the preparation of Mg/Pd in solution which must be filtered and dried before further use [16,20]. In this work, a method for producing mechanically alloyed Mg/Pd was developed, where the preparative steps are simplistic and time-efficient. This system was optimized for numerous variables including palladium loading, ball-to-mass ratios for milling, total loading quantity, and milling time. Trials were first carried out using a small-scale approach with a SPEX 8000M Mixer/Mill varying only palladium loading and milling time (manufacturer recommended loading quantities and ball-to-mass ratios were used). Upon optimization of the bimetal material, a scaled-up study was completed using a Red Devil 5400 Twin-Arm Paint Shaker where all of the aforementioned variables were optimized for field application of the bimetal which would require large amounts to be produced at one time. Our previous studies have discussed the mechanism of dechlorination of PCBs with this system, activity loss of the bimetal over time [21], and solvent effects on the system [22], but an optimization process had never been completed for the bimetal preparation process.

2. Experimental

2.1. Chemicals

Neat PCB single-congener standards were obtained from Accustandard, Inc. Optima©grade methanol and toluene were obtained from Fisher Scientific. Magnesium (~4 μ m) was obtained from Heart Metals, Inc. Palladium on graphite (1 wt.%) was obtained from Engelhard. Palladium on graphite (10 wt.%) and sodium sulfate were obtained from Aldrich Chemicals. All chemicals were used as received.

2.2. Preparation of Mg/Pd bimetal on SPEX 8000M Mixer/Mill

Mg/Pd bimetals were prepared by first placing 5.50 g of magnesium powder and varying quantities of 10 wt.% palladium on graphite, 0.010, 0.070, 0.100 g, in a 55.4-mL tungsten carbide canister purchased from SPEX CertiPrep[®] Group. Two 10.00-g stainless steel balls, also purchased from SPEX CertiPrep[®] were added to the canister, and the canister was flushed with argon gas prior to ball milling. Milling times were 1, 3, and 7 min.

2.3. Preparation of Mg/Pd bimetal on scaled-up mill

Mg/Pd bimetals were prepared similarly to that prepared in Section 2.2 with the following changes: galvanized steel pipes with an internal diameter of 5.03 cm and length of 17.80 cm, fitted with end caps were used as milling vessels with 1.6cm³ stainless steel ball bearings, each weighing 16.32 g. A Red Devil 5400 Twin-Arm Paint Shaker, fitted with custom plates to hold the milling canisters, was used as the mill as seen in Fig. 1.

Optimization of the scaled-up milling procedure was carried out by varying each of the following parameters separately: the number of milling balls used, the amount of palladium (relative to magnesium) used, the total loading quantity (mass of bimetal) in the mill, and the length of time of a milling cycle. Each parameter was isolated and varied while leaving all other parameters constant at the midpoint of their variable range. For instance, milling time was the first parameter to be optimized by filling the canister 50 vol.% with ball bearings and 15 vol.% with the magnesium/palladium mixture. Various milling times were used and each resulting material was tested for its effectiveness in degrading PCB 151. Using the above method, it was determined that the optimum milling time was 30 min. Remaining variables were tested in a similar fashion while keeping the 30-min milling time. Values for each parameter tested in the optimization study are outlined in Tables 1–3.

Table I	
Palladium loading optimization parameters	

Table 1

Mass of 1% Pd/C (g)	% Pd in bimetal
1.00	0.0118
5.00	0.0588
7.00	0.0824
9.00	0.1059
14.00	0.1647



Fig. 1. Scaled-up milling procedure using Red Devil 5400 Twin-Arm Paint Shaker.

Ball-to-mass ratio optimization parameters.

# of milling balls used	Ball-to-Mg/Pd mass ratio
8	2.0988
12	3.1482
16	4.1976
20	5.2471
24	6.2965
32	8.3953

Table 3

Table 2

Additional optimization parameters tested.

