



CASE STUDY

Dechlorination of selected polychlorinated biphenyl congeners using metal-impregnated pulverized shrimp shell catalyst from waste

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ABSTRACT

BACKGROUND AND OBJECTIVES: Polychlorinated biphenyls are pervasive contaminants that are receiving attention worldwide. Due to their well-known propensity to have harmful impacts on both humans and the environment, polychlorinated biphenyls have been internationally banned for use. In this study, dechlorination of five polychlorinated biphenyl congeners, 2,2',5,5'-tetrachlorobiphenyl, 2,2',4,5,5'-pentachlorobiphenyl, 2,2',3,4,4',5'-hexachlorobiphenyl, 2,2',3,4,4',5'-hexachlorobiphenyl, 2,2',4,4',5,5'-hexachlorobiphenyl, and 2,2',3,4,4',5,5'-heptachlorobiphenyl, are evaluated. The chlorines from polychlorinated biphenyl congeners were removed using a heterogeneous catalyst synthesized via microwave-assisted impregnation of zinc metal onto pulverized shrimp shell waste.

METHODS: The five polychlorinated biphenyl congeners were dechlorinated through treatments combination of time (1–4 hours), heat (150–250 degree celsius), and catalyst proportion (1–5 percent weight/weight basis). The dechlorination trials followed the Box–Behnken experimental design and then analyzed using response surface methodology. Levels of the remaining polychlorinated biphenyl congeners were monitored by using a gas chromatograph equipped with an electron capture detector.

FINDINGS: The results of the trials demonstrated that among the five polychlorinated biphenyl congeners, only 2,2',3,4,4',5,5'-heptachlorobiphenyl did not respond to the provided treatments. Three congeners, namely, 2,2',5,5'-tetrachlorobiphenyl, 2,2',4,5,5'-pentachlorobiphenyl, and 2,2',4,4',5,5'-hexachlorobiphenyl, showed positive response, and one congener 2,2',3,4,4',5'-hexachlorobiphenyl showed negative response to the provided treatments. These findings suggested that chlorine attached to the para position of the biphenyls ring was easier to remove. The efficiency calculation of total polychlorinated biphenyl concentrations after treatments was approximately 25 percent. Such a low degree of effectiveness may be caused by the catalyst becoming inactive, either chemically through the deposition of chlorines that have been removed from the biphenyl ring or mechanically by the leaching of zinc from the surface of the pulverized shrimp shell due to insufficient mechanical strength. Optimization via response surface methodology produced optimal results for dechlorination at 150 degree celsius for 2.4 hours with 5 percent additional catalyst.

CONCLUSION: The total amount of polychlorinated biphenyls that remained after dechlorination was not significantly impacted by the treatment combination of temperature, duration, and weight of the catalyst. However, the treatments had significant effects on the chlorine removal at the para positions of the biphenyl ring. In this case 2,2',5,5'-tetrachlorobiphenyl, 2,2',4,5,5'-pentachlorobiphenyl, and 2,2',4,4',5,5'-hexachlorobiphenyl congeners have positive responses and 2,2',3,4,4',5'-hexachlorobiphenyl congener has a negative response. For polychlorinated biphenyl congeners having more than six chlorines, no chlorine removal was observed.

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INTRODUCTION

Polychlorinated biphenyls (PCBs) are a group of compounds that are classified as persistent organic substances (Melymuk *et al.*, 2022). As part of persistent organic pollutants, the PCBs might travel at a very long distance due to natural phenomena such as water flow, wind force, and climate change (Carlsson *et al.* 2017; Wang 2016; Hageman *et al.* 2015, Kalleborn *et al.* 2015; Sadler and Connell 2012). PCBs could travel transboundary (Magulova, 2012) from their initial locations and arrive at or deposited in different geographical conditions (Hanedar *et al.* 2019; Jimenez *et al.* 2015). The characteristics and features of PCBs have been used in a wide range of industrial applications (Evans *et al.*, 1991), either as closed or opened implementations (Dai *et al.*, 2016). Examples of close applications are thermal fluids in transformers and capacitors. Open applications are carbonless duplicating paper, pesticide extenders, plasticizers, adhesives, ink, insulating cable of electricals, solenoids of discharge lamps, and flame retardants (Khalid *et al.* 2021; Erickson and Kaley, 2010; ATSDR, 2000). The expected property of PCBs for their wide applications is heat resistance. PCBs are xenobiotics (i.e., man-made products), and the substances in the environment resulted from anthropogenic activities. Numerous studies have shown that PCBs are hard to naturally degrade in the environment (Elangovan *et al.*, 2019; Zhang *et al.* 2015; Alcock *et al.*, 1996), present high risks to humans through food chain mechanisms (Brazova *et al.* 2012; Roveda *et al.* 2006), and may even prevent neurodevelopment of early stages of life (Marsan and Bayse, 2020; Crinnion, 2011). As a result, the use of PCBs for any application has been banned globally. The previous usage of PCBs have created accumulation problems in the environment and demonstrated potential transport into various environmental compartments that might end up in human and biota through food chain or food web mechanism (Montano *et al.*, 2022; Umasangaji *et al.* 2020; Reddy *et al.*, 2019). The molecular structure of PCBs comprised 10 homologs with a total of 209 congeners whose chlorine content is in the range of 19 – 71 percent (%) (Ghavami and Sajadi, 2010). The PCB congeners are identified based on a code developed by Ballschmitter and Zell (1980), which is usually abbreviated as the Ballschmitter and Zell (BZ) number. Although PCBs theoretically are composed

