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Use of Bamboo Powder Waste for Removal of Bisphenol A in Aqueous Solution

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ABSTRACT

The scarcity of clean water affecting many parts of the world encourages efforts to improve water reclamation processes, which rely on their capability to remove diverse types of water pollutants and contaminants. Thus this study reports the application of bamboo fiber powders as potential low-cost sorbent for removal of noxious organic compounds in aqueous solution. Bisphenol A, a bio-refractory endocrine disruptor compound, was chosen as model compound in order to easily follow the separation process. Principal component analysis of the FTIR spectra and BET surface area measurements were performed on treated bamboo fiber powders. Treatment of the raw powders with alkali, ionic and non-ionic surfactants appeared to improve the bisphenol A removal performance of the bamboo fiber powders with the best removal efficiency reached at 39 % for a sorbent dosage of 4 g.L⁻¹ gained after a bamboo treatment using the cationic surfactant. Effects of contact time, sorbent dosage and particle sizes (55µm, 300 µm and 1000 µm) of cationic surfactant treated bamboo fiber powders towards removal of bisphenol A were further assessed in a batch system with an optimum removal observed for 55 µm particle size.

Keywords: bamboo, bisphenol A, biosorbent, surfactant

1 **Introduction**

2 The scarcity of clean water affecting many parts of the world encourages efforts to improve water reclamation
3 processes, which rely on their capability to remove diverse types of water pollutants and contaminants. Moreover
4 the rising quantity of wastes released into water bodies and the emergence of new varieties of noxious pollutants
5 such as endocrine disruptor compounds (EDCs), as a result of rapid industrial and urban development, necessitate
6 approaches for emerging pollutant removal from water treatment units as well as from wastewater to prevent
7 leaching into the environment. Bisphenol A (BPA) is an estrogenic recalcitrant compound frequently used as
8 additive in polycarbonate and epoxy resins and used globally in numerous consumer products including the lining of
9 food and beverage goods in order to prevent contamination and to extend shelf life (Krishnan et al., 1993; Schug et
10 al., 2012). This endocrine-disrupting chemical is often discharged by factories or leaks out from thrown litter into
11 water bodies. Statistics show that untreated leachate concentrations at Japanese landfills reached a maximum value
12 of 17.2 mg/L (Yamamoto et al., 2001). This refractory pollutant eventually get transferred from wastewater
13 treatment plants effluents and waste landfill leachates into the aquatic system and necessitate lengthy period to be
14 eliminated naturally under aerobic conditions (Kang et al., 2007; Suzuki et al., 2004). This stresses the need for a
15 simple and cost-effective way to remove BPA from aqueous solutions.

16
17 Various methods for removal of recalcitrant EDCs in water and wastewater treatments had been devised. These
18 methods include physical separation such as membrane filtration and adsorption, chemical treatment with advanced
19 oxidation process or biological degradation using microorganism (Masuda et al., 2007; Matsumura et al., 2009;
20 Oshiman et al., 2007; Rubin and Soto, 2009; Sasaki et al., 2005; Vandenberg et al., 2007; Yamanaka et al., 2007).
21 Among these methods, adsorption is a simpler and more adaptable process compared to removal through membrane
22 filtration or advanced oxidation processes (Grassi et al., 2012) as it requires lower capital investment with lesser
23 procedures involved in the treatment. Besides operating as stand-alone process in column reactor, adsorption system
24 has also been adapted as membrane pre-treatment method to remove varieties of organic and inorganic contaminants
25 in order to reduce the incidences of membrane fouling (Tsujimoto et al., 1998). The most commonly used sorbent
26 material for removal of organic compound is activated carbon, however they present several limitations such as
27 flammability, regeneration difficulty and presence of impurities which renders them weakly hydrophilic (Blocki,
28 1993). Efforts to develop alternative sorbent materials to handle the limitations of activated carbon for removal of

