

INTERFACE SCIENCE AND TECHNOLOGY

# Advanced Low-Cost Separation Techniques in Interface Science

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Edited by  
George Z. Kyzas  
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# Advanced Low-Cost Separation Techniques in Interface Science

# Interface Science and Technology

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# Advanced Low-Cost Separation Techniques in Interface Science

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Edited by

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# Low-cost materials in gas-phase adsorption

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## 1. Introduction

Adsorption technology is broadly applied in many fields. Industrial application of adsorbents is mainly focused on wastewater treatment and water purification [1,2], namely for removal of dyes [3,4], heavy metal ions [5,6], and other compounds, i.e., pharmaceuticals [7]. Comparing available literature for liquid-phase adsorption with that of gases adsorption, there is a gap for the latter case, especially concerning the application of low-cost and environmental friendly materials. A low-cost material is best determined by the precursor used for its fabrication; some of the criteria considered are its abundance/availability, the ratio of material obtained per amount of raw material needed, adsorption capacity, etc. [8] On the other hand, eco-friendly materials are those that conform with green technology in respect to their synthesis process as well as their reusability [9]. Many of the aforementioned aspects are reached by the implementation of carbon-based materials derived from various sources: biomass [10], wastes of many different kinds (food waste [11,12], tires [13,14], agro-wastes [15,16], etc.), and others [17].

Application of adsorbents for gas-phase adsorption is concentrated on toxic and hazardous gases produced by industrial processes and other sources, involving human activity. Greenhouse gases are those of high concern due to the formation of greenhouse effect. According to Intergovernmental Panel on Climate Change (IPCC), greenhouse effect is the warming process of the planet's surface due to radiation retainment by its atmosphere. Climate change and environmental protection is the number one priority worldwide, while

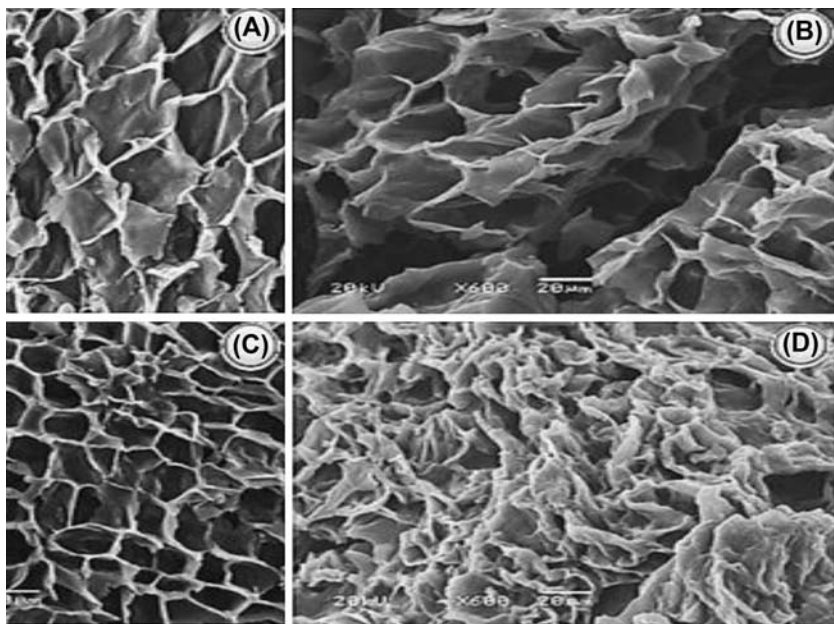
technologies toward mitigation of greenhouse gas emissions are developing and upgrading constantly to reach the desired balance [18].

As it was already mentioned, carbonaceous materials are the most common adsorbents because of their physical and, when modified, physicochemical properties. Materials such as activated carbon, biochar, and their composites are of big interest through older and recent history. The presented work has six sections; an introduction part where the most common carbonaceous materials (activated carbon and biochar) are briefly presented; the second section includes adsorbent trends for CO<sub>2</sub> capture; in the third section the reduction technologies of acidifying gases (NO<sub>x</sub> and SO<sub>x</sub>) are discussed; the fourth section presents adsorbents used for CH<sub>4</sub>; the fifth for volatile organic compounds (VOCs), and in the last sections there are conclusions based on the context of this chapter. Although there are a sufficient number of released reviews on gas-phase adsorption, most of the works select to discuss adsorbents applied for adsorption of one gas at a time, with CO<sub>2</sub> holding a leading role. Finally, this chapter provides a summarized overview of updated studies in respect to carbonaceous materials for gas-phase adsorption with focus on the main greenhouse gases. Materials for VOCs adsorption are discussed briefly as well, while at the end of each section there is a table for a quick overview of recently developed materials.

## 1.1 Activated carbon

Activated carbon is a well-known material, presenting high surface area, mechanical stability, and reusability. The common procedure for activated carbon synthesis is a two-step process: (1) carbonization of the precursor and (2) activation of the resulted char [19]. There are many cases, that activation step is completed along with carbonization, so we have only one-step process. Most of the cases of the latter, activation that takes place is a physical one with thermal treatment at a selected gas atmosphere (i.e., activation with CO<sub>2</sub>), while when chemical activation is selected, the process performed is usually a two-step one [20]. The obtained properties of the final material are related to many parameters. For example, both nature of the precursor and synthesis conditions have a crucial impact on the resulted characteristics of adsorbent [6,21]. One of the most important factors that affect surface properties of the activated carbon were found to be carbonization and/or activation temperature like literature suggests. In Fig. 6.1, one can realize the role of carbonization temperature on the evolution of pores at wood-based activated carbon surface. The pore structure of activated carbon is usually micropore-dominant, a fact that makes it a strong candidate for gas-phase adsorption [22].