Total bimetal loading per canister (g)	Milling time (min)
15.91	3
26.54	8
31.81	15
42.43	23
58.31	30
85.00	38
127.19	45

2.4. Sample analysis/experimental setup for Mg/Pd bimetals prepared on SPEX 8000M Mixer/Mill and scaled-up mill

Initially, optimization studies were carried out by varying the amount of palladium loading (magnesium mass was held constant). Sample vials were prepared by placing 0.1250 g of the selected Mg/Pd bimetal and 5.00 mL of 10.0-ppm PCB 151 (a hexachlorinated PCB congener - 2,2',3,5,5',6-hexachlorobiphenyl) in methanol in 20-mL screw-cap vials. Note: the ratio of 25 g bimetal/L 10 ppm PCB solution was chosen based off of our previous findings [21] regarding active site blocking by degradation byproducts. Control vials were prepared in an identical fashion both without the addition of the bimetal and as 0-h samples with the addition of the bimetal (extracted 30s after the addition of the PCB 151 solution). At the required reaction time, 5.00 mL of toluene was added to each vial. The vials were shaken for 2 min, after which a 5.0-mL lure-lock SGE brand gas-tight syringe, fitted with a 0.45-µm nylon filter, was used to remove 4 mL of solution. The solution, containing 50% methanol and 50% toluene solvents with PCBs was then transferred to a 15-mL glass centrifuge tube, and 4 mL of deionized water added. The mixture was shaken for 1 min, then centrifuged for 5 min; the toluene layer, containing the PCBs, was transferred to a clean 4-mL screw-cap vial. Sample analysis was completed on a Thermo Finnigan Trace DSQ GC-MS using an RTX-5 (30 m, 0.25 mm I.D., 0.25 μm df) column. Initial oven temperature was 120 °C held for 1.00 min, then ramped up to 200 °C at a rate of 20 °C/min, then ramped up to a final temperature of $270 \,^{\circ}$ C at a rate of $10 \,^{\circ}$ C/min and held for 3 min with a column flow rate of $1.20 \,$ mL/min and sample volume of $1.0 \,\mu$ L. The MS was run on full-scan mode with the source temperature set to $200 \,^{\circ}$ C. Experimental conditions for the remaining optimization parameters followed the above procedure.

2.5. Experimental setup for extraction efficiency/adsorption study

Separate sample vials were prepared by placing 0.1250 g of the optimized Mg/Pd bimetal, 0.1118 g Mg, or 0.0132 g 1% Pd on graphite. These masses were chosen as they were representative of the respective quantities found in 0.1250 g of the optimized Mg/Pd. PCB addition, extraction, and analysis were completed in an identical manner as described above.

3. Results and discussion

Complications involved in the quantitative analysis of PCB mixtures are well documented [2,7,9,15] and are due to the multitude of possible congeners present. Commercial PCB mixtures are known to generally contain 100 or more congeners possessing similar physical and chemical properties, leading to difficulty in separation and analysis. Differences in the degree of volatilization and solubility among different congeners have also led to significant error in quantification processes [15]. Further error encountered in PCB quantification is due to the step-wise nature of the hydrodehalogenation mechanism which leads to the appearance of congeners not present in the original sample. On a chromatogram, PCB mixtures appear as a characteristic cluster of peaks known as a PCB envelope (Fig. 2).

These characteristic patterns are utilized by EPA Method 8082, "Polychlorinated Biphenyl by Gas Chromatography," and are dependent on the analyst's aptitude for pattern recognition. The previously mentioned problems with congener volatility, solubility, and unpredictable degradation pathways make this pattern recognition approach somewhat unreliable [15]. Alternately, EPA Method 508A, "Screening for Polychlorinated Biphenyls by Perchlorination and Gas Chromatography," uses a procedure to convert all congeners to decachlorobiphenyl prior to analysis but still produces up to a 25% error in quantification [15]. In order to avoid the multitude of quantification problems listed above, a single congener (PCB-151) was chosen for our degradation studies which led us to more accurately determine degradation kinetics for our reaction system.