of 209 congeners, only approximately 150 (Velazco *et al.* 2015) are found in the environmental samples and are still under consideration due to their toxic or dioxin-like characteristics (USEPA, 2010). According to the Stockholm Convention, PCBs will be totally phased out in 2028. Any remaining PCBs in signatory countries of the international convention, either those present in old stocks, still running electrical equipment, or those present in environmental matrices as pollutants must be stopped, properly managed, and then safely destroyed not later than the aforementioned year. Although the use of PCBs has been banned since the 1970s, worldwide PCB contamination problems are still present in various environmental matrices such as rivers, lakes, and harbors (Bedard, 2014); soils (Danielovic *et al.*, 2015); and buildings (Herrick *et al.*, 2007). Several methods have been utilized, either thermal, physico-chemical, or biological, to transform PCB congeners into safer molecules for humans and the environment. Thermal methods usually implemented incineration or plasma arc techniques that will produce carbon dioxide gas and water vapor as the final products (Seok *et al.*, 2005; Western *et al.*, 1995; Barton and Mordy, 1984). Thermal methods require a temperature of at least 850 degree celsius (°C) to avoid dioxin formation (Shibamoto *et al.*, 2007). The plasma arc method can completely destroy PCBs; however, its implementation requires high energy, typically at 250 kilowatts (kW), to arrive at a very high temperature, that is, 25,000 °C (Barton and Mordy, 1984). The plasma arc method is a costly operated procedure. Although biological methods are considered environmentally sound techniques and low cost, these techniques proceed at slow rate kinetics, which sometimes takes months or even years (Ang *et al.*, 2018; Wang and He, 2013). A combination of physical and chemical methods is developed to manage PCB contamination (Akhondi and Dadkhah, 2018; Velazco *et al.*, 2015). In this study, the results of five PCB congener dechlorination treated with a catalyst developed from pulverized shrimp shell waste impregnated with zinc (Zn) metal are presented. The last three congeners shown in the Table 2 could represent hydroxylated PCB precursors or metabolites that show more toxic characteristics (Park *et al.*, 2007). The five PCB congeners are typical markers in most environmental pollution (Costabeber *et al.*, 2018; Ahmadkhaniha *et al.*, 2013) and are thus chosen for this study. The present study aims

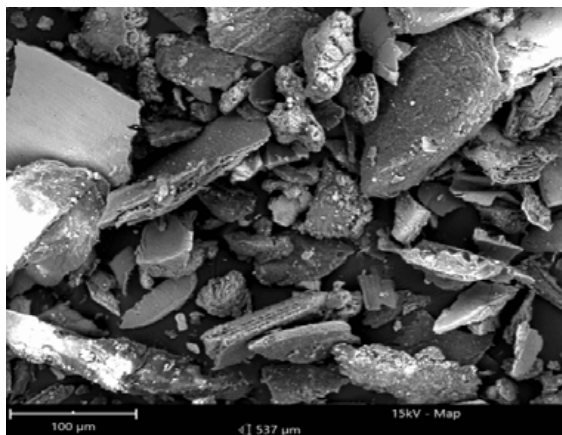


Fig. 1: Topography of pulverized shrimp shell by using SEM

Table 1: Experimental design in dechlorination PCBs using the Box–Behnken

Treatment (Factor)	Unit	Level		
		-1	0	1
Dechlorination time (A)	h	1	2.5	4
Dechlorination temperature (B)	°C	150	200	250
Catalyst weight (C)	%	1	3	5

to evaluate the performance of a heterogeneous catalyst developed from pulverized shrimp shell waste synthesized through the microwave-assisted impregnation method after heating and quenching treatments. The study was carried out in both Institut Pertanian Bogor (IPB) University and Indonesian National Research and Innovation Agency and Badan Riset dan Inovasi Nasional (BRIN) laboratory facilities from 2021 to 2022.

MATERIALS AND METHODS

Samples for PCBs treatment

Samples of transformer oil that contains PCBs were obtained from previous inventory activities carried out by the United Nations Industrial Development Organization (UNIDO) and the Ministry of Environment and Forestry Republic of Indonesia (KLHK). All the collected samples were put into dark glass bottle samples and stored in a refrigerator until use.

Catalyst activation

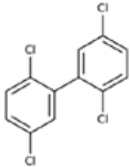
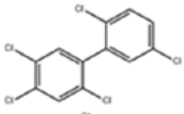
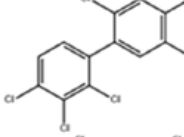
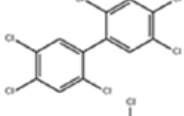
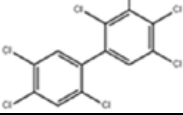
The catalyst used in this study was prepared from 90 micrometer (μm) in particle diameter of pulverized shrimp shell waste obtained from

Wirontono Baru Ltd, a company located in Balaraja Regency, Banten Province, Indonesia, that produced annual 8000 tons of processed Vannamei output harvested after 90 days nursery shrimp farming. The catalyst was prepared by heating treatment for 1 h at 550 °C followed by immediate quenching using liquid nitrogen after which impregnation of zinc metal assisted with microwave at 1800 W power rate was carried out for 20 minutes (min.). The catalyst then was activated through heating at 250°C for 2 hour (h) prior to use in PCB dechlorination trials.