Modification of activated carbon surface can lead to improvement of adsorption, mechanical, and other properties of the material. Modification is accomplished by introducing functional groups onto the surface resulting in



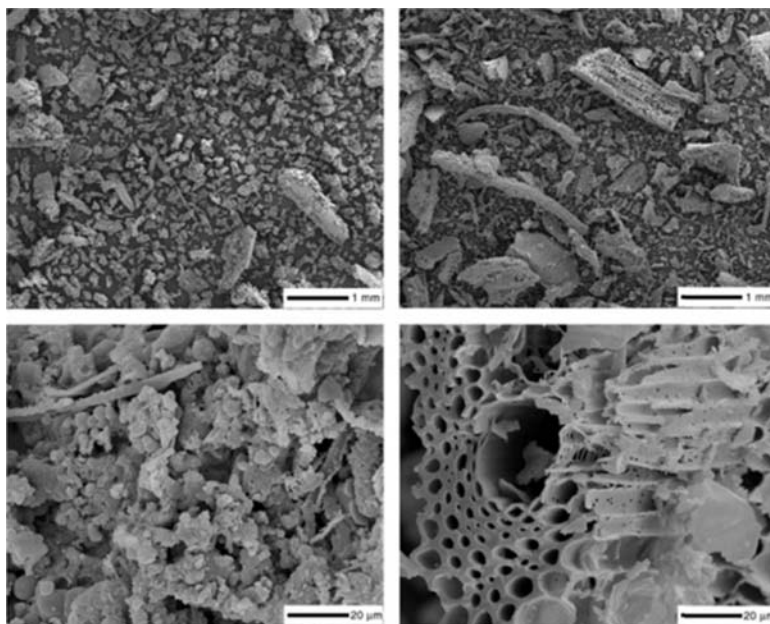
**FIGURE 6.1** SEM micrographs of wood-based activated carbon at different carbonization temperature with  $\text{H}_3\text{PO}_4$  impregnation ratio of 4 at (A) 300°C, (B) 400°C, (C) 500°C and (D) 600°C [23].

chemical properties alteration, hence achieving higher adsorption capacity of the target adsorbate [24,25].

## 1.2 Biochar

In contrast to activated carbon, biochar is commonly produced after slow pyrolysis usually at low temperature and total absence of oxygen [26,27]. Additionally, a recent trend is hydrothermal treatment of biomass, concluding in the so-called hydrochar. Hydrochar production process offers some significant advantages over the former; no necessary drying of biomass, no emissions, and by-products compared with pyrolysis as well as higher carbon yield are some of them [28]. As it is shown in Fig. 6.2, structural characteristics of biochar differ from those of hydrochar.

In fact, biochar used as adsorbent gains popularity among other carbonaceous materials. Biochar is considered to cost less than activated carbon while having superior performance even for gas-phase adsorption [29–31]. As it is common for all pyrogenic materials, synthesis parameters are those that have the key role of the overall adsorbent performance. Like activated carbon, biochar can be modified by various chemicals in order to reach ultimate quality and/or specific application requirements [32,33].



**FIGURE 6.2** SEM micrographs of (left) biochar and (right) hydrochar. *Reprinted with permission by Elsevier from J. Fang, L. Zhan, Y.S. Ok, B. Gao, Minireview of potential applications of hydrochar derived from hydrothermal carbonization of biomass, J. Ind. Eng. Chem. 57 (2018) 15–21. doi: <https://doi.org/10.1016/j.jiec.2017.08.026>.*

## 2. Carbon dioxide (CO<sub>2</sub>) capture

Carbon dioxide is a greenhouse gas that comes first regarding its production rate and total emission amounts worldwide. According to Environmental Protection Agency (EPA) and reports from other related sources, CO<sub>2</sub> holds 76% of the greenhouse gas emissions. The majority of CO<sub>2</sub> is emitted from the agricultural sector and industrial processes such as power production. Due to global warming and its ever increasing trend, there have been developed CO<sub>2</sub> capture and sequestration technologies [34], involving trapping, transportation, and its long-term storage. Adsorption is one of the most common methods for both trapping and storage of the emitted CO<sub>2</sub>. Although CO<sub>2</sub> storage refers to storage in geological formations, trapping of CO<sub>2</sub> is achieved by adsorption onto synthesized materials such as MOFs, modified zeolites, and various membranes. The particular step of CO<sub>2</sub> capture (trapping) can take place at different stages of CO<sub>2</sub> production/emission cycle; such options include (1) precombustion, (2) postcombustion, (3) oxy-fuel, and (4) CO<sub>2</sub> capture from the air stream prior to combustion process.

As was mentioned before, many materials have the potential to be an effective adsorbent with high CO<sub>2</sub> uptake. However, the implementation of

some of them has restrictions regarding performance under specific operation conditions [35], the vapor content of the stream [36,37] and the overall cost estimation [38]. Adsorption of CO<sub>2</sub> by carbon-based materials is the most promising approach especially in terms of cost-effectiveness and adsorption efficiency. Numerous studies have been conducted regarding carbon-based materials for the specific purpose, suggesting new synthesis processes [39,40] and materials or enhancing material properties by surface modification [41,42]. One of the most important cost reduction parameters of any kind of adsorbents is the abundance of their precursors. Carbonaceous materials can be derived from many sources including waste materials, resins, used tires [43], and many others, presenting good stability [44,45], fast kinetics [35], as well as reusability [46].

From the very early stage of industrialization, the most common adsorbent for industrial use was activated carbon. Until recently, activated carbon was preferred for processes such as wastewater treatment [47,48]. Although activated carbon has a poor adsorption performance of gaseous phase, modification of its surface in numerous ways is currently being studied. The interest to this approach comes from the fact that activated carbon has a low cost, and it can be manipulated easily in order to reach the required properties also in a relatively low cost [49].

Sea mango was the precursor of the synthesized activated carbon for CO<sub>2</sub> adsorption studied by Ali et al. [50]. The produced material was activated chemically by phosphoric acid (H<sub>3</sub>PO<sub>4</sub>), while a portion of the resulted material was treated further with various amines following Box–Behnken Design and the surface area was modified. During the synthesis, factors such as temperature for sea mango carbonization and H<sub>3</sub>PO<sub>4</sub> concentration of activation process were investigated as well. After optimization of the mentioned conditions, both cases (modified and virgin activated carbon) were tested for CO<sub>2</sub> uptake capacity. After amine impregnation, materials were evaluated regarding common adsorption parameters and their performance in a bed column. As it was expected, amine groups blocked some of the pores of the activated carbon, leaving functional groups proper not only for efficient CO<sub>2</sub> adsorption but also for regeneration ability. The activated carbon that had the best performance in the bed column was modified with AMP amine resulting in adsorption bed capacity of 23.05 mg CO<sub>2</sub>/g of the material.