organic aromatic pollutants such as BPA had been explored, among these adsorbent materials are carbon nanomaterials (Lin and Xing, 2008; Zhao et al., 2015), metal organic frameworks (Park et al., 2013; Qin et al., 2015), silicate based mesoporous materials (Fan et al., 2011; Kim et al., 2011), clay or mineral materials (Wang et al., 2015) and zeolite (Dong et al., 2010; Tsai et al., 2009). Higher preparative cost of most of these novel sorbents has driven the research on the usage of waste products of biological origin or bio-sorbent as they are versatile, inexpensive, abundant and broadly attainable (Bailey et al., 1999). However, only few studies had been done on the utilization of wastes for removal of organic pollutants. Due to their inferior adsorptive nature, these wastes often require physical and chemical pre-processing prior to application as efficient sorbent materials (Zhao et al., 2013; Zhou et al., 2012). Agricultural wastes such as coir pith, coconut shell and durian peel have been treated with concentrated 98 % sulfuric acid and reported for their enhancement of BPA adsorption with maximum adsorption capacity in the range of $\sim 4 \text{ mg.g}^{-1}$ (Lazim et al., 2015). Modified peat waste has also been reported with maximum BPA sorption capacity of 31.40 mg.g^{-1} (Zhou et al., 2011).

Bamboo forests of diverse species are prevalent in Asia-Pacific areas (Bystriakova et al., 2003) and are maintained due to their role in sustaining ecological, social and economic stability of the regions, while industry thereof enables the availability of large amounts of waste which can be used for such industries as described herein. Bamboo-derived materials have been applied as bio-sorbents for removal of heavy metals in their native form (Asberry et al., 2014; Hsu et al., 2009; Luo et al., 2011; Mondal et al., 2013). These studies utilize the negative surface charge of bamboo sorbent, induced by adjustment of pH, to remove the cationic metal ions from the solution. To date, there is still limited studies being done on the removal of organic material such as BPA using bamboo-based materials (Jiang et al., 2013). Bamboo fiber powder is one of the waste products of bamboo poles slicing and sanding of bamboo planks. This byproduct is generally thrown away or burnt by the factories which may pollute the air. Here, we evaluate the potential usage of bamboo fiber powders as an alternative low-cost adsorbent for removal of BPA among many other organic contaminants. In previous studies, chemical treatments of biomass with surfactants have been reported to improve their adsorption performance towards heavy metals (Bai and Abraham, 2002; Mondal et al., 2013). However, there is a lack in the comparison studies on the effect of treatments of biomass using different type of surfactants on removal of organic pollutant. This work focused on chemical modification of bamboo fiber powder waste as sorbent for BPA removal. In this study, the bamboo fiber powders were treated with ionic and non-

1 ionic surfactants, alkali and acid solutions. The treated bamboo powders were characterized and their performances
2 for removal of BPA were evaluated. The treatment producing the best improvement in BPA removal was then
3 selected and studied further in batch reactor. The effect of particle sizes and adsorption isotherm were elaborated.

4 5 6 **Materials and methods**

7 8 **Chemicals**

9 All chemicals used in this study were of analytical grade. Bisphenol A was obtained from Sigma (BPA,
10 $(\text{CH}_3)_2\text{C}(\text{C}_6\text{H}_4\text{OH})_2$, Sigma 239658). Stock solution of 100 mg.L^{-1} of BPA was prepared by stirring in de-ionized
11 distilled water for 2 d. Bamboo fiber powders of $55 \mu\text{m}$ (Grade 6), $300 \mu\text{m}$ (Grade 4) and $1000 \mu\text{m}$ (Grade 3)
12 particle sizes were obtained from Bamboo Fibers Technology. The herein employed bamboo fiber powders were
13 collected by passing the sawdust from 5-year old *Thysostachys siamensis* Gamble and *Dendrocalamus strictus*
14 Nees bamboo species through meshes until uniform size distribution of the powder particles were achieved.
15 Sodium dodecyl sulfate (SDS, $\text{C}_{12}\text{H}_{25}\text{NaO}_4\text{S}$, Sigma L3771), Hexadecyltrimethylammonium bromide (CTAB,
16 $\text{C}_{19}\text{H}_{42}\text{BrN}$, Sigma H6269), Triton X-100 (TX-100, $t\text{-Oct-C}_6\text{H}_4\text{-(OCH}_2\text{CH}_2)_x\text{OH}$ with $x=9\text{-}10$, Sigma T9284)
17 were of analytical grade.