Catalyst characteristics

The characteristics catalyst used in this study were summarized as the following. Topography of pulverized shrimp shell by using SEM (scanning electron microscopy) showed a highly irregular morphological arrangement (Fig. 1) with a size ranging from a few to hundred microns that could be correlated with the chitin component of the pulverized shrimp shell. The pulverized shrimp shell materials are predominantly composed of calcium, carbon, and oxygen since the major component of the shrimp shell is composed of calcium carbonate (Gbnedor *et al.*, 2016). The diffractogram peaks of

Table 2: PCB congeners' identity used in this study (Ballschmiter and Zell, 1980)

BZ number	IUPAC name	Chemical formula	Homolog	Molecular weight (g/mole)	Chlorine content (%)	Chemical structure
CB52	2,2',5,5'-tetrachlorobiphenyl	C ₁₂ H ₆ Cl ₄	Tetrachlorobiphenyls	289.9	49	
CB101	2,2',4,5,5'-pentachlorobiphenyl	C ₁₂ H ₅ Cl ₅	Pentachlorobiphenyl	323.9	54	
CB138	2,2',3,4,4',5'-hexachlorobiphenyl	C ₁₂ H ₄ Cl ₆	Hexachlorobiphenyl	357.8	59	
CB153	2,2',4,4',5,5'-hexachlorobiphenyl	C ₁₂ H ₄ Cl ₆	Hexachlorobiphenyl	357.8	59	
CB180	2,2',3,4,4',5,5'-heptachlorobiphenyl	C ₁₂ H ₃ Cl ₇	Heptachlorobiphenyl	391.8	63	

*gram per molecule (g/mole)

XRD (X-ray diffraction) at $2\theta = 29.6, 39.8, 43.5, 47.7,$ and 48.7) resemble calcite mineral that dominated calcium carbonate component of the pulverized shrimp shell (Gbnedor *et al.*, 2017; Render *et al.*, 2016). The surface area based on Brunauer–Emmet–Teller calculation of the original pulverized shrimp shell was 1.272 square meter per gram (m^2/g) and increased to arrive at $30.43 m^2/g$ after heating treatment at $550^\circ C$.

PCBs dechlorination treatment

Dechlorination, that is, removal of chlorines from parent molecules, of PCBs is carried out using the Box–Behnken experimental design (Montgomery, 2017; Myers *et al.*, 2016), comprising three independent factors, namely, dechlorination time (h), dechlorination temperature ($^\circ C$), and catalyst proportion percent based on weight to weight ratio (w/w), as shown in Table 1. The table shows that three levels for each independent factor are set. Minimum and maximum values are set based on the state-of-the-art physicochemical destruction

methods of PCBs (Akhondi and Dadkhah, 2018; Huang *et al.*, 2007; Ryoo *et al.* 2007). Dechlorination time was set in the range of 1–4 h, dechlorination temperature in the range of $150^\circ C - 250^\circ C$, and the proposition of catalyst weight in the range of 1%–5%. The intermediate or mid values for each independent factor are set automatically by the Box–Behnken experimental design.

PCB congener analysis

The determination of five PCB congeners (Table 2), namely, 2,2',5,5'-tetrachlorobiphenyl (CB52), 2,2',4,5,5'-pentachlorobiphenyl (CB101), 2,2',3,4,4',5'-hexachlorobiphenyl (CB138), 2,2',4,4',5,5'-hexachlorobiphenyl (CB153), and 2,2',3,4,4',5,5'-heptachlorobiphenyl (CB180) concentrations followed the method as described in International Standard IEC 61619: Insulating Liquids—Contamination by polychlorinated biphenyls—method of determination by capillary column gas chromatography, except for cleanup step. Instead, of using conventional glass chromatographic

Table 3: Experimental results of PCB congener dechlorination

Run order	Dechlorination temperature (°C)	Dechlorination time (h)	Catalyst weight (%)	PCB congeners (part per million, ppm)					Total PCBs (ppm)	Efficiency (%)
				CB52	CB101	CB138	CB153	CB180		
1	150	1.0	3	0.1776	0.4183	0.1025	0.4795	0.0155	2.7717	12.63
2	150	4.0	3	0.1699	0.4006	0.1304	0.5086	0.0231	2.8422	10.40
3	250	1.0	3	0.1838	0.4775	0.4588	0.5576	0.0220	3.4692	-9.56
4	250	4.0	3	0.1800	0.4192	0.0963	0.5341	0.0164	2.8847	9.06
5	150	2.5	1	0.1747	0.4118	0.1115	0.3515	0.0187	2.6593	16.27
6	150	2.5	5	0.1416	0.3288	0.0836	0.2529	0.0125	3.2254	-1.97
7	250	2.5	1	0.1696	0.3883	0.1023	0.1521	0.0150	2.4155	24.00
8	250	2.5	5	0.1762	0.4474	0.1186	0.5237	0.0168	3.0368	4.21
9	200	1.0	1	0.1769	0.4717	0.1372	0.3765	0.0237	3.3527	-5.89
10	200	1.0	5	0.1729	0.4530	0.1348	0.1839	0.0204	2.6992	14.92
11	200	4.0	1	0.1662	0.4331	0.1152	0.1670	0.0159	2.5559	19.53
12	200	4.0	5	0.1617	0.4027	0.1302	0.1675	0.1484	2.6503	16.53
13	200	2.5	3	0.1574	0.3812	0.1000	0.1513	0.0148	2.4242	23.71
14	200	2.5	3	0.1584	0.3929	0.0900	0.1563	0.0152	2.3713	25.38
15	200	2.5	3	0.1671	0.4275	0.1064	0.1766	0.0159	2.5117	20.93
16 (OIL)	-	-	-	0.2066	0.5300	0.1462	0.2172	0.0275	3.1703	0