Another emerging technology for efficient CO<sub>2</sub> uptake is the modification of activated carbon surface by developing nitrogen active sites [51]. Nitrogen-enriched carbonaceous materials present superior properties of adsorption capacity due to added functional groups that contribute to the polarization of CO<sub>2</sub> molecules [52]. In a study, conducted by Yami et al., of carbon material from coconut shell, nitrogen doping of the derived activated carbon provided remarkable textural and surface properties to the final material [46]. The material was activated chemically by KOH, and its performance was investigated for a fixed-bed column. As it is reported, adsorption properties were



enhanced due to surface modification reaching a CO<sub>2</sub> ultimate uptake of 4.23 mmol/g of the material under mild operation conditions. Parameters that affected the CO<sub>2</sub> adsorption capacity found to be the initial concentration of CO<sub>2</sub>, the temperature as well as the flow rate of the stream. Besides the good performance, the final material showed good stability after 20 adsorption–desorption cycles, indicating its reusability. A recent work of Manmuanpom et al. [53], investigated the CO<sub>2</sub> capacity of activated and nonactivated carbons. Specifically, both sample series were produced by carbonization of polybenzoxanine at a temperature of 800°C, while for activated samples a further physical activation under CO<sub>2</sub> at 900°C was followed. As it was revealed, activated carbon samples presented a higher specific surface area compared with that of nonactivated ones. In addition, XPS analysis showed N-sites onto the structure of the material. The adsorption capacity of the samples was investigated at different temperatures (from 30 to 50°C) and pressures (from 1 to 7 bars). The highest CO<sub>2</sub> uptake (3.59 mmol/g) was obtained for samples examined at a combination of 30°C and 7 bar, where the samples had the highest surface area and micropore volume. The overall good performance of the nitrogen-enriched carbons was attributed to both textural properties due to activation process and chemisorption mechanism induced by formed N-sites.

Coconut shell, in a study conducted by Yue et al., was used as precursor for porous carbon production by chemical activation [54]. In this work, coconut shell was urea-modified and activated by K<sub>2</sub>CO<sub>3</sub> after the carbonization process. Again, samples were examined regarding their CO<sub>2</sub> capacity at two different temperatures (0 and 25°C) keeping the pressure at 1 bar. Results of the experiment showed that the decrease in temperature leads to increase of CO<sub>2</sub> uptake, hence at 0°C 1 g of sample could adsorb up to 5.12 mmol compared with 3.71 mmol/g at 25°C. Interestingly, findings of simulation-based investigation of CO<sub>2</sub> adsorption capacity revealed that besides nitrogen content and microporosity of the adsorbent, the distribution of the micropores also plays an important role in material's CO<sub>2</sub> capture capacity at mild conditions.

Similarly, a detailed characterization of KOH- and NaOH-activated carbons [55] support the outcomes of the aforementioned study. Argan fruit shell, this time, was carbonized and then chemically activated with the mentioned acids. The conditions of CO<sub>2</sub> uptake measurements were 1 bar and 25°C as previously. Once more, the high N content along with large surface area of narrow micropores domain reached a CO<sub>2</sub> uptake of 5.63 mmol/g, a value close to the previously presented one at same conditions. The comparison of the two cases can lead to the conclusion that micropore structure and significantly high nitrogen content are indeed the two major factors of carbonaceous material CO<sub>2</sub> capture performance.

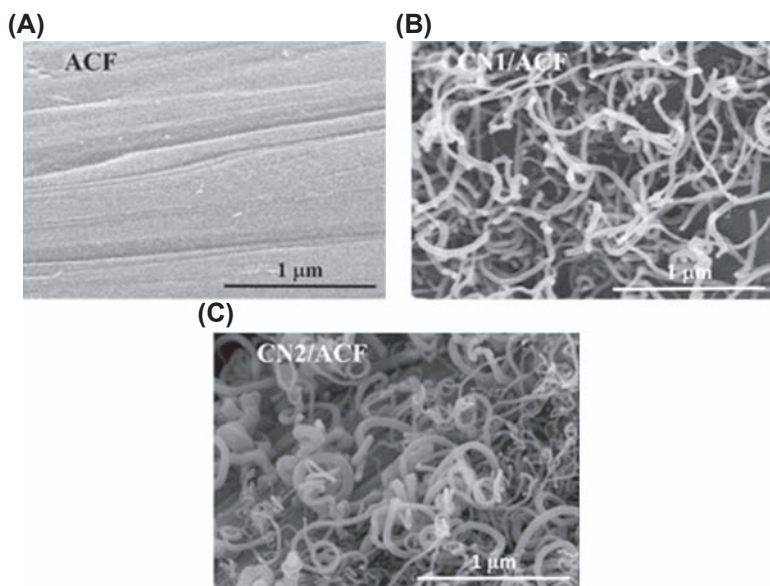
It is widely accepted that, except activation conditions and surface modification, the performance of a material is also attributed to its form. For

example, there are comparative studies upon powdered and activated fiber forms, suggesting that activated carbon fibers have advantage over the powder form for reasons of packing density [56]. Activated carbon fibers can be produced from any fabric waste or synthesized from various precursors; woven cotton [57] and denim [58] waste, poly(acrylonitrile) (PAN), and other synthetic polymers [59] can be the raw material for carbon fibers manufacturing.

Carrott et al. conducted a study investigating the effect of a preoxidation of industrial acrylic fibers on structural properties of the final activated carbon fibers [60]. In this work, high carbon yield during carbonization of acrylic fibers was achieved by prior oxidation of the precursor. This approach resulted in maximization of carbon content at the stage of carbonization and minimizing the reaction of the char with  $\text{CO}_2$  at activation stage. A BET surface area of  $2064 \text{ m}^2/\text{g}$  and total pore volume of  $1.15 \text{ cm}^3/\text{g}$  were achieved under a controlled range of activation temperature and time. The temperature range as well as the activation time influenced dramatically the microstructure of the material providing a wide range of porosities, from ultramicropores to pores of 4 nm in size. However,  $\text{N}_2$  adsorption and consequent  $\text{CO}_2$  adsorption analysis revealed that the samples with ultrafine micropores which are well developed and distributed at lower activation temperatures had the highest  $\text{CO}_2$  uptake ( $2.1 \text{ mmol/g}$ ) reaching even  $\text{CO}_2$  capacities obtained from chemically activated carbons measured at same conditions. Chemically activated industrial sisal fiber was used for investigating the effect of carbonization parameters on the surface characteristics of the final carbonaceous material [61]. Although this study was not conducted for examining  $\text{CO}_2$  capacity, it is interesting to notice that parameters such as  $\text{N}_2$  flow rate and carbonization temperature and time have the opposite effect; higher temperature and longer hold time develop higher BET surface area and pore volume, whereas higher  $\text{N}_2$  flow rate has negative impact on the aforementioned properties.