18 19 **Preparation of bamboo fiber sorbents**

20 Solutions of 0.1 N sodium hydroxide and hydrochloric acid were used for alkali/acid treatments. Aqueous solutions
21 of SDS, CTAB and TX-100 at their respective critical micelle concentrations of 8 mM, 0.9 mM and 0.2 mM
22 respectively were selected. In brief, 2 grams of bamboo powder was mixed with the acid/alkali/surfactant solutions
23 and stirred for 24 h at ambient temperature of 22°C . After treatment, dry powder was prepared by centrifugation of
24 the solution at 9,000 rpm for 5 min followed by removal of supernatant and washing with distilled deionized water.
25 The centrifuge-washing process was repeated at least twice. The obtained powder was then dried at 80°C overnight.

26 27 **BPA removal experiments**

Sorbent was added into BPA solutions of known concentrations in glass containers. The containers were agitated at 150 rpm at 30°C with an incubator shaker. Aliquots of the solution were taken at specified times to monitor the putative removal of BPA from the aqueous solution over time. The pH of the BPA solution was adjusted using 0.1 N hydrochloric acid and 0.1 N sodium hydroxide. BPA removal efficiency and adsorption capacity was calculated as follows:

$$R_t (\%) = (1 - [BPA]_t/[BPA]_0) \times 100 \% \quad (1)$$

$$q_t = ([BPA]_0 - [BPA]_t) \times V/m \quad (2)$$

where R_t represents the percentage of BPA removed at time t ; $[BPA]_0$ and $[BPA]_t$ represent concentration of BPA in the solution (mg.L^{-1}) initially and after time t ; q_t (mg.g^{-1}) represents the amount of BPA adsorbed at time t , V (L) and m (g) refer to volume of the solution and mass of the adsorbent respectively. The experiments were performed independently in duplicate with the mean value of the readings presented on the graphs.

Characterizations

Measurement of BPA concentration

BPA concentrations were estimated based on the absorbance value measured using a UV-Vis spectrophotometer (Shimadzu UV3600) at the wavelength of 276 nm. The concentration of the BPA was inferred based on the calibration curve of the solution with known BPA concentrations.

Zeta potential

Zeta potential measurements were performed using Malvern Zetasizer Nano ZS. One gram per litre dosage of bamboo fiber powder particles was added into tubes containing distilled water at different pH. The pH of the solution was adjusted using 0.1 N hydrochloric acid and 0.1 N sodium hydroxide. 750 μl of the sample was placed into the cell and run for 5x with 10 measurements for each run.

Fourier Transform Infra-red Spectroscopy

Fourier Transform Infra-red Spectroscopy (FTIR, Perkin Elmer Spectrum) was used at transmission spectra of 4000-400 cm^{-1} at resolution of 4 cm^{-1} with each sample scanned for 60x. Approximately 2 mg of each sample was mixed with 400 mg of KBr powder to prepare the pellet with triplicate experiments per sample.

Principal Component Analysis

Principal component analysis (PCA) is a chemometric method for handling multi-variable data sets. The objective of PCA is to group the variables into a smaller set of principal components which depict the variability of the data sets (Jolliffe, 2002). Application of PCA on FTIR spectrum will enable easier discrimination of indistinguishable spectra from multiple datasets as well as identification of the bands responsible for the discrimination. Matlab (v.2012b) and “Saisir package” (Cordella and Bertrand, 2014) was used to perform the Principal component analysis (PCA) “PCA function” and the analysis of variance (ANOVA) “Anova1 function”. Before classification, spectra were centered by subtraction of the average of each group. The observation and discrimination between spectra were based on PC1 and PC2 scores because these first principal components explain the majority of variance of the dataset.

N₂ adsorption/desorption analysis

The surface area, pore volume and average pore diameter were estimated based on Brunauer-Emmet-Teller (BET) and Barrett-Joyner-Halenda (BJH) methods using Quantachrome Nova 3200E.

Results & Discussion

Effect of contact time and chemical treatments of bamboo powder on removal of BPA

As a preliminary experiment, the contact time required to reach equilibrium in the batch study was evaluated. As control, treated bamboo powders were incubated at 30°C and 150 rpm in distilled de-ionized water for more than 24 h. **Figure 1** shows the fast sorption kinetics of 100 mg.L⁻¹ BPA on 4 g.L⁻¹ dosage of untreated and pre-treated bamboo powders. It can be observed that the percentage of BPA removed from the solution increases with time for both pre-treated and untreated BPs. The rapid sorption at the first hour may be attributed to the high availability of binding sites on the adsorbent surface. The BPA removal percentage gradually lessened and attained equilibrium condition at a contact time of less than 8 h independent of the adsorbent types. A contact period of more than 8 h is therefore selected for subsequent batch experiments for BPA removal.