column or solid phase extraction the Dextech unit for sample cleanup was used. Using Agilent 7890B gas chromatograph equipped with an electron capture detector, the five PCB congeners are eluted and assessed.

RESULTS AND DISCUSSION

Table 3 presents the results of the dechlorination of polychlorinated biphenyls. Fig. 2 presents the analysis of the data using the Box–Behnken response surface methodology (RSM) with Minitab Software Version 21.1. As shown in Fig. 2a, Fig. 2b, and Fig. 2d, congeners CB52, CB101, and CB153 provided positive responses respectively. Conversely, as shown in Fig. 2c negative response was obtained for CB138 congener. Contrary to the three previous PCB congeners whose levels tended to increase along with the dechlorination process, the CB138 congener tended to decrease along with the dechlorination process. Additionally, as shown in Fig. 2e CB180 is the only congener that has no response to the provided treatments. As shown in Fig. 2f, the analysis of the total congeners demonstrated that no significant effect of the given treatments was found. The results also indicated that dechlorination did not proceed for congeners that contain more than six chlorine atoms. In Table 4, the statistical analysis provided regression coefficients. As shown in Table 4 the regression equation yields an R^2 value greater than 90% for significant responses and less than 75% for inconsequential responses. The

general equation of the regression is presented using Eq. 1 (Myers *et al.*, 2016).

$$\text{Response} = \text{constant} + a (\text{dechlorination temperature}) + b (\text{dechlorination time}) + c (\text{catalyst weight}) + a^2 (\text{dechlorination temperature})^2 + b^2 (\text{dechlorination time})^2 + c^2 (\text{catalyst weight})^2 + ab (\text{dechlorination temperature}) (\text{dechlorination time}) + ac (\text{dechlorination temperature}) (\text{catalyst weight}) + bc (\text{dechlorination time}) (\text{catalyst weight}) \quad (1)$$

When the changes pattern of PCB congeners level and the chemical structures of the congeners were examined then it can be inferred that the dechlorination occurred to chlorine atoms attached to the biphenyl ring at 4 and 4' positions. Thus, it is predicted that the PCB dechlorination proceeds from 2,2',4,4',5,5'-hexachlorobiphenyl via 2,2',4,5,5'-pentachlorobiphenyl and then arrives at 2,2',5,5'-tetrachlorobiphenyl, as shown in Fig. 3. The results of dechlorination trials according to the given combination treatments provided the highest efficiency of total PCB dechlorination was approximately 25% (Table 3). Optimization by implementing the RSM technique on data presented in Table 4 to obtain optimized parameter dechlorination condition provided results in dechlorination temperature 150°C, dechlorination time 2.4 h, and catalyst weight 5%, as presented in Fig. 4. Similar efficiency of dechlorination was