Enhancement of activated carbon fibers properties over  $\text{CO}_2$  capacity was done by nitrogen-enriched carbon nanotube (CNT) growth onto PAN-derived fibers. This attempt conducted by Chiang and his coworkers [52] resulted in spaghetti-like and randomly oriented CNT grafted to activated carbon fibers. For comparison reasons, a portion of used fibers were immersed to a cobalt acetate solution prior CVD process for CNT growth. Fig. 6.3 shows the results of each treatment.

The above figure, Fig. 6.3, presents clearly the effect of preimmersion of activated carbon fiber on the resulted CNTs sizes. According to authors, images b and c reveals the remaining particles of the used catalyst, a fact that indicates the low reaction of the catalyst with the surface of the fibers while the open pore end is attributed to catalyst particle falling-off during CVD process.



**FIGURE 6.3** FESEM micrographs of (A) activated carbon fibers as-received, (B) carbon nanotube (CNT)-grafted activated carbon fibers, and (C) immersed to cobalt/acetate solution CNT-grafted activated carbon fibers. *Reprinted with permission by Elsevier from Y.-C. Chiang, R.-S. Juang, Surface modifications of carbonaceous materials for carbon dioxide adsorption: a review, J. Taiwan Inst. Chem. Eng. 71 (2017) 214–234.*

Spherical form of activated carbon has shown superior properties over various applications: high mechanical and thermal stability, better packing, surface smoothness, and pore manipulation are some of them [62].

Nitrogen- and sulfur-enriched spherical activated carbon were prepared by Sun et al. as  $\text{CO}_2$  adsorbents [62]. Spheres of some millimeters in size were synthesized by a three-step process via final chemical activation. Resin of poly (styrene-vinylimidazole-divinylbenzene) was first  $\text{H}_2\text{SO}_4$ -sulfonated, carbonized, and finally activated with KOH. The styrene and nitric components of resin at different ratios were the sources for carbon and nitrogen, while  $\text{H}_2\text{SO}_4$  and KOH were responsible for simultaneous cross-linking and sulfur-enriching the surface thermally. The as-prepared sample with the higher styrene/N-vinylimidazole ratio (1:0.75) had the best performance at different operation conditions for  $\text{CO}_2$  adsorption affected by the concentration of N-sites. Remarkably, obtained comparative results at different operation pressure revealed that at lower pressure the efficiency of adsorption was due to surface chemistry, while at increased pressure the mechanism was better attributed to microporosity. Finally, authors concluded that the suggested adsorbent has good potentials for  $\text{CO}_2$  capture due to enhanced properties of regeneration stability and fast kinetics at various operation conditions (Table 6.1).

**TABLE 6.1** The most recent studies of adsorbents for CO<sub>2</sub> capture.

Adsorbent	Precursor	Carbonization	Activation	Uptake	References
Porous carbon xerogels	Resorcinol-formaldehyde-phloroglucinol gels	900°C	—	2.89 mmol/g	[63]
Three-dimensional porous carbon frameworks	Mangosteen peel	700°C	KOH	6.93 and 4.77 mmol/g (0 and 25 °C)	[64]
Heteroatom-doped hierarchically porous carbons	Glucose hydrogels	600–1000°C	KOH	4.4 mmol/g	[65]
N-doped porous carbon	Imidazole-based hyper-cross-linked polymers (hcps)	—	KOH	180–258 mg/g (273 K, 1.0 bar) 4173 mg	[66]
Activated carbon	Date seeds	600–900°C	Physically activated	141.14 mg	[67]
Activated carbon	Wood sawdust	400°C	H <sub>3</sub> PO <sub>4</sub> , NaOH, and KOH	0.96 mol/kg	[68]
Porous carbons	EFB biomass	600 or 800 C	KOH	5.184, 3.712, and 2.343 mmol/g	[68]
N-doped nanoporous carbon	Walnut shell	600°C	KOH	7.42 mmol/g 14.03 mmol/g (1 and 10 bar, respectively)	[69]
N-doped active carbon	Chitosan	550 C	KOH	1.86 mmol/g	[70]
Nanoporous carbons	Cigarette butts	600 C	KOH	6.0 mmol/g	[71]

### 3. Acidifying pollutants (NO<sub>x</sub>, SO<sub>x</sub>)

Nitrous oxides, sulfur oxides, and their derivatives are indirect greenhouse gases. Although NO<sub>x</sub> comprises only 6% of the total greenhouse gases emissions, they are responsible for phenomena such as acid rain and smog [9,72]. In addition, photo chemical reaction of NO<sub>x</sub> in the atmosphere leads to the production of tropospheric ozone [12]. The main sources of all acidifying pollutant emissions are internal combustion engines (road transport) with 13% [11], energy production 28%, while the agriculture sector have the 27% of the total share. Emissions of NO<sub>x</sub> and SO<sub>x</sub> from on-road sources have been reduced significantly by the implementation of clearer fuels and more efficient catalysts [10,15]. However, there is still space for emerging technologies or improvement of existing ones for NO<sub>x</sub> and Sox reduction by adsorption in other sectors. Nitrogen oxide emissions, and especially that of N<sub>2</sub>O resulting from agricultural activities, are suppressed by soil enrichment with various substances known as amendment [73]. In general, soil amendment is a substance added to soil in order to improve textural properties [74], fertilization of soil [75], etc.

Biochar is the most common soil amendment for N<sub>2</sub>O reduction and soil chemical stabilization [76]. Biochar is produced by pyrolysis of biomass at total or partial absence of oxygen [27]. Biochars present excellent physical and chemical properties such as high surface area, well developed porosity, high sorption capacities, etc. Surface structure properties are influenced massively by conditions of the synthesis process; Fig. 6.2 shows the effect of carbonization temperature on porosity development of wood biomass after slow pyrolysis [77].