Effect of chemical treatments of bamboo powders with different surfactants, acid and alkali towards the removal of BPA is also shown in **Figure 1**. It can be seen in **Figure 1** that untreated bamboo powders is capable to remove BPA with an average percentage removal of 13 ± 0.40 % from initial BPA solution of 100 mg.L^{-1} . The pre-treatment of bamboo powders has been observed to generally improve the BPA removal according to the following order: CTAB-treated bamboo powders (39 ± 2.97 %) > SDS-treated bamboo powders (33 ± 1.49 %) > NaOH-treated bamboo powders (29 ± 0.78 %) $\approx$ TX-100 treated bamboo powders (28 ± 2.23 %) > HCl-treated bamboo powders (15 ± 0.40 %) > untreated bamboo powders (13 ± 0.40 %). Based on the result in the preliminary batch experiment, the CTAB-treated bamboo powders (CBPs) which showed the highest improvement in BPA removal was selected for subsequent adsorption experiments.

Effect of bamboo powders dosage in BPA removal

The adsorption profile of BPA at different CBPs dosage of 2, 4, 8, 15 and 30 g.L^{-1} is shown on **Figure 2**. It is apparent that the percentage of BPA removal increased with the increase in CBPs dose. This pattern can be attributed to the increase in surface area and availability of active sites for adsorption with the increase in adsorbent dose (Sari and Tuzen, 2009).

Sorption isotherm at different particle size

The sorption isotherm relates to the amount of sorbate adsorbed per weight of adsorbent phase with the amount of solute remaining in the aqueous phase in equilibrium under constant temperature. The isotherm result of BPA adsorption in aqueous solution onto CBPs is shown in **Figure 3**. It can be deduced that the adsorbed amount of BPA per g of CBPs at equilibrium is in proportional relationship with the residual BPA concentration in the solution for the particle sizes tested with reasonable linearity over the range of tested concentrations. In this study, linear and Freundlich adsorption isotherm models were used to provide a quantitative evaluation of the system. The linear model is the simplest sorption isotherm model and typically infers that the energies involved in the sorption are uniform with the increase in solute concentrations. This model is generally associated to a system with very low solute concentration or sorbent with low sorption potential (Pal and Deb, 2012). The linear model is described as follows:

$$q_{e,\text{exp}} = K_d \times C_e \quad (3)$$

where $q_{e,exp}$ ($mg.g^{-1}$) is adsorption of BPA by the CBPs at equilibrium, $C_{e,exp}$ ($mg.L^{-1}$) is the equilibrium concentration of BPA in aqueous solution and K_d ($L.g^{-1}$) is the linear partition or distribution coefficient of BPA in the aqueous solution and CBPs.

Freundlich isotherm (Sasaki et al., 2005) is the other commonly used isotherm model. The model stands on the assumption that the interaction between equilibrium concentration of the solute in aqueous solution and solid phase is governed by multilayer adsorption on heterogenous surfaces. The Freundlich model is described as:

$$\log q_{e,exp} = (1/n) \times \log C_{e,exp} + \log K_F \quad (4)$$

Where the definition for $C_{e,exp}$ ($mg.L^{-1}$), $q_{e,exp}$ ($mg.g^{-1}$) is the same as described previously for a linear model; K_F ($mg^{1-1/n}.L^{1/n}.g^{-1}$) refers to the Freundlich constant in multilayer adsorption capacity and $1/n$ (constant) represents sorption intensity. The Freundlich coefficients, K_F and n , can be obtained by plotting $\log q_e$ over $\log C_{e,exp}$.