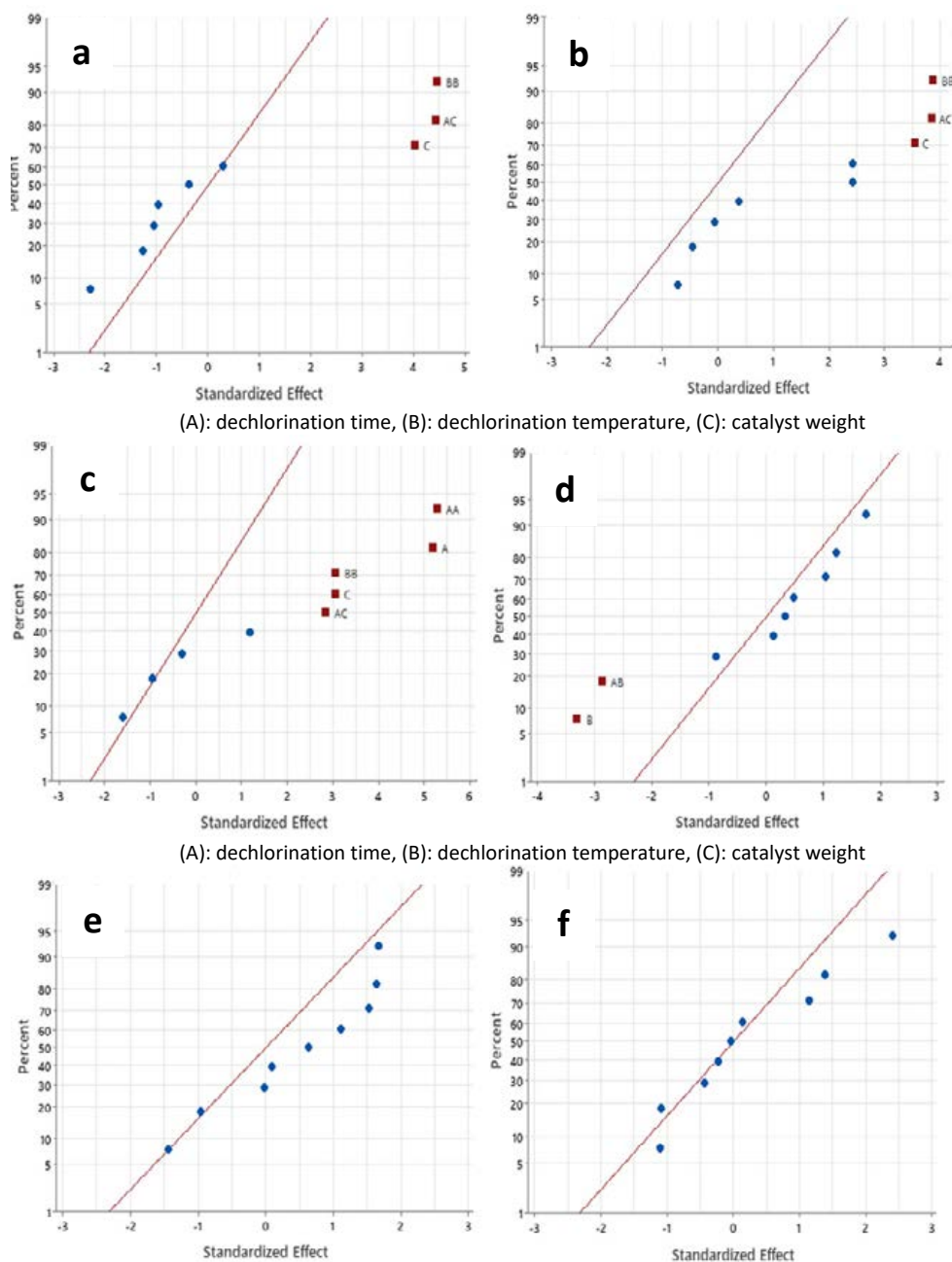


Fig. 2: Effect (at $\alpha = 0.05$) of combination treatments. (A): dechlorination time, (B): dechlorination temperature, (C): catalyst weight on PCB dechlorination: (a) CB52, (b) CB101, (c) C138, (d) C153, (e) CB180, and (f) total PCBs. Dot blue color: not significant, square red color: significant

obtained using sequential combination anaerobic and aerobic microbial treatments of PCBs for a total 98-day experiment comprised of 70 days anaerobic treatment and 28 days aerobic treatment when 5 days

anaerobic and 2 days aerobic cycle treatments were applied (Long et al., 2015). PCB dechlorination favors chlorines usually attached to meta and para positions in comparison to ortho positions (Hansen, 1999). The

Table 4: Regression coefficient and R^2 of PCB congener dichlorination

Term	Code	CB52	CB101	CB138	CB153	C180	Total
Constant		0.3294	0.231	1.377	5.69	-0.390	-6.22
Dechlorination time (h)	a	-0.00407	-0.0631	-0.384	-0.137	-0.0212	0.621
Dechlorination temperature (°C)	b	0.1305	-0.163	0.870	4.484	-0.324	-6.90
Catalyst weight (%)	c	0.02356	0.0836	0.0426	0.325	-0.0012	-0.050
Dechlorination time (h)· Dechlorination time (h)	a ²	0.00461	0.01654	0.0275	0.0583	0.00896	0.0475
Dechlorination temperature (°C)· Dechlorination temperature (°C)	b ²	0.0259	-0.0354	0.146	0.910	-0.0648	-1.299
Catalyst weight (%)· Catalyst weight (%)	c ²	-0.000483	0.00060	-0.00781	-0.0172	0.00416	-0.0073
Dechlorination temperature (°C)· Dechlorination time (h)	ab	0.00131	-0.0135	-0.1302	-0.0175	-0.0044	0.287
Dechlorination temperature (°C)· Catalyst weight (%)	bc	0.00990	0.03553	0.0111	0.1176	0.0020	-0.036
Dechlorination time (h)· Catalyst weight (%)	ac	-0.000048	-0.00098	0.0015	0.0161	0.01132	0.0322
R^2 (%)		91.84	93.59	80.21	91.05	75.30	55.04

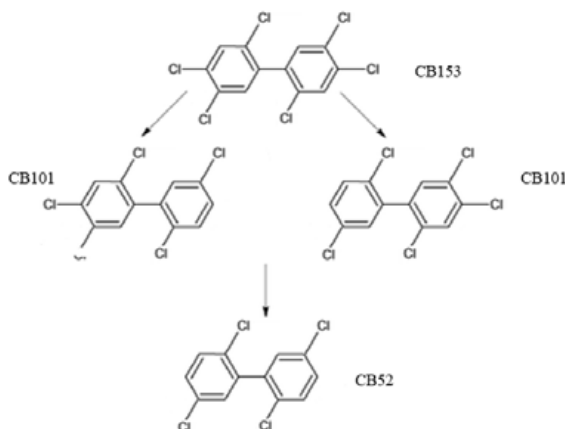


Fig. 3: Sequential dechlorination of CB153 to produce CB52

meta and para-attached chlorines readily undergo dechlorination meanwhile ortho-attached chlorines are the most difficult to remove (Shields *et al.*, 2015). The order and preference of dechlorination depend on the presence or absence of adjacent chlorines as well as the position of the adjacent chlorines (Shields *et al.*, 2015). The density functional theory calculation revealed that the addition of a substituent onto ortho, meta, and para positions resulted in increasing the energy distance of the highest occupied molecular orbital to the lowest unoccupied molecular orbital in that order (Ahmed and Abduljalil, 2019). The results of the calculation were in accordance with the findings of this study, that is, substituents at the para

position are more prone to undergo dechlorination followed by meta and ortho positions.

