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Activated Carbon Monoliths by Pressureless Technique for Environmental Applications

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Published online 19 May 2015 in Wiley Online Library (wileyonlinelibrary.com). DOI 10.1002/ep.12143

Carbon monoliths have been prepared through simple procedure by carbonization in inert atmosphere at 550°C from commercial activated carbon and from unburned carbon in waste bagasse fly ash, using two different kinds of phenolic resin binders. The bagasse fly ash was collected from sugar mills where bagasse is used as a biomass based fuel for cogeneration. Commercial activated carbon based monoliths where 5 and 10 wt % of the activated carbon had been replaced by iron oxide (hematite) were also prepared. Results indicate that BET surface area decreases upon carbonization and loading of hematite. Scanning electron microscopy studies shows that the iron oxide is well distributed over the monoliths and X-ray diffraction shows that it is reduced to magnetite during carbonization. Temperature programmed reduction experiments show that the iron oxides on the monoliths are redox active. The monoliths based on commercial activated carbon showed 80% removal of phenol in diluted phenol based water solutions whereas unburned carbon derived monoliths showed 51% removal in similar solution. © 2015 American Institute of Chemical Engineers

Keywords: adsorption, carbon monolith, electron microscopy, powder diffraction, thermogravimetric analysis

INTRODUCTION

Activated carbon has many advantages because of its tunable textural properties, thermal stability, and the ability to modify the surface properties. For many applications, activated carbon has been used in the form of monoliths [1–4]. Monoliths have good strength, greater bulk density, lower attrition, lower pressure drop, surface flexibility, and recovery after use. They have been used for gas storage, separation, catalysis, and energy storage. Activated carbon is obtained from many sources such as coal, biomass, and agro-residues. Among these is unburned carbon in bagasse fly ash generated as a waste in sugar mills using bagasse as a fuel for cogeneration.

Monoliths are typically prepared with binders using extrusion or hot pressing at high temperatures and pressures. In some instances, they are also prepared by binderless process wherein the tar produced during chemical treatment of the carbon acts as the binder [5]. However, there are still reports of requirement for “new, easier and cheaper routes” [6] thus making it economically and technically feasible to explore the uses of alternate processes and sources of carbon such as unburned carbon in fly ash. Monoliths can also be modified with other additives, to meet specific application requirements [7].

Iron/iron oxide carbon composites have been prepared in many studies to take advantage of magnetic or catalytic properties of iron [8,9]. They are typically prepared by wet chemical method using iron salts. Takahama in a patent describes a dry method for incorporating metal in carbon by mixing metal oxide powder with a carbon precursor followed by carbonization where the metal oxide is reduced to metal with fine particulate size [10].

In this study, a simple slurry casting method followed by heat treatment in a closed vessel is used for preparing carbon monoliths using commercial activated carbon and phenolic resin binders. In some samples, iron oxide was incorporated to check its possible reduction during carbonization. Iron oxide was incorporated in dry form into a mixture of carbon and precursor binder. The formation of reduced iron oxide or iron could also impart magnetic properties, which could be required for certain applications. In addition to commercial activated carbon, unburned carbons (from bagasse fly ash) in the separated unactivated form and activated form have been used as the starting materials for monoliths. Bagasse fly ash, generated in sugar mills using bagasse as fuel in their boilers, contains high amount of unburned carbon, which can be easily separated [11]. It is of large interest to find uses for this waste material, which otherwise create disposal problems. The main purpose of this study is to explore a relatively simple method for monolith preparation and to examine the utility of waste derived powdered carbon for monolith preparation by comparing their properties with commercial activated carbon derived monoliths. A brief comparison of their adsorption capacity towards phenol, a liquid phase pollutant has also been done to ascertain their applicability for such environmental applications. Phenolic wastewater is highly toxic and harmful even at very low concentrations and adsorption has been considered as one of the best technology to remove phenol from wastewater [12]. Adsorbents such as activated carbons from different precursors and a few monoliths have been used to remove phenol [3,9,12]. Compared to powdered activated carbon, monoliths are a better alternative due to their compact structure, almost negligible pressure drop and ease of
The dried monoliths were carbonized at 550°C and was dried for 1–2 days and was taken out from the mold at room temperature. The mixture was then poured into a mold with 50 wt % binder and 2 as 40 wt % binder. The samples indicate the different binder percentage referring 1 as 1% of iron is indicated (F5 and F10). 1 and 2 in CCP denotes the type of starting carbon (CC, SC, and AC) followed by the ratio of the components used for the synthesis of carbon monoliths. In the naming of sample codes, the first two letters show the type of starting carbon (SC and AC) showed best results (in terms of BET surface area) with 40 wt % binder. Therefore, SC and AC based monoliths (SC and AC) showed best results (in terms of BET surface area) with 40 wt % binder. Therefore, SC and AC based monoliths were prepared with 40 wt % binder. In the preparation of CC based monoliths, both 50 and 40 wt % of binder amount was used whereas waste derived monoliths (SC and AC) followed by the single letter for binder type (P and S) and lastly the percentage of iron is indicated (F5 and F10). 1 and 2 in CCP samples indicate the different binder percentage referring 1 as 50 wt % binder and 2 as 40 wt % binder. The carbon monoliths were prepared by mixing the components in ethanol followed by vigorous stirring for 30 min at room temperature. The mixture was then poured into a mold and was dried for 1–2 days and was taken out from the mold. The dried monoliths were carbonized at 550°C for 4 h in the absence of air. Table 1 shows the mixing ratio of the components used for the synthesis of carbon monoliths. In the naming of sample codes, the first two letters denote the type of starting carbon (CC, SC, and AC) followed by the phenol to formaldehyde ratio which is <1 for the former and >1 for the latter giving a lower degree of crosslinking for the novolacs. Nine different carbon monoliths were prepared using these three components in different ratios. The SC was obtained through floatation technique and activated SC was prepared by heating a mixture of SC and water (1:7 ratio) at 800°C for 3 h in the absence of air. The surface area measurement was done using nitrogen adsorption method (Gemini 2375RT). Before analysis, the samples were degassed at 350°C for 12 h in vacuum and BET method was used to determine the surface area of the activated carbon monoliths. The porosity and pore size distribution of the monoliths was studied using mercury porosimetry (Micromeritics 9320 pore sizer).

The morphology of the samples was examined using a scanning electron microscope (JEOL JSM7000F) equipped with an Oxford INCA SIGHT energy dispersive detector (EDS). The samples were coated with carbon before analysis. EDS analysis was also performed to confirm the distribution of iron oxide particles in the carbon monoliths. Particle size distribution of the commercial activated carbon and iron oxide powders were analysed using laser light diffraction technique (Sympatec, Helos-BF).