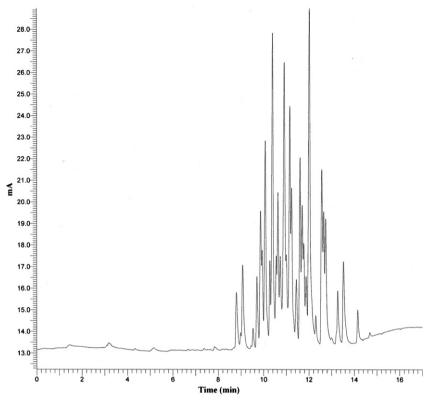


Fig. 2. Typical PCB envelope for Aroclor 1260.

3.1. Small-scale Mg/Pd optimization using the SPEX Centiprep 8000M

Sample analysis for the Mg/Pd bimetal prepared via the smallscale method described in Section 2.2, for studies carried out over time range of 0-24 h, focused on the decrease in the area count of the PCB-151 peak in the GC chromatogram to determine the kinetic rate of PCB-151 degradation. Successful dechlorination was noted by the disappearance of the PCB-151 peak and appearance of lesser-chlorinated products, supporting the documented step-wise dechlorination mechanism discussed in the literature [20-22]. It was determined that the degradation rates (all normalized to the mass of bimetal used) followed a pseudo-first order rate law with respect to the concentration of the PCB-151. The relative activity for each sample were first ordered highest to lowest for each milling time of a particular Pd loading (Table 4), then the resulting highest rates for each Pd loading were ordered to determine the optimal bimetal sample.

As seen in Fig. 3, the bimetal with the highest activity was determined to be that containing 0.1257% Pd and ball milled for 3 min. There appeared to be no distinguishable correlation between milling time and bimetal activity as shown in Table 4 where the highest kinetic rates for each Pd loading occurred at different milling times in each case. While it may be expected that the reactivity of a Mg/Pd bimetal would increase with an increase in the amount of Pd loading, however, a plateau effect is seen. An explanation for this observation is that, at a point, the graphite content of the bimetal becomes so high that the magnesium particles become thoroughly coated with graphite, limiting the magnesium surface and preventing the necessary oxidation of the particle. Work has yet to be conducted to validate this hypothesis.

Table 4
Pseudo-first order rate constants for the degradation of PCB-151 for small-scale mill
samples with time variation.

% Pd loading	Milling time (min)	Mass-normalized rate constant (s ⁻¹ g ⁻¹ catalyst)
0.0181	1	6.60E-3
	3	2.31E-3
	7	2.85E-3
0.0362	1	9.58E-3
	3	3.42E-3
	7	6.32E-3
0.0901	1	6.05E-3
	3	4.93E-3
	7	1.41E-3
0.1257	1	1.07E-2
	3	1.76E-2
	7	1.08E-2
0.1786	1	1.73E-2
	3	8.10E-3
	7	1.90E-2

3.2. Large-scale Mg/Pd optimization using the Red Devil 5400 Twin-Arm Paint Shaker

Sample analysis for the Mg/Pd bimetal prepared via the largescale method described in Section 2.3 was completed in the same fashion as that for the small-scale samples described in the previous section. As described in Section 2.3, the first variable tested was milling time, by using the median values for the other parameters as a starting point.

As seen in Fig. 4, 23 min of milling time resulted in the highest bimetal reactivity. Thus, all remaining parameter optimization studies were carried out using the 23 min mill time (Figs. 5–7).

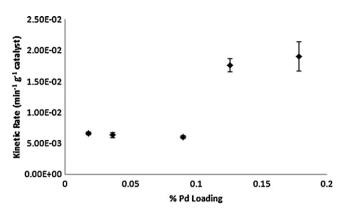


Fig. 3. Pseudo-first order rate constants for the degradation of PCB-151 vs. Φ loading on small-scale mill.

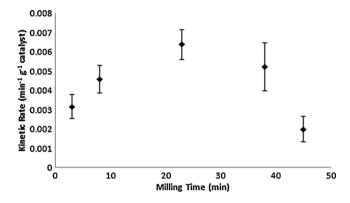


Fig. 4. Pseudo-first order rate constants for the degradation of PCB-151 vs. milling time on scaled-up mill.