In this study, the low percentage of PCBs dehalogenation efficiency might be attributed to low active sites of pulverized shrimp shell catalyst. Such low active sites may originate from several causes that include poisoning (Gurraala *et al.*, 2021; Bartholomew and Argyle; 2015) onto the surface of the solid catalyst where impregnated zinc particles are expected to be centers of the active sites. The poisoning could have resulted from chlorine atoms that are detached from biphenyl rings during dechlorination being deposited and then covering the zinc atoms that should act as active sites. This

Dechlorination of selected PCB congeners

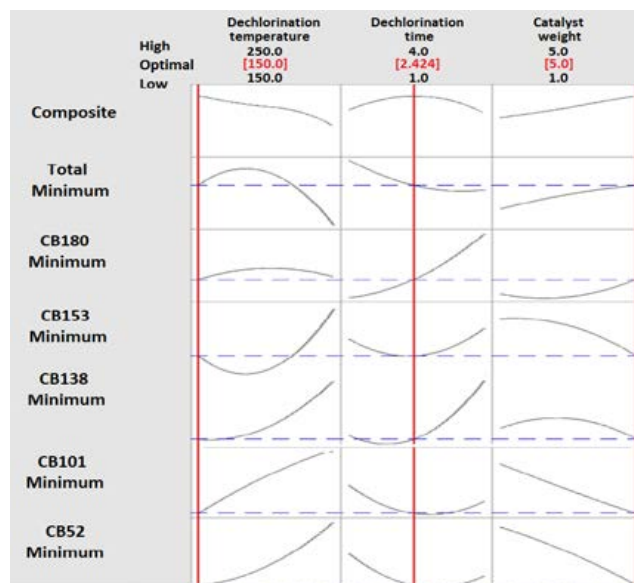


Fig. 4: Minitab output on optimization of PCB dechlorination using the RSM technique

situation may prohibit the zinc atoms from interacting with the remaining PCB particles in the mixture. Another cause of decreasing catalytic activities is related to a leaching phenomenon (Yang *et al.*, 2017; Gui *et al.* 2015) of zinc from the surface of the pulverized shrimp shell. The leaching of zinc can result from mechanical stirring during PCB dechlorination. Leaching of zinc correlates with the mechanical properties of the solid catalyst. Another aspect that should also be considered is the preparation of pulverized shrimp shells through heating in the range of 350°C–550°C followed by quenching. The purpose of the treatment was to obtain an irregular surface solid to promote the impregnation of zinc ions to the solid surface. During the heating treatment of the pulverized shrimp shell, the thermogravimetric analysis (TGA) and differential thermal analysis (DTA slope) sign records, as shown in Table 5, indicate the escape of substances from the matrices of pulverized shrimp shell. The released substances may include hydroxyl, acetyl, and acetamide groups of chitin, as shown in Fig. 5, which should play a prominent role in maintaining active centers through complexation (Nehra *et al.*, 2019). The appearance of two inflection points (breakthrough) in the range of 300 °C – 400 °C and 400 °C – 550 °C as shown in Table 5 is in accordance with two exothermic peaks in another study using pure chitin (Vlaev *et al.*, 2015). The

forementioned groups are expected to retain zinc ions as active centers for catalysis via coordination linkage in the solid matrix of pulverized shrimp shells. Escaping the aforementioned group from pulverized shrimp shells might cause the impregnated zinc in the pores of the solid matrix to leach.

A study of FTIR revealed that several disappearing absorption peaks were detected along with the increasing heating temperatures (Table 6). Such disappearing absorption peaks can be attributed from releasing of the adsorbed water, volatile matters, decomposition of residuals, and release of proteins and the acetyl group of chitin (Biondo *et al.*, 2015). Broad absorption of infra-red that appeared between 3500 and 3200/cm (attributed from N–H groups vibration and also O–H bond stretching of a water molecule) was missing after heating at 350°C. The peak absorption at around 2920 and 2850/cm that originates from the vibration of C–H bond stretching of aliphatic hydrocarbons also disappeared after heating at 350°C. The characteristic absorption peak at 1637/cm that was assigned for the presence of carbonyl (C=O) from amide groups was also missing after heating at 350°C. Heating was continued up to 550°C demonstrating that further disappearance of peak at 1556–1540/cm, which is characteristic of amide, occurred. The rest of the peaks that appeared after heating at 550°C is the characteristic absorption