X-ray diffraction was done using an XPert MPD diffractometer with monochromatic CuKα radiation in the 2θ range of 10° to 80° with a 0.02° step size and 1 h hold time. The temperature programmed reduction (TPR) experiments were conducted in a flow of 5% H2 in Argon to check the redox behaviour of iron oxide containing monoliths. The samples were pretreated at 250°C in pure He gas for almost 1 h. Approximately 100 mg of sample was loaded and was reduced by raising the temperature from RT to 800°C at the rate of 10°C/min.

Elemental (CHN) analysis was done on carbonized monoliths using a Perkin Elmer PE2400 elemental analyzer.

Phenol Adsorption

Hundred milliliter solution of required phenol concentration (mg/L) was taken in a conical flask and the required adsorbent dose (g/L) was added. The mixture was shaken for 1 h in a shaker (Shaker Orbitek) at 160 rpm and 25°C. After shaking, the solution was centrifuged for 10 min at 7000 rpm and filtered in a filter assembly using 0.45 μm filter. For phenol analysis, the supernatant was diluted with water to make the phenol concentration 5 mg/L. 0.2 mL 0.5 M NH4OH and 0.1 mL 1 M phosphate buffer were added to adjust the pH at 7.9. Subsequently, 0.1 mL 4-aminoantipyrine (0.1M) and 0.1 mL of potassium ferricyanide (0.25M) were added. The absorbance was measured in an UV spectrophotometer (Aquamate, Thermo Scientific) at 500 nm after 15 min.

RESULTS AND DISCUSSIONS

Thermal Analysis

The thermal analysis of the samples prepared from CC is shown in Figure 1. In case of P used as binder (CCP1), the weight loss due to removal of volatiles takes place in two steps: around 120–320°C and from around 450–650°C. The remains are assumed to be carbon and inorganic ashes in the CC. It is observed that with addition of 5 and 10% iron oxide (CCPF5, CCPF10) the carbonization and weight loss starts already at ~50°C, the remaining part being very similar. In case of S resin in the CCs samples, the major weight loss is shifted to slightly higher temperatures, between 500 and 750°C. However, with 5% iron oxide addition (CCS5), it also shows weight loss in two steps although the first step is

<table>
<thead>
<tr>
<th>Sample code</th>
<th>Percentage of starting carbon</th>
<th>Percentage of binder</th>
<th>Percentage of iron oxide</th>
</tr>
</thead>
<tbody>
<tr>
<td>CCP1</td>
<td>50 wt % of CC</td>
<td>50 wt % of PR</td>
<td>0 wt %</td>
</tr>
<tr>
<td>CCPF5</td>
<td>45 wt % of CC</td>
<td>50 wt % of PR</td>
<td>5 wt %</td>
</tr>
<tr>
<td>CCPF10</td>
<td>40 wt % of CC</td>
<td>50 wt % of PR</td>
<td>10 wt %</td>
</tr>
<tr>
<td>CCS</td>
<td>50 wt % of CC</td>
<td>50 wt % of S</td>
<td>0 wt %</td>
</tr>
<tr>
<td>CCSF5</td>
<td>45 wt % of CC</td>
<td>50 wt % of S</td>
<td>5 wt %</td>
</tr>
<tr>
<td>CCSF10</td>
<td>40 wt % of CC</td>
<td>50 wt % of S</td>
<td>10 wt %</td>
</tr>
<tr>
<td>CCF2</td>
<td>60 wt % of CC</td>
<td>40 wt % of PR</td>
<td>0 wt %</td>
</tr>
<tr>
<td>SCP</td>
<td>60 wt % of SC</td>
<td>40 wt % of PR</td>
<td>0 wt %</td>
</tr>
<tr>
<td>ACP</td>
<td>60 wt % of AC</td>
<td>40 wt % of PR</td>
<td>0 wt %</td>
</tr>
</tbody>
</table>
moved to higher temperatures ~160–370°C, while the second step is moved to lower temperatures 420–520°C, compared to CCPF5. The weight loss of the first step is in the same range as for CCPF5 while it is significantly larger for the second step. Thus, for both P and S based samples it can be seen that the iron particles slightly enhance the decomposition of the phenolic resin bound materials during carbonization. It is known that the presence of iron compounds influences the pyrolysis or decomposition of phenolic resins. Similar findings about the addition of iron based particles have been reported in literature where the addition of ferrocene [13] and iron oxide [14] reduced the temperature of weight loss marginally. Iron oxide has also been added as a decomposition catalyst in a patent describing recycling of cured phenolic resin [15]. The different temperature behavior and large weight loss for the CCSF5 sample probably reflects the different nature of the binder, P is a resol phenolic resin while S is a novolac phenolic resin having a lower content of formaldehyde. The iron oxide obviously has a higher reactivity towards the latter less crosslinked novolac phenolic resin [16]. The weight loss of SCP and ACP were similar to that of CCPF1 (figure not shown).

**Textural Properties**

Table 2 shows the surface area of starting carbons and carbonized monoliths. The surface area of starting activated carbon is substantially reduced on forming the monoliths. This has been observed in many studies and has been attributed to filling of pores and free space between agglomerates in the carbon by the binder [17,18]. The surface area of monolith with SC is higher than that of the one with activated separated carbon (AC). This is a bit surprising but we have observed this also with other binders and it indicates an activation of separated carbons by the volatiles formed from the resins during carbonization. The BET surface area of SC and AC containing monoliths was higher when binder amount was 40 wt % and it was decreased drastically (from 121 to 7 m²/g in case of ACP) when binder percentage was increased to 50 wt % whereas such a high difference in surface area was not observed in case of CC monoliths. This is possibly due to high ash content of starting SC, AC [11] as compared to CC; the carbon burn out taking place during carbonization of the monolith could have led to even lower carbon content in case of AC, which could be the reason for the low surface area. Therefore, SC and AC containing monoliths were prepared with 40 wt % binder. The pore size distribution obtained by mercury intrusion is shown in Figure 2. The addition of iron oxide, leads to a reduced porosity for both CCPF5 and CCPF10 and in addition a shift to finer pore sizes for the latter. The reduced porosity is in agreement with a partial pore blocking as observed for the surface area above. Table 3 shows that the carbon content is in the range 73–88% and is lower for the ones with iron loading as expected.

**SEM Analysis**

The morphology of the monoliths was examined by SEM using both secondary electrons and back scattered electrons. Figure 3 shows images of CCPF10 (a) and CCSF10 (b) where iron particles are well distributed on the carbon. EDS analysis confirmed the light particles to be iron rich. Both samples

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**Table 2.** Surface area of starting carbon and carbonized monoliths.