The data from adsorption isotherm study, as shown in **Figure 3** and **Table 1**, follow both linear and Freundlich models with correlation coefficients values above 0.910. High correlation with Freundlich model suggested that the neutral BPA molecules were adsorbed on the surface of CBPs in this system in multi-layers fashion. It is observed that the K_F values, which indicate that the adsorption capacity declined at bigger particle sizes. It is expected that higher BPA removal can be attained using smaller particle size due to the higher surface area available for attachment of surfactant molecules during treatment and consequently higher BPA retention. This observation indicated that smaller particle size of CBPs exhibits better adsorption capacity. The linearity of the isotherm indicated the role of a hydrophobic partitioning process in the system (Bée et al., 2011). The value of sorption intensity ($1/n$) from Freundlich model at pH 6.0 for particle size of 55 μm is closed to unity which further supported the notion of linearity of the system. Similar linearity behavior has been reported in other sorption systems with natural organic materials (or bio-sorbents) such as in the case of pentachlorophenol adsorption on pine bark whereby the $1/n$ constant value is also close to unity (Brás et al., 2005). From the sorption isotherm data attained at equilibrium for different particle sizes at pH 6.0, the value of linear distribution coefficient K_d for larger particles was lower in comparison to smaller particle size. This poorer sorption performance resulted in the fact that, under

the same dosage, particles with smaller size will have increased available surface areas for attachment of surfactant, and consequently the available binding sites, to interact with BPA or any other organic compounds.

The maximum sorption capacity of CBPs used in our study was determined based on the sorption at initial BPA concentration of 100mg.L^{-1} , which was the highest BPA concentration tested in our study. While direct comparison with the results of previous studies is intricate due to the difference in experimental parameters such as initial concentrations, dosage and temperature, the maximum BPA sorption capacities of CBPs in our study are similar in values to carbonaceous materials reported in the literature (Asada et al., 2004; Lazim et al., 2015; Nakanishi et al., 2002; Zhou et al., 2011). Nevertheless, it should be noted that most of these bio-derived sorbent materials reported requires preparation at high temperature of $400^{\circ}\text{C} - 1000^{\circ}\text{C}$ for carbonization. The exclusion of the need for preparative treatment at high temperature makes our CBPs simpler to be prepared in comparison to sorbent derived activated carbon. It is worth to note that cationic surfactant modified peat (Zhou et al., 2011), another low-cost sorbent, had been reported previously for their high BPA sorption capacity of up to 31.40 mg.g^{-1} . However the concentration of surfactant used during the modification was much higher (0.1 M and 50 mM), which on itself may be a potential source of pollution, than the one used in our study (0.9 mM). Moreover, bamboo based sorbents have been demonstrated recently to show affinity towards heavy metal pollutants (Asberry et al., 2014; Mondal et al., 2013). Their capability to be used for adsorption of diverse range of pollutant species will enable them to be used in column reactor for simultaneous removal of multiple pollutants from water. Since bamboo powder waste is abundant and can be easily prepared, the low-cost adsorbent proposed may have application potential for removal of non-polar aromatic pollutants in developing countries.

Proposed mechanism

Bamboo is a type of lignocellulosic biomass which is composed of a porous structure of hemicelluloses, cellulose and lignin in layered formations (Liese, 1985). **Table 2** shows the surface analysis results of bamboo powders before and after treatment. It has been known that the physical characteristics of bio-sorbents differ with the source of plant materials used; in this study the BET surface area of untreated bamboo fiber powders with $300\mu\text{m}$ particle size were estimated to be $6.682\text{ m}^2.\text{g}^{-1}$. The measured surface area for untreated bamboo powders is slightly higher than the reported surface area for water bamboo husk particle with BET of $1.866\text{ m}^2/\text{g}$ (Asberry et al., 2014).

Surface analysis measurements of pre-treated bamboo powders (**Table 2**) depict the significant increase in surface area for CTAB treated bamboo powders. While it can be deduced that removal of BPA by CTAB treated bamboo powders is contributed by the higher available adsorption sites in comparison to untreated bamboo fiber powders, the surface area measurements of bamboo powders treated with non-ionic, anionic surfactants and alkali solutions do not differ much in comparison with untreated bamboo powders, suggesting the presence of other factor influencing the adsorption of BPA besides surface area. The PCA analysis of FTIR spectrums for treated and untreated bamboo powders is shown in **Figure 4**. The obtained results show that the acid treatment by HCl does not affect the bamboo surfaces (results are similar to untreated surfaces). According to these results, HCl spectra were eliminated and not used in the statistical methods (PCA and ANOVA).