Table 5: DTA and TGA data records and slope change between temperature range

Temperature (°C)	DTA (μV)	Slope ($\Delta\text{DTA}/\Delta\text{temperature}$)	TGA (mg)	Slope ($\Delta\text{TGA}/\Delta\text{temperature}$)
25	0	0.24	30.53	0.00
50	0	-0.50	30.42	-0.03
100	0	0.34	28.89	-0.03
150	10	0.52	27.42	-0.01
200	30	0.20	26.90	-0.01
250	40	0.68	26.57	-0.02
300	50	1.92	25.49	-0.09
350	100	0.13	20.92	-0.13
400	200	-0.54	14.64	-0.03
450	170	0.71	13.05	-0.03
500	160	-0.29	11.32	-0.04
550	170	-2.72	9.23	-0.04
600	0	-1.05	7.42	-0.01
650	-25	-0.35	7.06	-0.01
700	-50	-0.18	6.75	-0.02
750	-55	-0.22	6.00	0.00
800	-60	-0.19	5.86	0.00
850	-80	-1.52	5.77	0.00

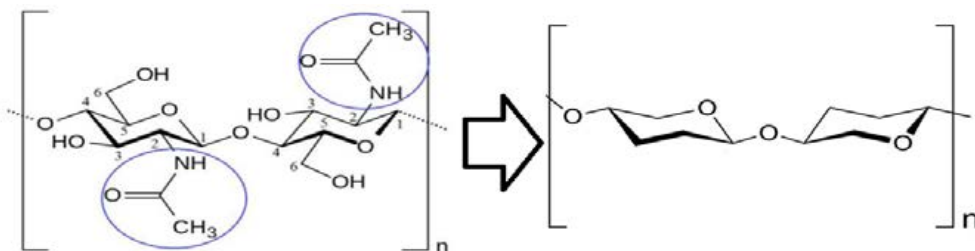


Fig. 5: Releasing of hydroxyl, acetyl, and acetamide groups of chitin

of calcite at approximately 870/cm and also calcium region (Rahman and Halfar, 2014). The study of the FTIR profile shows that the loss of several components from pulverized shrimp shells during heating as depicted by TGA and DTA records was confirmed. The findings of this study demonstrate that heating pre-treatment may affect the release of important functional groups from the pulverized shrimp shell. Such release may significantly affect the integrity of zinc impregnated onto the surface of the pulverized shrimp shell because the missing functional group capable of complexing the zinc may strongly influence the zinc atom to perform as the center of the catalysis sites. Precaution must be established to protect important functional groups that are present in the pulverized shrimp shell as such the synthesized solid heterogeneous catalyst could provide better performance in the dechlorination of PCBs. Compared with several other physicochemical methods in PCB dechlorination, this study demonstrated inferior

results. For example, by using the base-catalyzed decomposition technique, chlorine removal from PCBs of arochlor 242, 1254, and 1260 mixtures obtained 99.8% efficiency when dechlorination was carried out for 4 h at 250°C (Akhondi and Dadkhah, 2018). Another method using potassium polyethylene glycol (KPEG) as a nucleophilic agent that can remove chlorine from PCBs of Sovtol-10 (containing tri to hexachlorobiphenyl) with efficiency in the range of 53%–89% (Velazco *et al.* 2015) if the dechlorination was carried out for 120 min at 90°C. The lowest efficiency was obtained when KPEG/Sovtol-10 ratio was 15, and the highest was obtained when the ratio was 30. The same technique reported no significant chlorine removal of arochlor 1016 after 14 days of evaluation but achieved 99% efficiency for arochlor 1260 in 2 days of dechlorination when the process was carried out at a temperature of 120°C (Tabaei *et al.* 1992). Irrespective of the inferior results of this study, chlorine removal of PCBs is still

Table 6: FTIR wavelength absorption characteristics of pulverized shrimp shell

Absorption (/cm)	Characteristics	Peak at temperature (°C)		
		Ambient	350	550
3500–3200	N–H groups vibration and also O–H bond stretching of water molecule	Present	Disappeared	Disappeared
2920 and 2580	Vibration of C–H bond stretching of aliphatic hydrocarbons	Present	Present	Disappeared
1637	Carbonyl (C=O) from amide groups	Present	Disappeared	Disappeared
1556–1540	Characteristics of amide	Present	Present	Disappeared
1400–800	Calcium region	Present	Present	Present
870	Characteristics of calcite	Present	Present	Present

challenging and needs further study to develop an appropriate set of parameters for chlorine removal. Additionally, the present study offered the utilization of waste materials from shrimp processing instead of nonrenewable materials for catalyst preparation. Certainly, this approach will contribute benefits to the environment, particularly in promoting waste to product approach, as well as the circular economy point of view.