<table>
<thead>
<tr>
<th>Sample code</th>
<th>Surface area (BET) (m²/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CC</td>
<td>685</td>
</tr>
<tr>
<td>CCP1</td>
<td>384</td>
</tr>
<tr>
<td>CCP2</td>
<td>356</td>
</tr>
<tr>
<td>CCPF5</td>
<td>360</td>
</tr>
<tr>
<td>CCPF10</td>
<td>290</td>
</tr>
<tr>
<td>CCSF10</td>
<td>277</td>
</tr>
<tr>
<td>SC</td>
<td>172</td>
</tr>
<tr>
<td>AC</td>
<td>420</td>
</tr>
<tr>
<td>SCP</td>
<td>215</td>
</tr>
<tr>
<td>ACP</td>
<td>121</td>
</tr>
</tbody>
</table>

**Table 3.** Carbon content of prepared samples.

<table>
<thead>
<tr>
<th>Sample code</th>
<th>Carbon content (wt %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CCP1</td>
<td>84</td>
</tr>
<tr>
<td>CCPF5</td>
<td>78</td>
</tr>
<tr>
<td>CCPF10</td>
<td>73</td>
</tr>
<tr>
<td>CCS</td>
<td>88</td>
</tr>
<tr>
<td>CCSF5</td>
<td>78</td>
</tr>
<tr>
<td>CCSF10</td>
<td>74</td>
</tr>
</tbody>
</table>
show regions of discrete particles and agglomerated particles; the agglomerated particles appear more in CCPF10. The size of the iron oxide particles in the SEM images is smaller than that measured for the starting powder, which showed a bimodal distribution (Figure 4). There seems to be a deagglomeration may be due to a reaction between iron oxide and binder/carbon during carbonization.

**XRD Analysis**

The XRD patterns for CCPF10 and CCSF10 before and after carbonization are shown in Figure 5. The samples having highest percentage of F particles were selected since the iron peaks were more distinct and the changes during carbonization could be better analyzed. The samples before carbonization show the peaks for hematite (Fe$_2$O$_3$), which was added to the samples. In both the samples, after carbonization, the peaks of hematite have been replaced by that of a spinel (magnetite (Fe$_3$O$_4$) or maghemite (γ-Fe$_2$O$_3$), which have very similar XRD patterns). Heating of iron nitrate impregnated activated carbon in nitrogen at different temperatures have shown that the hematite formed is transformed to magnetite. However, small particles of magnetite, like in the present study, are known to be easily oxidised to maghemite in air at room temperature [19].

**TPR Analysis**

The TPR profile for monoliths with 5% and 10% iron oxide (CCPF5, CCPF10, and CCSF10), is shown in Figure 6. The monoliths having highest percentage of F particles were tested for TPR analysis and one monolith with 5% F was also selected to observe the effect of percentage of F particles on redox behavior. In the case of CCP samples, the one with 5% iron oxide has a single peak at a temperature of around 575°C, while for the sample with 10% iron oxide, a small shoulder is observed at around 460°C and the main peak temperature is reduced to around 550°C. In case of second binder (CCSF10), the shoulder is more pronounced as a distinct peak at 460°C and the main peak is at a higher temperature close to 600°C. This is in agreement with their different behavior upon heating in N$_2$ gas in the TGA as discussed above. The carbonized samples are magnetite/maghemite containing as seen in XRD analysis and the peaks in the TPR analysis reflect the reduction behavior of magnetite/maghemite. The reduction peaks are at temperatures higher than 500°C, and could be reflecting the reduction of magnetite since Fe$_2$O$_3$ (hematite/maghemite) reduction has been observed at lower temperatures [19,20]. The reduction of magnetite to iron has been observed to take place in one or two steps depending on the heating rate, sample size etc. [21,22]. TPR of magnetite has shown a residual peak before

Figure 3. Backscattered SEM images of (a) CCPF10 and (b) CCSF10 from inner portions of the monolith.

Figure 4. Particle size distribution of starting commercial activated carbon and iron oxide. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Figure 5. XRD patterns of 10 wt % F containing monoliths before (CCPF10 and CCSF10) and after carbonization (CCPF10-C and CCSF10-C); h-hematite, m-magnetite/maghemite. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Figure 6. TPR profiles for monoliths with 5% and 10% iron oxide (CCPF5, CCPF10, and CCSF10).
the main peak, which has been attributed to reduction of residual hematite [14] or maghemite formed on the surface of magnetite [23]. Such a shoulder is observed in the 10 wt% iron oxide sample. In the 10% iron oxide samples, there is a difference in behavior depending on the binder used, in spite of similar surface area and morphology of these samples. The difference in reactivity is due to the nature of the binder and iron oxide–carbon interactions. The higher reactivity of novolac resin towards iron oxide seems to activate the latter. TPR of NiO on carbonized ion exchange resin showed different reduction temperatures based on type of carbonization procedure used even in samples with similar surface area and Ni dispersion. This was attributed to metal support interactions [24]. Thus iron oxide carbon composites prepared by incorporating iron oxide powder in the monolith preparation step itself have good redox activity and potential in catalysis and adsorption applications.

Overall, the properties of prepared monoliths showed that they are thermally stable and have adequate BET surface area, which was reduced as the amount of iron particles was increased. The reduced porosity was also confirmed through pore size distribution studies. The elemental analysis was also in correlation with textural properties showing the decrease in carbon content, which is the main contributor to surface area, with the increase in iron particles. The morphological studies confirmed the homogeneous distribution of iron particles. The peaks in TPR correlate with the phases observed during XRD and both TGA and TPR reflect different behavior with different binders.