Many studies have been conducted for examining the adsorption capacity of produced biochars for the specific purpose. Although biochar is extensively used in soil with great benefits, regarding its role as a good adsorbent of N<sub>2</sub>O very few works have been presented. For determination of the importance of biochar adsorption capacity, adsorption measurements of N<sub>2</sub>O onto wood-derived biochar were conducted by Xiao et al. [78]. At this work, wood biomass-based samples were prepared by anaerobic pyrolysis at a temperature range from 300 to 700°C. After carbonization, samples were further heated in the presence of air at 400°C. Adsorption measurements of CO<sub>2</sub> were conducted, and surface area was determined correlating well with Langmuir capacity. Furthermore, simulated pressure conditions in soil were used in order to investigate if adsorption-desorption properties of chars are influenced by char coating with soil matter. The results revealed that coating does not affect sorption characteristics of the biochar which take place in the developed micropores; therefore a biochar concentration of 1% in the soil could have great potentials to retain N<sub>2</sub>O.

Nitrogen oxide and sulfur oxide adsorption by activated carbon have been reviewed with focus on surface properties and consequently required char

treatment processes to reach the desired adsorption capacities [79–81]. One example of surface chemistry importance is provided by Ghouma et al. [82]. This study investigates three samples of activated carbons derived from agro-industrial wastes and prepared by both chemical and physical activation. Nitrogen oxide ( $\text{N}_2\text{O}$ ) capacity onto the as-prepared samples was investigated at ambient temperature in a fix-bed column with low  $\text{N}_2\text{O}$  flow rate. The findings of this study revealed that chemically activated carbon has greater  $\text{N}_2\text{O}$  uptake at those conditions than the physically activated one. This result indicates that functional groups present on the surface of the material have a key role in  $\text{N}_2\text{O}$  adsorption mechanism. Furthermore, authors suggest that pore structure has also a crucial role regarding  $\text{N}_2\text{O}$  diffusion rates. Therefore, as activated carbon prepared by  $\text{H}_3\text{PO}_4$  activation presents a mesoporous surface structure,  $\text{N}_2\text{O}$  is diffused more efficiently compared with  $\text{CO}_2$ -activated samples.

Adsorption of  $\text{NO}_x$  and  $\text{SO}_x$ , from flue gas at the same time, onto activated carbon monoliths supporting metal oxide group ( $\text{CO}_3\text{O}_4$ ) was investigated [83]. The scope of Silas and his coworkers' study was to investigate the contribution of the deposition precipitation, pore volume impregnation, and hydrothermal methods to the efficiency of as-synthesized materials. The outcome of this research was that hydrothermally prepared samples showed higher adsorption capacities of the two target gases than those prepared differently; with an uptake of 123.1 and 130.2 mg/g of  $\text{SO}_2$  and  $\text{NO}_x$ , respectively. According to the obtained results of breakthrough curves (Fig. 6.4), material performs higher efficiency for  $\text{NO}_x$  adsorption attributed to its physicochemical properties as well as experimental conditions (Fig. 6.5).

Additionally to the previous analysis, reusability of adsorbents was evaluated after two cycles of adsorption/regeneration. The reduction of adsorption capacity both for  $\text{NO}_x$  and  $\text{SO}_x$  was minor, while the regeneration efficiency was kept above 94% for both samples after the total two cycles.

#### 4. Methane separation

Although  $\text{CO}_2$  is considered the main culprit gas for global warming,  $\text{CH}_4$  is also a potent greenhouse gas. According to EPA,  $\text{CH}_4$  has a 14% of greenhouse gas emissions with the majority derived from agricultural and energy sectors. Separation technologies are usually studied in respect to  $\text{CH}_4$  separation from a mixed gas stream in natural gas refining processes. The most common technologies applied for this purpose are pressure swing adsorption, temperature swing adsorption, vacuum swing adsorption, or the vacuum pressure swing adsorption [84]. Various adsorbents are used in the referred methods including MOFs [85], zeolites [86], silica-based, and others. Carbonaceous materials are often utilized in the aforementioned processes for efficient gas mixture separation [85,87,88].

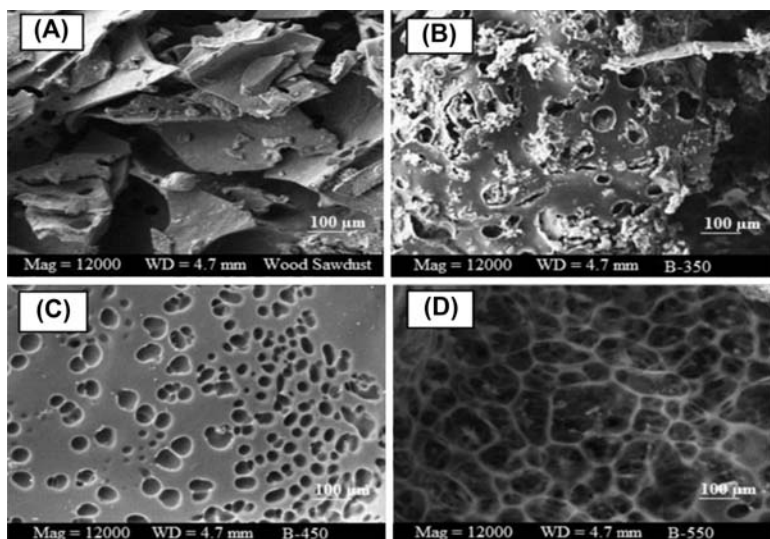


FIGURE 6.4 SEM micrographs of (A) wood biomass and (B) carbonization at 350°C, (C), at 450°C, and (D) at 550°C. Reprinted with permission by Elsevier from Z.Z. Chowdhury, M.Z. Karim, M.A. Ashraf, K. Khalid, Influence of carbonization temperature on physicochemical properties of biochar derived from slow pyrolysis of durian wood (*Durio zibethinus*) Sawdust, *Bio Resour.* 11 (2016).