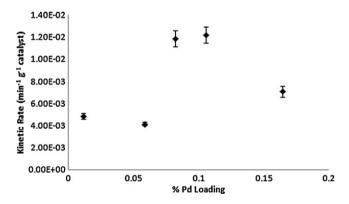


Fig. 5. Pseudo-first order rate constants for the degradation of PCB-151 vs. Φ loading on scaled-up mill.

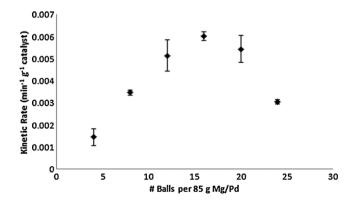


Fig. 6. Pseudo-first order rate constants for the degradation of PCB-151 vs. number of ball bearings used on scaled-up mill.

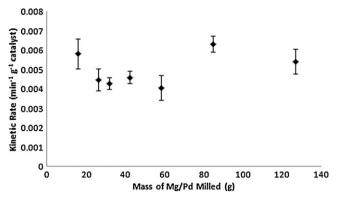


Fig. 7. Pseudo-first order rate constants for the degradation of PCB-151 vs. mass of Mg/Pd milled on scaled-up mill.

It is seen from the above results that the Mg/Pd sample with the maximum reactivity was prepared by milling 85g of Mg/Pd (76g magnesium and 9g 1% Pd on graphite) for 23 min with 16 milling balls to give a bimetal which contained 0.1059% Pd. It is noted that there is no simple correlation between reactivity and total mass of bimetal milled, and it is unknown why this peculiar behavior was observed when there were well-distinguished results for the other tested variables. Furthermore, the percent of Pd loading results showed a similar plateau phenomenon as that observed for the small-scale material tests. Where the point at which the amount of Pd on carbon began to hinder the kinetics of the system was not seen for the small-scale mill (Fig. 3), it was encountered in the scaled up process. This is most likely due to the differences in the milling parameters such as a different ball-to-material mass ratio and different speed mills between the two sets of experiments. While the goal of this work was to determine the material with the highest kinetic rate for dechlorination of PCBs in a scaled-up process, based on the results of the study, it would be feasible to increase the mass of milled bimetal to 130 g and decrease the percent Pd loading to 0.0823% without significant loss of reactivity for further increased economic benefit. For even larger scale application of this bimetal, these parameters may act as a starting point to bimetal optimization from an economical perspective.

3.3. Extraction efficiency of large-scale optimized Mg/Pd bimetal

Because the analytical technique used in this study is dependent on measuring the concentration of PCB-151 in the reaction solutions and because the bimetals contain activated carbon, a common sorbing agent for many organic molecules, the efficiency of the extraction procedure had to be verified. This was achieved by comparing the measured concentration of PCB-151 at different time intervals in separate samples allowed to come into contact with the bimetal and each of its individual components as outlined in Section 2.5. Fig. 8 gives the chromatographic results of this study, and shows that the extraction procedure is highly efficient with no loss of PCB concentration in the extracted solutions for any of the samples. These results also show that degradation products from the step-wise dechlorination of PCB-151 (penta-, tetra-, tri-chlorinated PCBs) are only seen for the ball-milled bimetal (Fig. 8E) material, which proves that the results reported in this paper are not due to adsorption of PCBs by the activated carbon but are, in fact, due to true degradation of PCBs.