**Phenol Adsorption**

The phenol adsorption capability was tested with selected samples (CCP2, SCP, and ACP see Table 1). The monoliths for phenol adsorption were selected on the basis of their BET surface area; SC and AC with 40 wt % binder were selected owing to their high surface areas. Although the CC monolith with 50 wt % binder gave slightly better results in terms of BET surface area, for a better comparison between commercial and waste derived monoliths, samples with similar percentage of binder in both the cases were considered. The addition of F particles reduced the surface area of monolith therefore F containing monoliths were not considered for phenol adsorption. The extent of phenol removal in water for two different phenol concentrations for the three monoliths is shown in Figures 7–9. It can be observed that at low phenol concentration, the removal is more than 80% in case of CCP2 whereas it is 51% and 36% in case of SCP and ACP, respectively. This is consistent with the surface area
values of the three monoliths, the highest adsorption being shown by high surface area monolith (CCP2). Thus we can say that the adsorption capacity was directly proportional to surface area of monoliths. The relative phenol removal reduces considerably in all the cases when the phenol concentration is increased. The phenol adsorbed is in the range of 1–2 mg/(g carbon) at 5 mg/L phenol initial concentration and 5–8 mg/(g carbon) at 100 mg/L phenol initial concentration in case of CCP2 monolith. The range of adsorbed phenol for SCP monolith is 0.5–1.5 mg/(g carbon) at 5 mg/L initial phenol concentration and 4–11 mg/(g carbon) at 100 mg/L initial phenol concentration. This is similar to other studies in literature. For example, phenol adsorption in carbon monoliths from coconut shell activated chemically and pressed without binder had a range from 7 mg/g carbon, for a sample with medium surface area (821 m²/g) at 500 mg/L initial phenol concentration and 108 mg/g carbon for a sample with high surface area (1315 m²/g) at 1500 mg/L phenol initial concentration, both after 48 h contact time [5]. Activated carbon chemically activated from coffee grounds showed about 80% removal and adsorption of 1–3 mg/(g carbon) at a phenol concentration of 20 mg/L after 1 h [25]. It should be mentioned that the observed adsorption capacity of SCP monolith is in the same range as that of the CCP2 monolith. SC monoliths with their low cost starting materials and simple preparation procedure therefore show promise for further investigation. One of the aspects being examined is the coating of unburned carbon-binder slurry on fly ash ceramic membrane filters to obtain composite monoliths [26].

CONCLUSIONS
Carbon monoliths prepared from commercial activated carbon and two different forms of unburned carbon of bagasse fly ash using a simple pressureless slurry method with phenolic resin binders were thermally stable and had adequate surface area. Increase in iron concentration resulted in decrease in both surface area and pore size. The reduction of hematite to magnetite took place during carbonization, which could be explored further for magnetic applications. Iron loaded carbon monoliths with both binders show redox activity. The applicability of such commercial and waste derived monoliths for environmental applications such as phenol removal showed that, at low phenol concentrations, removal of more than 80% of dissolved phenol was obtained with commercial carbon monolith whereas waste derived monoliths showed 51% and 36% removal. In terms of adsorption capacities, the values were in the range of 1–2, 0.5–1.5, and 0.3–0.4 mg/g for monoliths CCP2, SCP, and ACP, respectively at low phenol concentration whereas it was 5–8, 4–11, and 4–8 mg/g at high phenol concentration. Unburned carbon in bagasse fly ash thus shows promise as a low cost starting material for carbon monolith preparation and showed comparable adsorption capacity for phenol adsorption when compared with commercial activated carbon monolith’s adsorption capacity. Such a starting material (derived from bagasse fly ash) is being further explored for improving their properties through different carbonization procedures after that a detailed study of phenol adsorption will be carried out and results will be reported in future publications.

ACKNOWLEDGMENTS
This project has been supported by SIDA and the Swedish research council.

LITERATURE CITED


Removal of volatile organic compounds over bagasse ash derived activated carbons and monoliths

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ARTICLE INFO

Article history:
Received 18 July 2015
Received in revised form 28 January 2016
Accepted 17 February 2016
Available online 22 February 2016

Keywords:
Carbon monolith
VOC adsorption
Bagasse ash
Unburned carbon
Phenolic resin binder

ABSTRACT

The unburned carbon in bagasse ash was separated and used as a starting material for activated carbon and carbon monolith preparation. The separated carbon was steam activated in a rotary kiln under different conditions and compared with carbon activated in limited supply of air. The activated carbon from the rotary kiln showed surface areas ranging from 324 to 601 m²/g. Different phenolic resins and cellulosic binders were used to prepare the monoliths using slurry casting method. The effects of different preparation conditions on the properties of the monolith were studied. Upon monolith formation, the surface area was reduced slightly and surface areas in the range 92–479 m²/g were achieved. The activated carbons and monoliths were tested for static adsorption of volatile organic compounds (VOCs) using benzene, xylene, hexane and toluene as model compounds. With the activated carbons, adsorption capacities as high as 22.5, 311, 27.5 and 25.0 g/100 g sample were obtained for toluene, benzene, xylene and hexane, respectively. The adsorption capacities were reduced for the corresponding monoliths. The waste derived activated carbons and monoliths show promising results as adsorbents for the removal of volatile organic compounds.

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1. Introduction

Bagasse ash waste is generated in sugar mills from the use of bagasse as a fuel. This waste usually finds its way to landfilled sites where it is disposed. The large quantities of ash generated every year creates disposal problem since it is difficult to provide large land area. Also, due to the presence of light carbon particles in this ash, the water has to be sprayed over it regularly so that it does not catch fire. To get rid of disposal problems associated with this waste, alternate strategies are being looked into to utilize this waste in an efficient manner. This ash contains 16–35 wt% unburned carbon that can be easily separated and utilized in many applications [1–3]. The bagasse ash has been widely studied as an adsorbent for the removal of pollutants [4–6] but very few other studies have examined the utilization of unburned carbon in bagasse ash. The utilization of unburned carbon from coal fly ash as an adsorbent has however been examined [7–9]. We have recently reported the synthesis of activated carbon monoliths by utilizing unburned carbon of bagasse ash and their applications towards phenol removal, a liquid phase pollutant [10] have been tested successfully. Similarly, such waste derived monoliths can also be tested for other applications such as removal of volatile organic compounds (VOCs).

The presence of VOCs in indoor and outdoor system is of great concern because of their harmful effects on human health. For example, formaldehydes, ketones and benzene are present in building materials, furniture and office equipments, while presence of toluene could be seen in paints, detergents and cleaners and xylene is found in synthetic rubber and polyester clothing. Xylene and hexane are also most commonly used solvents in petroleum industry [11]. Adsorption is a well-studied method [12] for removing these volatile organic compounds and many adsorbents in different forms such as activated carbon fiber [13], granular activated carbon [14], zeolites [15] and monoliths [16] have been tested for its removal. Dynamic [14,15] and static [13,16] adsorption studies have been reported in literature for the removal of volatile organic compounds and significant adsorption capacities and extent of removal has been achieved. The present study examines the performance of waste derived monoliths and activated carbons for the removal of volatile organic compounds using static adsorption method. Due to the large number of samples involved in this study (three types of activated carbons
(AC) and different monoliths made of them), a simple static adsorption method was used to test their adsorption capacities.

2. Experimental

2.1. Preparation of separated and activated carbons

The bagasse ash was collected from a sugar mill in Uttar Pradesh, India. A carbon rich fraction was collected by sieving and floatation as reported before [2]. The resulting separated carbon was then crushed and sieved (through 38 microns) before utilizing it for monolith preparation. This carbon is hereby referred to as SC38.