CONCLUSION

The results of this study showed that dechlorination of PCB congeners at temperatures ranging from 150°C to 250°C for 1–4 h using 1–5% catalyst weight provided insignificant reduction levels, that is, 25% at best. Except for the 2,2',3,4,4',5,5'-heptachlorobiphenyl congener that has no effects upon the given treatments of dechlorination, the other four PCB congeners, namely, 2,2',5,5'-tetrachlorobiphenyl, 2,2',4,5,5'-pentachlorobiphenyl, 2,2',3,4,4',5'-hexachlorobiphenyl, and 2,2',4,4',5,5'-hexachlorobiphenyl have little affected by the set dechlorination parameters. Although 2,2',5,5'-tetrachlorobiphenyl, 2,2',4,5,5'-pentachlorobiphenyl, and 2,2',4,4',5,5'-hexachlorobiphenyl demonstrated positive (+) effect response to the given treatments of dechlorination, 2,2',3,4,4',5'-hexachlorobiphenyl showed negative (–) effect response to the given treatments of dechlorination. Optimization of the dechlorination process using RSM based on the given data responses provided optimized dechlorination parameters at 150°C dechlorination temperature, 2.4 h reaction time, and 5% weight/weight proportion of catalyst. The findings also revealed that the dechlorination of the PCB congeners favored chlorine attached at para positions of the biphenyl rings suggesting the transformation of 2,2',4,4',5,5'-hexachlorobiphenyl into 2,2',4,5,5'-pentachlorobiphenyl to

arrive at 2,2',5,5'-tetrachlorobiphenyl. Nevertheless, the dechlorination failed to start with heptachlorobiphenyl containing seven chlorine atoms attached to the biphenyl rings. Irrespective of the low level of dechlorination of PCB congeners, that is, 25%, obtained in this study, the microwave-assisted impregnation method could be used to apply for the synthesis of a heterogeneous catalyst made from shrimp shell waste. However, heating pre-treatment of the pulverized shrimp shell waste to 550°C followed by immediate quenching has ended up with a material composed of calcium carbonate predominantly containing calcite mineral. This will have a consequence of decreasing the robust mechanical strength of the matrix to maintain zinc metal to be active as center sites for the dechlorination process. Thus, certainly, this is a drawback or limitation of the heating method at high temperatures to synthesize catalysts from pulverized shrimp shell waste. Therefore, for future trials, precautions must be anticipated to protect the important groups, such as hydroxyl, acetamide, and amide that are present in the shrimp shell from releasing during catalyst preparation as those groups play a prominent role to maintain the zinc metal at certain positions to perform as center sites for PCB dechlorination.

AUTHOR CONTRIBUTIONS

D.B. Aviantara contributed to the data collection and observation, analysis, manuscript preparation and revision. N.S. Indrasti supervised and sharpened the background, extended the discussion, and improvement recommendations. G. Hadiko contributed on interpretation of analytical data and statistical data, depended the discussion, and prepared the manuscript. M. Yani, the corresponding author, supervised the manuscript preparation, revised the discussion, and proofread the manuscript.

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CONFLICT OF INTEREST

The author declares that there is no conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, and redundancy have been completely observed by the authors.

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ABBREVIATIONS

%	Percent
/cm	Wavenumber
<i>a</i>	Regression coefficient for dechlorination time
<i>b</i>	Regression coefficient for dechlorination temperature
<i>c</i>	Regression coefficient for catalyst weight

<i>A</i>	Dechlorination time
<i>B</i>	Dechlorination temperature
<i>BET</i>	Brunnauer Emmet Teller
<i>BRIN</i>	Badan Riset dan Inovasi Nasional
<i>BZ</i>	Ballschmitter and Zell
<i>C</i>	Carbon atom
<i>C</i>	Catalyst proportion
°C	Degree Celsius
<i>CB</i>	Chlorinated biphenyls
<i>CB52</i>	2,2',5,5'-tetrachlorobiphenyl
<i>CB101</i>	2,2',4,5,5'-pentachlorobiphenyl
<i>CB138</i>	2,2',3,4,4',5'-hexachlorobiphenyl
<i>CB153</i>	2,2',4,4',5,5'-hexachlorobiphenyl
<i>CB180</i>	2,2',3,4,4',5,5'-heptachlorobiphenyl
<i>Cl</i>	Chlorine atom
<i>cm</i>	Centimeter
<i>DTA</i>	Differential thermal analysis
Δ DTA/	Delta DTA / delta temperature
Δ temperature	
<i>DTG</i>	Differential thermal gravimetric
Δ TGA/	Delta TGA / delta temperature
Δ temperature	
<i>Eff.</i>	Efficiency
<i>FTIR</i>	Fourier Transform Infrared
<i>GC/ECD</i>	Gas Chromatograph/Electron Capture Detector
<i>g/mole</i>	Gram/molecule
<i>H</i>	Hydrogen atom
<i>h</i>	hour
<i>IEC</i>	International Electrotechnical Commission
<i>IPB</i>	Institut Pertanian Bogor
<i>KLHK</i>	Kementerian Lingkungan Hidup dan Kehutanan (The Ministry of Environment and Forestry – the Republic of Indonesia)

<i>KPEG</i>	Potassium polyethylene glycol
<i>kW</i>	Kilowatt
μm	Micrometer
μV	Microvolt
m^2/g	Square meter per gram
<i>Min.</i>	Minute
<i>N</i>	Nitrogen
<i>O</i>	Oxygen
<i>PCBs</i>	Polychlorinated biphenyls
<i>POPs</i>	Persistent organic pollutants
<i>ppm</i>	Part per million
<i>RSM</i>	Response Surface Methodology
<i>SC</i>	Stockholm Convention
<i>SEM</i>	Scanning Electron Microscopy
<i>TGA</i>	Thermogravimetric analysis
<i>UNIDO</i>	United Nations Industrial Development Organization
<i>w/w</i>	Weight to weight ratio
<i>XRD</i>	X-ray diffraction
<i>Zn</i>	Zinc

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