3.4. SEM analysis of large-scale optimized Mg/Pd bimetal

SEM analysis of the final optimized bimetal sample was carried out by the NASA KSC Applied Chemistry Laboratory at Kennedy Space Center, FL using a JOEL JSM-7500F Field Emission Scan-

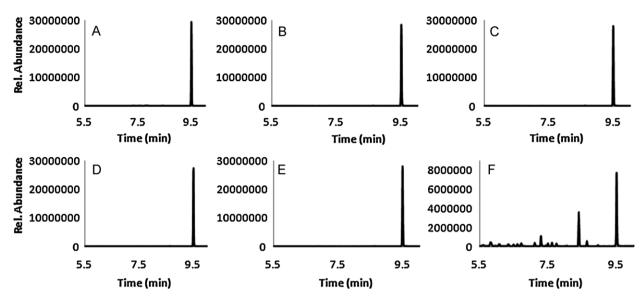


Fig. 8. Extraction efficiency chromatograms for 0-h (A) Mg, (B) 1% Pd on graphite, and (C) Mg/Pd bimetal samples and for 24-h (D) Mg, (E) 1% Pd on graphite, and (F) Mg/Pd bimetal samples.

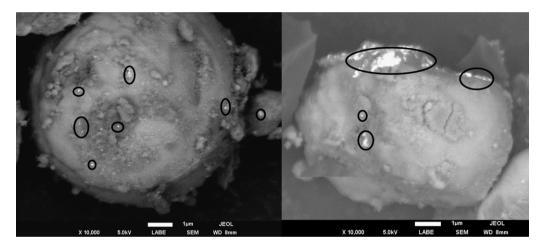


Fig. 9. SEM images of Mg/Pd bimetal prepared on scaled-up mill; circled areas are for easy identification of Pd on the particle, as supported by EDX analysis of the sample.

ning Electron Microscope. The micrographs seen in Fig. 9 show that the mechanical alloying process successfully embedded palladium/graphite fragments (small white patches, identified by circles) in the magnesium surface (grey). EDX analysis of the highest %Pd sample was able to distinguish these highly reflective, white areas as Pd and the rest of the particle (grey) as Mg. It was not able to distinguish the palladium in the lower %Pd loading samples, due to the small percentage of palladium present. However, it was able to map the carbon from the graphite and the magnesium; by seeing the graphite present throughout the ball-milled particles, it is deduced that the palladium (impregnated on the graphite) is also present. Also, since it was added as a raw material in the exact same manner as the highest %Pd, there is no doubt that it is present.

3.5. Scaled-up process success test using PCB-45

To determine if the scaling-up process was successful in producing equivalently reactive bimetal, the degradation rates for the dechlorination of PCB-45, a tetra-chlorinated biphenyl (2,2',3,6-tetrachlorobiphenyl, arbitrarily chosen as a second test PCB congener), were studied for both the optimized bimetal from the small-scale preparation method on the SPEX 8000M and

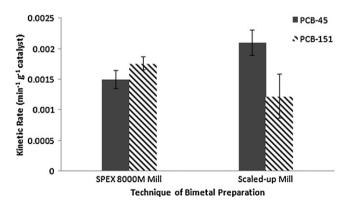


Fig. 10. PCB-45 (solid grey) and PCB-151 (black-stripe) kinetic rate comparisons for bimetals prepared on SPEX 8000M Mill and on scaled-up mill.

the optimized bimetal from the large-scale preparation method. Results are shown in Fig. 10.

The calculated rate constants for the degradation of PCB-45 $(0.00149 \text{ min}^{-1} \text{ g}^{-1} \text{ catalyst})$ and PCB-151 $(0.00176 \text{ min}^{-1} \text{ g}^{-1} \text{ catalyst})$ were similar when exposed to the material prepared on the SPEX 8000M mill. However, the rate constants for the reaction

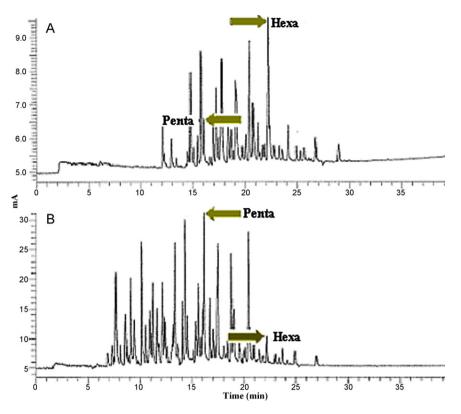


Fig. 11. GC-ECD chromatograms of 10.0-ppm Arochlor 1254 (A) control (1:100 dilution) and (B) after 24 days of exposure to bimetallic Mg/Pd (1:2 dilution).