This separated carbon was activated in a rotary kiln mounted on a tilting frame. The activation was carried out under steam atmosphere at 900 °C and an inclination of 8°, which led to a sample exit rate of approximately 7 g/min. To obtain a higher degree of activation, one sample was prepared at 950 °C and an inclination of 4°. An alternative method to activate the separated unburned carbon was also carried out, by mixing separated carbon and water in 1:7 ratio followed by heating at 800 °C for 3 h, in a limited supply of air [2], thereafter the obtained activated carbons were crushed and sieved through 38 microns. The three samples of activated carbons were named AC8, AC4 and 7AC38. The first two samples were named based on their degree of inclination during activation (4 in case of AC4 and 8 in case of AC8) whereas 7AC38 was named as it was activated using carbon and water ratio of 1:7 and was sieved through 38 microns after activation.

2.2. Monolith preparation

The starting carbons (SC38 and 7AC38) were mixed with different phenolic resin binders (Sumitomo PR311 (solid) (P3), PR-50273 (liquid) (P5) and Satya Cashew novolac resin (liquid) (S)) andcellulosic binder (solid) (CMC), in different ratios (ranging from 20–50 wt%) using appropriate solvents (ethanol for phenolic resins and water for cellulosic binder) to obtain a slurry. The slurry was then poured into a square mold (5 × 5 cm) placed on a smooth glass surface and kept for drying overnight. The square shaped flat monoliths were further dried and carbonized at 550 °C for 4 h, in a partially sealed box with a limited supply of air. Additional monoliths using AC8, AC4 and commercial activated carbon with surface area of 685 m²/g (CC) were prepared with P3 binder, based on its better performance. Since AC8 and AC4 were available in smaller quantities, only the best performing binder was used. The samples prepared are shown in Table 1.

2.3. Characterization of activated carbons and carbon monoliths

Detailed adsorption properties of activated carbons and monoliths were determined using single point (Smart Instruments, results in Tables 2 and 3) and multipoint nitrogen adsorption (Micromeritics ASAP 2020, results in Tables 4 and 5) at 77 K. In case of single point adsorption, the sample was degassed at 150 °C for 1 h before analysis and Brunauer–Emmett–Teller (BET) method was used to obtain surface area. For multipoint nitrogen adsorptions, the samples were degassed at 200 °C for 10 h in vacuum prior to analysis and BET, Dubinin–Radushkevich (DR) and Density Functional Theory (DFT) methods were used to determine the apparent surface area, pore volume and pore size distributions, respectively.

The characterization of activated carbons and monoliths was done through Scanning Electron Microscopy (SEM) using JEOL JSM7000 equipped with an Oxford INCAx SIGHT energy dispersive detector (EDS) and Transmission Electron Microscopy (TEM) with a JEOL3010 and a JEOL 2000FX equipped with EDS analyzer. For the TEM studies of activated carbons, the powders were dispersed in butanol and thereafter a drop of the suspension was placed on a holey carbon film supported by a copper grid.

The thermal analysis of binders and carbon monoliths was performed using thermogravimetric analyzer (PerkinElmer 519 N). The sample was placed in a platinum crucible and heated at the rate of 10 °C/min in nitrogen atmosphere in the temperature range of 20–900 °C.

Infrared spectroscopic analyses of pressed pellets of powder samples mixed with KBr, (1:100 ratio) were performed in a FTIR spectrometer (PerkinElmer, India) at room temperature over the frequency range of 400–4000 cm⁻¹.

Boehm titration [17] was performed to identify the amount of acidic (carboxyl, hydroxyl and lactonic groups) and basic

![Table 2](image)

<table>
<thead>
<tr>
<th>Binder</th>
<th>BET surface area (m²/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P3</td>
<td>221</td>
</tr>
<tr>
<td>P5</td>
<td>4</td>
</tr>
<tr>
<td>S</td>
<td>43</td>
</tr>
<tr>
<td>CMC</td>
<td>575</td>
</tr>
</tbody>
</table>

* single point measurements.

![Table 3](image)

<table>
<thead>
<tr>
<th>Sample</th>
<th>BET surface area (m²/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ACIP340</td>
<td>445</td>
</tr>
<tr>
<td>ACIP340</td>
<td>277</td>
</tr>
<tr>
<td>7ACIP340</td>
<td>179</td>
</tr>
<tr>
<td>CCP340</td>
<td>395</td>
</tr>
</tbody>
</table>

* single point measurements.
functional groups. 1.0 g of sample was added to 50 ml of 0.050 M aqueous solutions of NaOH, Na₂CO₃, NaHCO₃ and HCl. The solutions were sealed and shaken for 24 h and then filtered. 10.0 ml filtrate was taken and excess of base and acid was titrated against HCl and NaOH, respectively, using phenolphthalein as an indicator. NaOH (aq) neutralizes all the acidic (carboxyl, hydroxyl and lactonic) groups, Na₂CO₃ (aq) neutralises lactonic and carboxyl groups, whereas NaHCO₃ (aq) neutralizes only carboxyl groups. Based on this assumption, the total number of acidic and basic sites was calculated.

2.4. Adsorption tests

Static adsorption measurements were carried out, to test the performance of activated carbons and formed monoliths for removal of volatile organic compounds at room temperature (22–26 °C). The static adsorption experiments were carried out in a desiccator. Toluene, benzene, xylene and hexane were used as VOCs. For each VOC, 100 ml was poured into a beaker which was then placed in the desiccator. The carbon samples were first dried at 110 °C overnight and then a known weight of the sample was kept in a bottle within the same desiccator containing the VOC. The bottle containing the adsorbents was taken out after 24 h and then closed tightly and weighed. The bottle was again kept in the desiccator and weighed again every 24 h until the weight became constant, which was considered the saturation point. An empty bottle was also kept in the desiccator as reference. The adsorption capacity was calculated as the difference between increased weight and initial weight after saturation was achieved [16]. All the experiments (except for AC4 and AC8 carbons and monoliths, which were available in limited quantities) were performed in duplicates and the results varied within ±0.5%.

3. Results and discussion

3.1. Activated carbons

The surface area and pore characteristics of the activated carbon samples are shown in Table 4. The surface area of AC4 is highest followed by 7AC38 and AC8 and the same trend is observed for the total pore volume. In terms of micropore volume as a percentage of total pore volume, 7AC38 has the highest value.