The plot of PCA scores (PC1 and PC2) on **Figure 4(a)** shows the presence of 3 distinct groups with 80.2 % variance explained by PC1 scores. Spectra of CTAB and NaOH treatment are classified each in separate group while spectra of untreated, SDS and Tx-100 treatment were classified in the same one. This result infers that interactions between the reactants and bamboo powders have taken place after pre-treatment of bamboo powders; ANOVA test performed on the first PCA score on **Figure 4(b)** confirms that CTAB and NaOH treatments are different to the other treatment. The analysis of the first PCA loading on **Figure 4(c)** attributes the observed discrimination to 5 bands with strong contribution of the 820 cm^{-1} and 2910 cm^{-1} regions, corresponding to the asymmetric C-H bending of CH_2 group and CH_2 stretching and bending respectively. Taken together with the surface area measurement results, the FTIR analysis supported the notion that BPA removal by treated bamboo fiber powders may be influenced by other factors besides surface area.

Figure 5 shows the zeta potential measurements of untreated and CTAB treated bamboo fiber powders. It appeared that the surface of untreated bamboo fiber powders is negatively charged within a pH range of 3-11, while its treatment with CTAB resulted in the increase to generally a more positive surface zeta potential at pH lower than 10.5, which may be due to the displacement of natural counter-ion on the surface of untreated bamboo powders with cationic head of CTAB.

Inspired by a previous study (Dong et al., 2010) and aforementioned observations, we propose the possible interactions that occurred during pre-treatment of bamboo fiber powders and their resulted surface modifications after washing in **Figure 6(a) and 6(b)**. These surface modifications may inadvertently contribute towards the improvement in BPA removal by the treated bamboo powders. **Figure 6(a)** depicts the surface of untreated bamboo fiber powders during chemical treatment with cationic surfactant CTAB. The positively charged head of the surfactant will be oriented towards the negatively charged surface of bamboo fiber powders due to electrostatic attraction while the lipophilic tail may adopt either in a perpendicular orientation or become adsorbed on their surface due to hydrophobic interaction with aromatic groups and adopt a flat or tilted orientation. In this experiment, the pH of the BPA solution was adjusted to 6.0, less than the pKa of BPA which is reported to be in a pH range of 9.6-10.2 (Dorn et al., 1987) in which case the BPA will exist in its anion form. The BPA molecule at pH less than its pKa presents itself in its neutral form and may act as moderately hydrophobic organic compound with log K_{ow} of 3.4 (Munari et al., 1988). The elevation in BPA removal by pre-treated bamboo fiber powders may be attributed to the loading of surfactants. The surfactants may exist in a monolayer or bilayer form at sufficient concentration and thus, create a hydrophobic environment. The interaction of the surfactant with BPA is influenced by Van Der Waals and London forces and is supported by a partitioning process of hydrophobic BPA into the organic hydrophobic phase constructed by a lipophilic tail of the surfactant, the two hydrophobic benzene rings in BPA molecules will thus interact with the lipophilic tail of the surfactant and improve the affinity to BPA, which together result in hydrophobic sorption (Choi et al., 2011). In addition to the hydrophobic interaction, the neutral form of BPA may interact with the positively charged head of the surfactant due to the presence of oxygen as an electron donor in the phenol group of BPA with a positively charged head of the surfactant acting as electron acceptor.

In this regard, the highest improvement in BPA removal exhibited by CBPs is postulated to be due to the presence of both a positively charged cationic head and a lipophilic tail which both contribute towards BPA retention. The capability of untreated bamboo fiber powders to remove BPA from the solution, as shown in **Figure 6(b)** may be attributed to the amount surface area available as adsorption sites in the bamboo powders through a pore-filling mechanism, as well as, hydrophobic interaction between aromatic groups in bamboo fiber powders and benzene rings in BPA.

Conclusion

Bamboo fiber powders, easily derived from abundant bamboo industries can be used as an alternative sorbent for the removal of aromatic organic material. Their sorption capacity may be increased through pretreatment with surfactants and alkali solutions. Among the pre-treatment tested, CTAB-treated bamboo fiber powders showed highest improvement in removal of pollutant BPA from aqueous solution. Batch experimental studies indicated that the adsorption process in this system is dosage and particle size dependent and occurs in multi-layered fashion. The exclusion of the need for preparative treatment at high temperature makes these CBPs simpler to be prepared.

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