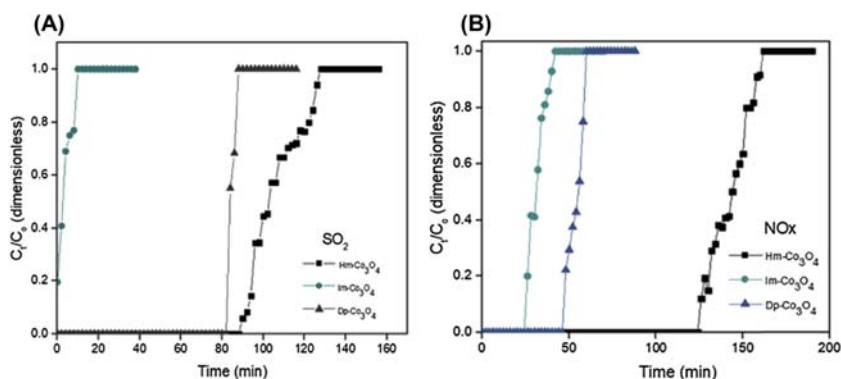


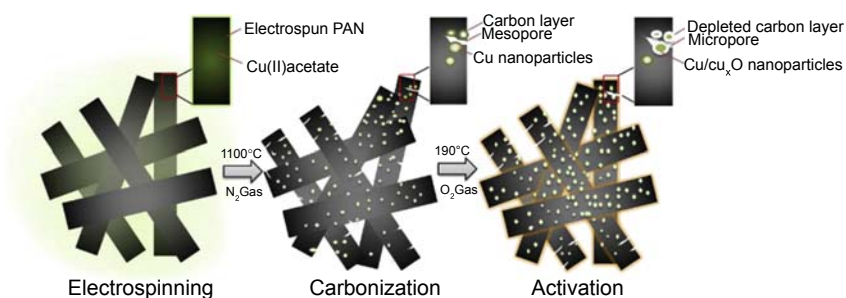
FIGURE 6.5 Breakthrough curves of simultaneous (A) NO<sub>x</sub> and (B) SO<sub>x</sub> adsorption onto hydrothermally derived adsorbent. Reprinted with permission by Elsevier from K. Silas, W.A.W. A. K. Ghani, T. S.Y. Choong, U. Rashid, Breakthrough studies of Co<sub>3</sub>O<sub>4</sub> supported activated carbon monolith for simultaneous SO<sub>2</sub>/NO<sub>x</sub> removal from flue gas, *Fuel Process. Technol.* 180 (2018) 155–165. doi: <https://doi.org/10.1016/j.fuproc.2018.08.018>.



## 4.1 CH<sub>4</sub>/CO<sub>2</sub>

The selection of the adsorbent in use is a matter of the (1) gas nature, (2) composition of the gas mixture, (3) operation conditions, and (4) thermodynamics of the system (kinetics, equilibrium, time, etc.). Four types of activated carbon were compared with zeolites regarding CO<sub>2</sub> removal from different gas mixtures. Experiments were conducted in order to evaluate adsorption performance of both types and their efficiency under a range of operation conditions. The degradation of material was examined as well. Activated carbon examined showed better characteristics over zeolites especially for reusability.

Gas binary mixtures of CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub> selective adsorption on carbon materials derived from starch sugar were studied by Wang et al. Four glucose-based carbons were synthesized differentiating in KOH/C ratio (2:1, 3:1, 4:1, and 5:1). Isothermic heat of adsorption for each gas was obtained by several techniques. Nitrogen adsorption revealed that the optimal from the as-prepared material was that with KOH/C ratio of 4:1; high surface area, and high CO<sub>2</sub> uptake reaching capacities even of best MOFs (3153 m<sup>2</sup>/g and 22.4 mmol/g, respectively). In addition, the KOH concentration seems to play a highly important role on surface chemistry of the resulted materials. Given the example of the presented study, at KOH/C from 2 to 5, pore structure was developing accordingly as it is depicted in Fig. 6.6. The selectivity of the adsorbents was estimated by ideal absorbed solution theory, predicting high selectivity rates at 30 operation pressure of binary gas mixtures. The outcome of this study promotes the implementation of this kind of materials as efficient adsorbents for CH<sub>4</sub> separation from gas mixtures.



**FIGURE 6.6** Schematic representation of Cu-/Cu<sub>2</sub>O-modified activated carbon fiber composites synthesis process. PAN, poly(acrylonitrile). Reprinted with permission by Elsevier from B. Bajaj, H.-I. Joh, S. M. Jo, J. H. Park, K. B. Yi, S. Lee, *Enhanced reactive H<sub>2</sub>S adsorption using carbon nanofibers supported with Cu/Cu<sub>2</sub>O nanoparticles*, *Appl. Surf. Sci.* 429 (2018) 253–257. doi: <https://doi.org/10.1016/j.apsusc.2017.06.280>.



## 4.2 CH<sub>4</sub>/H<sub>2</sub>S

Hydrogen sulfide (H<sub>2</sub>S) causes major health problems when one is exposed even to relevantly low concentration. Eye and skin irritation, breathing difficulties, and loss of odor are some of the symptoms after the exposure. According to Occupational Safety and Health Administration (OSHA), H<sub>2</sub>S causes asphyxiation and has serious effect on human nervous system, that is why H<sub>2</sub>S removal/adsorption technologies are in the prime line of investigation. Furthermore, H<sub>2</sub>S concentration in a natural gas stream may cause significant damage to piping network and natural gas treating units [89].