A typical SEM image of the rotary kiln activated carbon, see Fig. 1(a), shows that the sample retains the features of the unactivated separated carbon [1] and in some regions a very porous structure is seen, see Fig. 1(b). TEM images showed the presence of mostly amorphous carbon, as shown in Fig. 2. In some regions, lattice fringes were observed, which were not present in the unactivated separated carbon. It is probably from crystalline inorganic ash, which is relatively higher in the activated samples due to loss of carbon during activation.

3.2. Binder characteristics

The TG study of two Sumitomo phenolic resin binders (P3 and P5) as a function of temperature in N₂ (g) revealed almost similar weight losses, occurring in three main regions, see Fig. 3. The weight loss in first region occurs in the temperature range 150–400 °C, which is attributed to the removal of water and a mixture of phenol and cresol [18,19]. The major weight loss in the second region in the temperature range between 400 and 700 °C can be attributed to hydrogen, carbon dioxide, methane, phenols and water formed as by-products during the decomposition of phenolic resins [18,19]. The small amount of weight loss in the third region 700–900 °C can be explained mainly by the removal of
hydrogen (from methylene groups), together with small amounts of water, carbon dioxide and carbon monoxide decomposition products. The extent of weight loss in the different regions was different for the two binders. For P3, a solid novolac binder, the weight loss was mostly in the second region, while for the liquid resol binder, P5, the weight loss in the first and second regions was almost identical. A similar profile has been reported for a resol phenolic resin and the low temperature weight loss was then attributed to release of the solvent [20]. The phenolic resins pyrolyzed in an inert atmosphere usually shows around 50–60% weight loss [21]. Binder S, a novolac liquid binder, showed a major weight loss in the range 200–500 °C (Fig. not shown).

Fig. 1. (a) SEM image of rotary kiln activated carbon showing cellular structure similar to unburned carbon. (b) High magnification SEM image of rotary kiln activated carbon showing fine pores on the carbon surface.

Fig. 2. TEM image of amorphous carbon in rotary kiln activated sample.
The weight loss in case of cellulosic binder (CMC) occurred in the temperature range of 100–900 °C. It started at 100 °C and showed almost 20% weight loss up to 250 °C, due to loss of water. The majority of weight loss occurred in two main regions; one from 250–300 °C, where up to 30% of weight loss was observed indicating the decomposition of cellulosic chain, followed by minor losses up to 800 °C, second major weight loss occurred between 800 and 900 °C, see Fig. 3. It is known that weight loss above 300 °C is primarily due to evolution of different gases mainly hydrogen, carbon dioxide, carbon monoxide, methane, ethane and water [22–24]. Based on the TG results, a temperature of 550 °C was chosen for the carbonization of the binders.

For the phenolic resin binders, the surface areas, after carbonization, were highest for the P3 binder, see Table 2. The resol binder P5 is expected to have a higher degree of crosslinking compared to the novolac P3 and Satya (S) binders [19]. It has been observed that the surface area of uncrosslinked phenolic resins after carbonization is higher than that for crosslinked ones [25].
The carbonized cellulosic binder (CMC) showed highest surface area among all the binders.

SEM images of the carbonized binders are shown in Fig. 4. It can be observed that the cellulosic (CMC) binder, shown in Fig. 4(a), shows clear porous regions, while the other binders show much smoother surfaces. The liquid binders (P5 and S) shown in Fig. 4(c) and 4d have a smooth film like appearance after carbonization.

3.3. Separated carbon monoliths

The thermal analysis of green monoliths of separated carbon (SC38), with binders P3 and P5, are shown in Fig. 5. The weight loss is larger than that for the pure binder and could be due to presence of volatile materials in the separated carbon. Since these samples were dried (at room temperature) before thermal analysis, the low temperature weight loss observed for P5, seen in Fig. 3, is not present here.

The surface areas of the final monoliths are higher than that of unburned separated carbon and values up to 416 m²/g are obtained (SC38P330). For 30% binder composition, the P3 binder shows finer features and P5 has a smooth surface as seen in Fig. 6; overall, the porosity seems higher for the monoliths with more binder (40%), shown in Fig. 6(c) and (d).

3.4. Activated carbon monoliths

The weight loss profile for green monoliths made from activated carbons is similar to what observed for the monoliths

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**Fig. 5.** TGA profile of green monoliths with separated carbon.

**Fig. 6.** SEM images of carbonized monoliths (a) SC38P330 (b) SC38P530 (c) SC38P340 (d) SC38P540.
from separated carbon up to 400 °C, although the overall weight loss is less, see Fig. 7. In addition, the large weight loss observed above 400 °C for the separated carbons, is much smaller for the monoliths made from activated carbons. This can be explained by the removal of volatile materials from the carbon during the activation step. The BET surface areas for P3 based monoliths made from activated carbons are shown in Table 3. The AC4 containing monolith has highest surface area, higher than commercial carbon based monoliths (CC). The textural properties of the monoliths prepared from 7AC38 with binders of different types are shown in Table 5. The surface area varies with type and amount of binder. Monoliths with P3 show higher surface areas compared to the other binders and the surface area increases with binder content. The 7AC38P340 sample has the largest microporous surface area. It is interesting to note that the CMC binder, which itself gives porous structure with high surface area upon carbonization did not form a high surface area monolith. This can be explained by the observation that the weight loss for pure

Fig. 7. TGA profile of green monoliths with activated carbon (7AC38).

Fig. 8. Morphology of 7AC38P330 (a) green (b) carbonized. A part showing covering binder is marked with an arrow.
CMC during carbonization is significantly higher than for the phenolic resin binders (P3 and P5), see Fig. 3 and hence carbonized CMC formed a smaller fraction of the monolith. Thus the composition of binder and its behavior with temperature are deciding factors for the overall porosity of monoliths. The release of different compounds during carbonization from binder gives rise to rearranged carbon network which accounts for the porosity of monolith. In both SC and AC, P3 binder appeared to form monoliths with high porosity and surface area.

The SEM images of the green and carbonized 7AC38P330 monoliths are shown in Fig. 8. For both samples regions with the cellular structure from the unburned carbon can be seen. However, for the green sample areas covered by binder can be found, which are not observed for the carbonized monolith.