when exposed to material prepared on the scaled-up mill gave a slightly higher rate of degradation for PCB-45 (0.00209 min⁻¹ g⁻¹ catalyst) than for PCB-151 (0.00122 min⁻¹ g⁻¹ catalyst). Based on the average rate constants for the degradation on PCB-45, there was a 30% increase in activity when the bimetal prepared on the large-scale mill was used. Conversely, there was a 30% decrease in activity when PCB-151 was exposed to the material prepared on the large-scale mill. Though this difference may seem significant, when compared with the calculated standard deviations of the studies, and due to possible sample variation (bimetal particle size, milling parameter differences between the two mills, etc.), these differences in rate constants may not be significant. If a higher number of duplicate studies were used, it is expected that these differences would normalize with lower standard deviations to become more equivalent among the studies presented.

Further testing has also confirmed successful degradation of Arochlor 1254 as seen in Fig. 11. After 24 days of exposure, the penta-chorinated product grew to become the base peak while the hexa-chorinated product peak had nearly disappeared. The Arochlor mixture also demonstrated the step-wise dechlorination process as the PCB envelope changed over time to show the formation of lower-chlorinated products from the degradation of the higher chlorinated congeners in the original Arochlor mixture.

4. Conclusions

In this work, a bimetallic system of mechanically alloyed Mg/Pd was tested for its ability to dechlorinate polychlorinated biphenyls (PCBs) and optimized for optimal reactivity. Bimetal production was first carried out in a small-scale environment using a SPEX 8000M high-energy ball mill with 4- μ m-magnesium and palladium impregnated on graphite, with optimized parameters including milling time and Pd-loading. A 5.57-g sample of bimetal containing 0.1257% Pd, ball milled for 3 min, yielded a degradation rate of 0.00176 min⁻¹ g⁻¹ catalyst as the most reactive bimetal.

For a more economical production of the bimetal, the process was scaled-up, using a Red Devil 5400 Twin-Arm Paint Shaker, fitted with custom plates to hold milling canisters. Optimization parameters tested for the scaled-up process included milling time, number of ball bearings used, Pd-loading, and total bimetal mass milled. The most reactive bimetal in the scaled-up study consisted of an 85-g sample of bimetal containing 0.1059% Pd and ball-milled for 23 min with 16 ball bearings gave the most reactive bimetal with a degradation rate of 0.00122 min⁻¹ g⁻¹ catalyst. Testing of the optimized bimetal, along with its individual components, showed that there was little to no adsorption by any component or by the bimetal itself and that dechlorination products were only seen when using the bimetallic system, as opposed to any of its single components. Lastly, it was confirmed that the bimetallic system was successful in dechlorinating alternate PCB congeners at a similar degradation rate as well as useful for treatment of Arochlor mixtures.

This system shows great promise for future application in field treatment because it has a simplistic and time-efficient preparation method capable of producing large amounts of Mg/Pd bimetal compared to previous studies [13-16,20], has an optimized low Pd loading to be more economically friendly compared to other methods [16], effectively dechlorinates PCBs on a timescale similar to more traditional methods for highly chlorinated species [20], and can be reactivated by further ball milling upon activity loss [22]. Several aspects of this developed system still require further optimization. Currently, 25 g bimetal/L is being used; preliminary studies have shown that a sample containing PCB-151 exposed to the bimetal system over time, under the current conditions, can be re-spiked with more PCB-151 and still show degradation of the newly introduced contaminant as well as the lower-chlorinated byproducts of the degradation process. We have also shown that different solvent systems have dramatic effects on the effectiveness of PCB degradation, but optimization of the solvent system has not been investigated. Future work with this system will include optimization of the amount of bimetal used to treat a specified volume

of contaminant as well as that of the solvent system in which the degradation takes place.

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