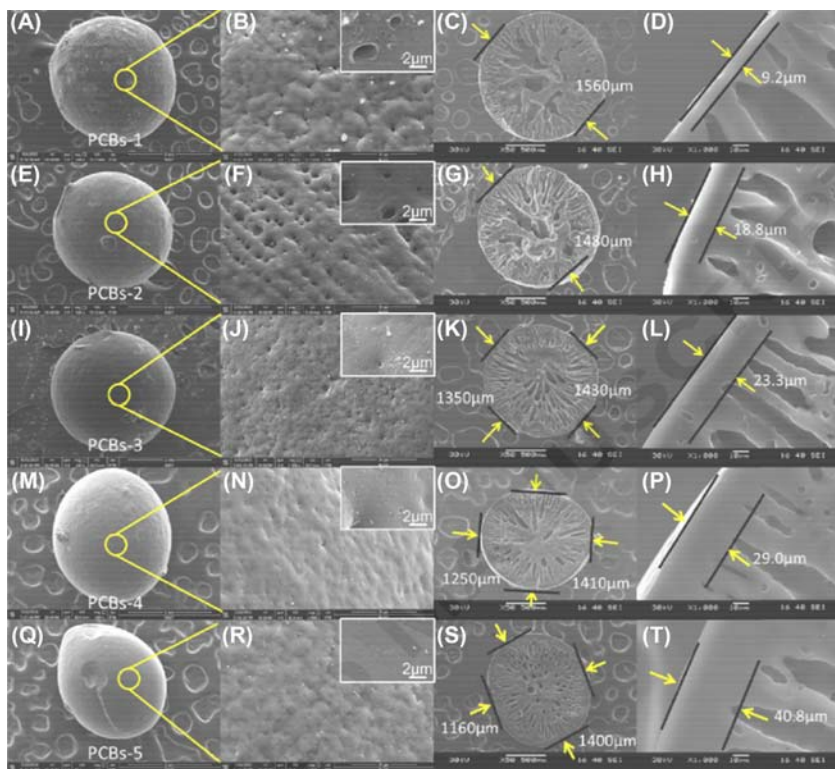
Selectivity along with adsorption capacity of an adsorbent is paramount for optimal efficacy of any chemical process. Regarding H<sub>2</sub>S, the most common applied materials for adsorption methods are zeolites [84], MOFs [90], and various carbon materials. Because H<sub>2</sub>S has low affinity to carbon, modification of the derived carbon material is promoted by researchers of the field [91].

In the previous section of this chapter, it was mentioned that carbonaceous materials in fiber gain an increasing interest due to its advantages. Also, it was mentioned that further modification of adsorbents surface is preferred in most of the cases. As an example for these two, utilized by a recently developed methodology of electrospinning, the work of Bajaj et al. is discussed [92]. This team used activated carbon fibers and modified the surface with nanoparticles of Cu/Cu<sub>2</sub>O. As source material, electrospun PAN/copper fibers were used. After carbonization process, the produced composite was activated at mild conditions. Schematic representation of the synthesis steps is clear and comprehensive as it is seen in Fig. 6.7.

The as-synthesized material was a result of electrospinning PAN stabilized under air at 280°C. The following steps include carbonization in nitrogen-rich environment at elevated temperature (1100°C) and activation at 190°C in O<sub>2</sub> as the final step. Surface elemental characterization was conducted by XRD and XPS, while SEM and TEM were used for structural properties. Characterization analysis indicates the presence of Cu/Cu<sub>2</sub>O onto the carbon fibers. Measurements of H<sub>2</sub>S uptake were conducted at 300°C in a fixed column bed. Pure activated carbon fibers were subjected to the breakthrough time measurements as well. The amount of nanocomposite used was half of the amount of pure fibers. The experiment stopped when H<sub>2</sub>S concentration reached 50ppm (from 761ppm). Finally, the presence of Cu enhanced the adsorption capacity by many times compared with pure activated carbon fibers, suggesting their potential for H<sub>2</sub>S adsorption applications.

## 5. Volatile organic compounds

VOCs is an umbrella term for any compounds, according to the European Union, that has an initial boiling point below 250°C. Emissions of VOCs, besides industrial processes, are produced from many every day activities.



**FIGURE 6.7** SEM micrographs of PCBs, from top to bottom; PCB-1 to PCB-5. The presentation of each sample includes surface and cross sections at different magnifications (from left-hand to right-hand side). Reprinted with permission by Elsevier from J. Qi, J. Li, Y. Li, X. Fang, X. Sun, J. Shen, L. Wang, *Synthesis of porous carbon beads with controllable pore structure for volatile organic compounds removal*, *Chem. Eng. J.* 307 (2017) 989–998. doi: <https://doi.org/10.1016/j.cej.2016.09.022>.

Compounds of this category cause harm not only to plants and animals but to human in the long term [93]. Moreover, VOCs contribute to climate change significantly through ozone formation [94] and smog [72] as NO<sub>x</sub> do. Technologies for VOCs limitation and management can be divided in two categories: destruction and recycling. Although the efficiency of the former is at a satisfying level, it requires massive energy consumption. The latter option includes absorption, adsorption, separation, and condensation. Photocatalysis is one of the most studied cases concerning air purification especially for indoor application, while adsorption onto materials for large-scale units is among the emerging technologies [95,96].

In many cases regarding industrial applications, spherical shaped adsorbents are preferred. Superior mechanical and surface properties of

carbonaceous spheres have attracted recently the interest of scientific community. In an experimental work of Qi and coworkers, beads of porous surface structure were synthesized by phase inversion method [97]. After phase inversion, the as-produced polymeric samples were carbonized. Beads were derived from four different polymers, namely PES, PSF, PAN, and PVDF. For achieving the desired shape, the resulted solution was introduced via a syringe to a coagulation bath with deionized water and iso-propanol mixture at different ratios (from pure water to 40:60, with a composition change step of 10). The different water/iso-propanol ratio contributed both to the size and to the microporosity of the beads. After this, oven drying was accomplished as an intermediate step prior to carbonization at 350°C and further at 950°C at nitrogen-rich environment. The as-prepared beads were designated regarding the order of ratio increase, so we have PCB (porous carbon beads)-1, PCB-2, PCB-3, PCB-4, and PCB-5 (where 1 is for 0:100 and so on). As it is observed in Fig. 6.6, the best structure was developed on samples of 10:90 (PCB-2) water/iso-propanol.

The optimized coagulation bath ratio provides materials with high surface area of 1166 m<sup>2</sup>/g as well as good adsorption capacity of benzene (1.467 g/g), toluene (1.229 g/g), n-hexane (0.600 g/g), and acetone (0.770 g/g). The almost unchanged performance of the material after six cycles of sorption process promotes its application for VOCs treatment.