The functional groups present on selected carbon monoliths were identified with the help of FTIR analysis, see Figs. 9 and 10. FTIR spectra of 7AC38 with P3, S and CMC binders are shown in Fig. 9. As can be seen, all the three samples showed sharp absorption bands at 462–472 cm⁻¹ (Si—O—Si from ash constituent of carbon), 748–783 cm⁻¹ (hydrogen bonded to aromatic groups), 2918–2970 cm⁻¹ (C—H), two broad bands in the range 1126–1020 cm⁻¹ due to C—O group and Si—O—Si (1150–1050 cm⁻¹ is the expected range for C—O) [26] and 3547–3319 cm⁻¹ due to OH group (expected range 3600–3200 cm⁻¹) [26]. P3 and Satya (S) samples showed additional peaks at 1570 cm⁻¹ (C=C), while CMC binder showed absorption bands at 1400 cm⁻¹ (C—H stretching). The C=C absorption band was absent for the CMC binder, as expected. Fig. 10 shows the spectra of

**Fig. 9.** FTIR spectra of 7AC38 monolith with different binders (a) P3 (b) S (c) CMC.
monoliths made from the different activated carbons using P3 as binder. Compared to 7AC38P340 (Fig. 9(a) and repeated in Fig. 10(a)), the main differences are the absence of OH absorption band (3600–3200 cm\(^{-1}\)) in case of AC4P340 and the presence of absorption band at 1712 cm\(^{-1}\) (C=O) in case of AC8P340. This is similar to the starting carbons where the OH absorption band gives a clear peak for AC8, a small peak in 7AC38 and is absent for the starting AC4. The OH groups thus appear to decrease in number with increasing degree of activation. This reduction and finally disappearance of the OH band in the IR-spectra with activation of bagasse ash carbon has also been reported by others [27]. In addition, the presence of the C=O band, which was observed for AC8 but not for AC4 can be attributed to the lesser degree of activation of the former. In the spectra of these three monoliths, except for the one at around 780 cm\(^{-1}\), the other bands found for carbonized binder P3 (figure not shown) were not observed. The band at 780 cm\(^{-1}\) corresponds to substituted hydrogen which generally requires higher temperature for decomposition (as explained in thermal analysis section) than the used temperature of 550 °C for carbonization. The removal of other bands in the monolith observed for carbonized P3 could be due to the effect of ash constituents, which may have promoted greater carbonization of P3.

The acidic and basic groups from Boehm titration for selected samples are shown in Table 6. It is seen that the monoliths made with CMC has the largest number of basic groups while the liquid
binder (P5 and S) monoliths have the largest number of acidic groups. The monolith 7AC38P340 has least number of acidic groups which is in agreement with FTIR results since it did not show the presence of acidic surface oxygen groups.

3.5. Adsorption

Many factors such as surface area of adsorbents, type of porosity, presence of acidic groups as well as properties of the VOC are known to affect the adsorption capacity. In the samples prepared, due to influence of these different parameters with varying effects on the adsorption capacity, the results were difficult to interpret based on any single assumption or parameter. The toluene adsorptions of the different activated carbons and their monoliths are shown in Fig. 11. It is seen that the adsorption capacity of activated carbons with higher surface area (AC4 and CC) are larger than for those with lower surface areas (AC8 and 7AC38). Their corresponding monoliths also show a higher adsorption capacity. The reduction in adsorption for CC and AC4 monoliths (CCP340, AC4P340) compared to the corresponding activated carbon can be attributed to the decrease in surface area due to partial pore blockage due to the binder addition [16,28].

Given the reduction in adsorption capacity of the monoliths from high surface area carbons, a different method of preparation to obtain high surface area monoliths was adopted and the dynamic adsorption and kinetics of these monoliths will be the topic of a subsequent publication. The high surface area CC was subjected to dynamic adsorption of toluene (adsorption at 40 °C, 400 ppm toluene, space velocity 0.5 Lmin⁻¹g⁻¹) and the kinetics were compared with pseudo first order and pseudo second order models [29]. Table 7 represents the constants of pseudo-first and second order kinetic models for toluene adsorption. The fit matched the pseudo first order kinetics (regression coefficient greater than 0.95) with good match between the experimental (26.1 g/100 g) and calculated (24.3 g/100 g) equilibrium adsorption capacities and a rate constant of $5 \times 10^{-3}$ h⁻¹. These results indicate that the toluene adsorption is governed by physisorption. The pseudo second order model was not well fitted and linear regression coefficient was very low.

A comparison of the adsorption properties of the different VOCs for the 7AC38 monoliths made with the different binders show, that those made with liquid binders P5 and S have low adsorption capacities irrespective of their surface areas as seen in Fig. 12. For 7AC38P550, it is probably due to the low surface area and the large number of acid surface oxygen groups (Boehm’s titration). 7AC550 has medium surface area; however, the low adsorption could be due to the high micropore volume (low mesopore volume) and the large number of acid surface oxygen groups. A reduction in VOC adsorption (o-xylene) with increase in acid surface oxygen groups has been reported [14]. Overall 7AC38CMC40 and 7AC38P340 show the best adsorption properties, although the adsorption for xylene is higher for 7AC38P320 and 7AC38P330 than for 7AC38P340.

The activated carbons with high adsorption capacities for toluene (AC4 and CC) also showed high adsorption capacities for the other VOCs tested as shown in Fig. 13.

In general the adsorption capacities for different VOCs did not show a clear trend for the different monoliths and activated carbons. In some cases, the maximum adsorption capacity was for xylene, which could be due to its high boiling point. The low adsorption capacity for benzene in some monoliths could be due to

---

**Table 6**

Results of Boehm titration (mmol/g).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Carboxylic</th>
<th>Lactonic</th>
<th>Phenolic</th>
<th>Basic</th>
<th>Acidic</th>
<th>All</th>
</tr>
</thead>
<tbody>
<tr>
<td>CC</td>
<td>2.9</td>
<td>0.3</td>
<td>0.8</td>
<td>23.8</td>
<td>4.0</td>
<td>27.8</td>
</tr>
<tr>
<td>7AC38</td>
<td>2.0</td>
<td>0.2</td>
<td>0.4</td>
<td>27.2</td>
<td>2.6</td>
<td>29.8</td>
</tr>
<tr>
<td>7AC38P1200</td>
<td>1.5</td>
<td>0.3</td>
<td>0.7</td>
<td>24.7</td>
<td>2.5</td>
<td>27.2</td>
</tr>
<tr>
<td>7AC38P330</td>
<td>1.4</td>
<td>0.7</td>
<td>0.4</td>
<td>23.8</td>
<td>2.5</td>
<td>26.3</td>
</tr>
<tr>
<td>7AC38P340</td>
<td>1.8</td>
<td>0.1</td>
<td>0.1</td>
<td>24.0</td>
<td>2.0</td>
<td>26.0</td>
</tr>
<tr>
<td>7AC38S50</td>
<td>2.4</td>
<td>0.2</td>
<td>0.4</td>
<td>27.4</td>
<td>3.0</td>
<td>30.4</td>
</tr>
<tr>
<td>7AC38P550</td>
<td>2.5</td>
<td>0.2</td>
<td>0.4</td>
<td>27.3</td>
<td>3.0</td>
<td>30.3</td>
</tr>
<tr>
<td>7AC38CMC40</td>
<td>1.9</td>
<td>0.3</td>
<td>0.6</td>
<td>35.0</td>
<td>2.8</td>
<td>37.8</td>
</tr>
</tbody>
</table>

**Fig. 11.** Adsorption capacity of toluene over powdered ACs and their monoliths. Their surface areas are presented as well.

**Table 7**

Comparison of pseudo-first and second order kinetic models for toluene adsorption.

<table>
<thead>
<tr>
<th>Adsorbent</th>
<th>$q_{exp}$ (g/100 g)</th>
<th>$q_0$ (g/100 g)</th>
<th>$k_1$ (h⁻¹)</th>
<th>$R^2$</th>
<th>$q_0$ (g/100 g)</th>
<th>$k_2$ (g mg⁻¹ h⁻¹)</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>CC</td>
<td>26.1</td>
<td>24.3</td>
<td>$5 \times 10^{-3}$</td>
<td>0.9531</td>
<td>0.83</td>
<td>$2.7 \times 10^{-1}$</td>
<td>0.2256</td>
</tr>
</tbody>
</table>
**Fig. 12.** Adsorption capacities of different VOCs over monoliths (from left to right—toluene, benzene, xylene, hexane).

**Fig. 13.** Adsorption capacities of different VOCs over activated carbons (from left to right—toluene, benzene, xylene, hexane).

**Table 8**  
Adsorption capacities of VOCs reported in literature.

<table>
<thead>
<tr>
<th>Type of adsorbent</th>
<th>Toluene adsorption (g/100 g)</th>
<th>Benzene adsorption (g/100 g)</th>
<th>Xylene adsorption (g/100 g)</th>
<th>Hexane adsorption (g/100 g)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Activated carbons</td>
<td>22.5</td>
<td>31.1</td>
<td>27.5</td>
<td>25.0</td>
<td>Present</td>
</tr>
<tr>
<td>Monoliths</td>
<td>18.3</td>
<td>11.1</td>
<td>14.7</td>
<td>13.0</td>
<td>[13]</td>
</tr>
<tr>
<td>(Viscose based) AC fibers</td>
<td>54</td>
<td>48</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td>VACF+Cu loading</td>
<td>62</td>
<td>49</td>
<td>–</td>
<td>–</td>
<td>[17]</td>
</tr>
<tr>
<td>Vaseline loaded expanded graphite</td>
<td>21.3</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>[30]</td>
</tr>
<tr>
<td>Physically &amp; chemically activated commercial AC</td>
<td>64</td>
<td>34</td>
<td>–</td>
<td>–</td>
<td>[31]</td>
</tr>
<tr>
<td>(Sewage sludge based) AC</td>
<td>35</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td>Commercial monolith</td>
<td>23</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>[32]</td>
</tr>
<tr>
<td>AC impregnated with acids &amp; alkali</td>
<td>37.2</td>
<td>49.9</td>
<td>67.1</td>
<td>–</td>
<td>[33]</td>
</tr>
<tr>
<td>Peat derived commercial AC</td>
<td>–</td>
<td>29.6</td>
<td>–</td>
<td>–</td>
<td>[34]</td>
</tr>
</tbody>
</table>
absence of narrow micropores, which are mainly responsible for benzene adsorption [30].

A comparative analysis of adsorption capacities as reported in different studies is given in Table 8. It can be seen that most of the studies are based on adsorption of VOCs over activated carbons and very few on monoliths. VOC adsorption has been tested both on commercial and waste derived activated carbons. The adsorption capacity is reported higher for commercial activated carbon, i.e. around 64 g/100 g for toluene [30], whereas for sewage sludge derived activated carbon maximum adsorption capacities up to 35 g/100 g have been reported [31]. The maximum adsorption capacity of 22.5 g/100 g for toluene in the present study is low compared to commercial activated carbon, but keeping in mind the low cost of such waste based adsorbents and environmental hazards caused due to such waste materials, these values seems promising. In case of other VOCs (benzene, hexane and xylene), similar differences were observed between commercial and waste derived activated carbons as in this study. For the monoliths, the adsorption capacity is known to decrease compared to the activated carbons and 23 g/100 g of toluene adsorption has been reported for commercial carbon based monoliths [32].

This is similar to what we observed for our bagasse ash derived activated carbon monolith, which showed a reduction in adsorption capacity from 22.5 to 18.3 g/100 g in case of toluene. Thus the bagasse ash derived monoliths could be seen as potential adsorbents which can replace the expensive commercial carbon monoliths keeping the overall cost of adsorption procedure at minimum. The static adsorption results for VOCs have given appreciable results in present study and therefore dynamic adsorption capacities should also be tested to ascertain the applicability of monoliths for commercial purposes.

4. Conclusions

The activated carbons produced through different activation methods employing bagasse ash showed good thermal stability and BET surface areas in the range of 324–601 m²/g, depending on the conditions of activation. The highest surface area was obtained when prepared in a rotary kiln at 950 °C with higher residence time in kiln (AC4). Since this carbon was made in a rotary kiln which is commonly used in industrial production of activated carbon, its industrial application is feasible. The second highest surface area was obtained for the carbon prepared by heating carbon–water mixture in limited supply of air (7AC38) while a lower temperature and lower residence time in kiln (AC8) yielded the lowest surface area. A well developed porous structure was confirmed through microscopic studies. The static adsorption capacities of such waste derived activated carbons towards toluene, benzene, xylene and hexane shows promising results although no clear trend was observed in the adsorption of different VOCs owing to many parameters overall affecting the adsorption capacity. The monoliths formed using activated carbons showed a reduction in the BET surface areas. The binder P3 gave higher surface areas than other binders; the value increased with increasing binder addition. The monolith adsorption capacity was reduced when compared to their activated carbon powder counterparts. The two solid binders (phenolic and cellulosic) gave better results than the two liquid binders (phenolic and novolac) tested. The adsorption capacity of the best monoliths prepared from unburned carbon were, however, comparable with commercial carbon monoliths. In order to increase the adsorption capacity, different carbonization conditions should be tested in order to further enhance the adsorption capacities. Overall, unburned carbon separated from bagasse ash can be successfully utilized for preparing adsorbents and can be employed for environmental related applications.

Dynamic adsorption study of a particular VOC i.e. toluene with high surface area monoliths will be published in future to understand the adsorption mechanism and kinetics.

Acknowledgements

The authors thank Swedish Research Council for providing financial support to carry out the above research work. AB is also grateful to Ministry of New and Renewable Energy (MNRE) for providing financial support as a Senior Research Fellowship.

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