Regeneration efficiency of an adsorbent plays a crucial role in the overall performance estimation. Especially for industrial applications, high thermal and mechanical stability are the main criteria. To this direction, studies of desorption method applied for regenerating a specific adsorbent, after having adsorbed a specific compound, are very important for process optimization [86,98]. Among other properties, desorption of benzene and toluene from reduced graphene oxide was investigated [99]. In specific, graphene oxide and its reduced form was synthesized from graphite following a slightly altered Hummer's method. Although the interlayer space of the sheetlike structure of the graphene oxide was found to be higher, reduced graphene oxide possesses a higher surface area of 292.6 m<sup>2</sup>/g over 236.4 m<sup>2</sup>/g of the former. Experiments conducted for benzene and toluene uptake capacities of the reduced graphene oxide showed values of 276.4 and 304.4 mg/g, respectively. Finally, the regeneration efficiency of the adsorbent was examined, and it was determined that reduced graphene oxide can be fully regenerated at low temperatures (150°C).

In general, separation techniques require more strong chemical bonds between the adsorbate and the adsorbent. Surface modification and/or chemical activation are the most common approach to improve original material's chemical properties. Table 6.2 summarizes carbon-based materials for (1) CH<sub>4</sub> separation from gas mixtures, (2) H<sub>2</sub>S rementation and VOCs reduction by adsorption.

**TABLE 6.2** Summary of carbon-based materials for various greenhouse gases (with exception of CO<sub>2</sub>).

Adsorbent	Precursor	Carbonization	Activation	Uptake	Application	Ref.
N-doped activated carbon	Chlorella, isochrysis, and <i>Platymonas subcordiformis</i>	500°C	—	7.27 mmol/g	CO <sub>2</sub> /CH <sub>4</sub>	[100]
Activated carbon	Lignin	800°C	KOH	600 mg/g	VOCs	[96]
Activated carbon fiber	Mesophase pitch fiber, acf textile kynol acf-15 (nippon kynol)	800–1000°C	CO <sub>2</sub> /H <sub>2</sub> O	Toluene 59.6 g/100 g Ethanol/methanol 54.4/49.2 g/100 g	VOCs	[101]
Monolithic tubular activated carbon	Cotton stalks	Ac-p 400°C Ac-z 600°C	Phosphorous acid and zinc chloride	328 mg/g wet 258 mg/g dry 221 mg/g wet	VOCs	[102]
Activated carbon	Corn cob wastes	350, 400, 450, 550, and 650°C	ZnCl <sub>2</sub>	414.6 mg/g 3000 mg/m (25 °C)	VOCs	[103]
Activated carbon	Gingko leaf and peanut shell	800°C	N <sub>2</sub>	CO <sub>2</sub> , CH <sub>4</sub> , and N <sub>2</sub> 2.13/0.64/0.2 mmol/g, 3.63/1.38/0.45 mmol/g, 2.26/0.89/ 0.31 mmol/g, 1.62/ 0.45/0.17 mmol/g	CO <sub>2</sub> , CH <sub>4</sub> , N <sub>2</sub>	[88]
N-doped carbon	Crab shell	600–900°C	N <sub>2</sub> /CO <sub>2</sub>	6.8 mmol/g 2.49 mmol/g	CH <sub>4</sub> /N <sub>2</sub> , CO <sub>2</sub> /N <sub>2</sub>	[100]
Activated carbon	Cherry stones	—	CO <sub>2</sub>	—	CO <sub>2</sub> /CH <sub>4</sub>	[87]
Activated carbon cloth	Viscose rayon cloth	630°C	CO <sub>2</sub>	2.68 mmol/g or 10.55 wt% 0.99 mmol/ g or 1.56 wt%	CO <sub>2</sub> /CH <sub>4</sub>	[104]
Activated carbon	Coconut shell	1173K	CO <sub>2</sub>	6.0 mmol/g 4 mmol/g	CO <sub>2</sub> /CH <sub>4</sub>	[105]

## 6. Conclusions

This chapter reviews all the recent literature on carbonaceous materials for gas-phase adsorption. The increased concern on climate change and its irreversible impact on the planet induced the scientific research toward environmental protection technologies. Aspects that accord with eco-friendly processes utilization involve the implementation of technologies for gas purification and clean energy consumption, toxic and hazardous gas reduction, greenhouse effect mitigation, and so on. Up to the very recent history, adsorption was linked with decontamination technologies of liquid phase. Nowadays, adsorption seems to have a key role in the mentioned emerging technologies with high level of efficiency for gas-phase applications. However, the selection of the proper adsorbent is a multicriteria decision: cost efficiency, physicochemical properties, and mechanical properties are some of the most important. In the perspective of all mentioned parameters, and especially cost, carbon-based materials have gained popularity. Carbonaceous materials can be derived from a wide range of precursors resulting with high surface and good stability properties. The performance of each adsorbent depends mainly on factors such as the source, the synthesis process, and surface chemistry. The latter is easily adjusted by surface modification techniques. The present work encloses studies of gas-phase adsorbents with potentials for large-scale applications. In addition, to best of our knowledge, this is one of the few works that reviews the performance and properties of carbon-based material for several different greenhouse gases. At this point, it should be noticed that the number of published studies upon CO<sub>2</sub> is disproportionate regarding the existing literature for other harmful gases, and further research is suggested toward this direction.

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# Advanced Low-Cost Separation Techniques in Interface Science

Edited by George Z. Kyzas and Athanasios C. Mitropoulos

Concise reference providing expertise knowledge in how to use separation techniques in interface science at minimal cost and energy usage

## Key features

- Focuses on cost and energy saving separation techniques in interface science
- Discusses multiple techniques including flotation, adsorption, materials synthesis, etc.
- Combines in a single source both separation techniques, advanced methodologies, and low-cost potential of the techniques
- Describes techniques that are attractive for both research and industrial purposes

*Advanced Low-Cost Separation Techniques in Interface Science* helps scientists and researchers in academia and industry to gain expert knowledge in how to use separation techniques at both minimal cost and energy usage. It handles a broad range of highly relevant topics including modern flotation techniques, low-cost materials in liquid- and gas-phase adsorption, new trends in molecular imprinting, graphenes in separation, nanobubbles and biopolymers in interface science, green techniques for wastewaters, and concludes with modelling in environmental interfaces. It shows that these techniques are both attractive for research purposes and for industrial purposes.

*Advanced Low-Cost Separation Techniques in Interface Science* is intended for chemical engineers working in wastewater treatment industries, membrane industries, pharmaceutical industries, textile or tanneries industries, hybrid-topic industries, and energy industries.

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