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Biomass Derived, Hierarchically Porous, Activated Starbons® as Adsorbents for Volatile Organic Compounds

Victoria Baird,^[a] Ryan E. Barker,^[a] Benjamin Longhurst,^[a] C. Rob McElroy,^[a, b] Siyu Meng,^[a] Michael North,^{*[a]} and Junzhong Wang^[a]

The use of potassium hydroxide activated Starbons® derived from starch and alginic acid as adsorbents for 29 volatile organic compounds (VOCs) was investigated. In every case, the alginic acid derived Starbon (A800K2) was found to be the optimal adsorbent, significantly outperforming both commercial activated carbon and starch derived, activated Starbon (S800K2). The saturated adsorption capacity of A800K2 depends on both the size of the VOC and the functional groups it

contains. The highest saturated adsorption capacities were obtained with small VOCs. For VOC's of similar size, the presence of polarizable electrons in lone pairs or π -bonds within non-polar VOCs was beneficial. Analysis of porosimetry data suggests that the VOC's are being adsorbed within the pore structure of A800K2 rather than just on its surface. The adsorption was completely reversible by thermal treatment of the saturated Starbon under vacuum.

Introduction

Volatile organic compounds (VOCs) are defined as being organic compounds with a boiling point of up to 250 °C at 101.3 kPa.^[1] VOC's are produced both biogenically and anthropogenically with their low boiling points allowing them to readily vaporize and enter the atmosphere resulting in atmospheric pollution such as photochemical smog.^[2] Large scale biogenic sources of VOCs include the emission of isoprene and terpenes from forests^[3] and halogenated compounds from the oceans.^[4] Anthropogenic sources of VOCs include vehicle emissions^[5] and application of paints and other coatings.^[6] Adsorption of VOCs onto solid adsorbents is an important and economically viable^[7] method to prevent human exposure and to facilitate the environmental sensing, monitoring and control of VOCs. The effectiveness of VOC adsorption is known to depend on the properties of both the VOC and the adsorbent as well as the adsorption conditions.^[8] Materials including metal-organic frameworks (MOFs), clays, silicas and organic polymers have been studied as adsorbents for VOCs,^[9] though

most work has investigated the use of carbon-based adsorbents.^[7,8,10] However, only a small number of VOCs (typically 1–3) are usually investigated with any particular adsorbent and the adsorbents often require modification such as nitrogen doping which reduces their sustainability and economic viability.

Starbons® are sustainably sourced, mesoporous carbons prepared from polysaccharides (starch,^[11] alginic acid^[12] or pectin^[13]) present in waste biomass without the use of a template.^[14] They are now commercially available and prepared by a three-step process involving gelatinisation and retrogradation in water,^[11] freeze-drying to form a mesoporous aerogel^[15] and carbonisation at 300–1200 °C.^[16] The polysaccharide precursor determines the morphology^[17] of the Starbon and also the pore size and shape.^[18] Alginic acid and pectin derived Starbons® have larger mesopore volumes compared to starch derived Starbon.^[12,14] The chemical groups present in Starbons® are mainly determined by the carbonation temperature.^[16] At 300 °C, carbonisation retains oxygen containing functionalities. By 600 °C, monocyclic aromatic rings predominate and at higher temperatures fused polycyclic aromatics are formed. Starbons® have found applications including in catalysis,^[19] electrochemistry^[20] and as adsorbents.^[12,17,18,21] Whilst the predominantly mesoporous nature of Starbons® results in favourable mass-transfer rates, a higher surface area associated with a more microporous structure would be beneficial to their use as adsorbents. Therefore, it was recently shown that Starbons® could be activated by treatment with potassium hydroxide, oxygen, or carbon dioxide to produce hierarchically porous activated Starbons® with greatly increased surface areas and micropore volumes.^[22] The morphology of the activated Starbon could be controlled by choice of activating agent, activation temperature and activation time. Activated Starbons® with optimal pore structures and surface areas were shown to be excellent adsorbents for both carbon dioxide^[22] (from a mixture with nitrogen) and dyes (from aqueous solution).^[23]

[a] V. Baird, R. E. Barker, B. Longhurst, Dr. C. R. McElroy, S. Meng, Prof. M. North, J. Wang
Green Chemistry Centre of Excellence
University of York
York, UK, YO10 5DD (United Kingdom)
E-mail: Michael.north@york.ac.uk

[b] Dr. C. R. McElroy
School of Chemistry
University of Lincoln
Lincoln UK, LN6 7DL (United Kingdom)

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In view of the successful application of activated Starbons[®] as adsorbents for both small gases and large dyes, we decided to investigate their application as adsorbents for a range of 29 VOCs and in this paper, we show that the optimal, activated Starbon (A800K2) can significantly outperform both conventional activated carbon and unactivated Starbons[®] in this application. The inclusion of 29 VOCs in the study shows the generality of this adsorption process and allows the influence of the VOC structure on the adsorption capacity to be investigated, something that is not possible when only a small number of VOCs are studied.

Results and Discussion

The two most effective activated Starbons[®] for carbon dioxide adsorption were S800K2 and A800K2 where S and A represent materials prepared from starch and alginic acid respectively, 800 is the activation temperature (in °C) and K2 indicates that activation was achieved using two mass equivalents of potassium hydroxide.^[22] Since the smallest VOCs are comparable in size to carbon dioxide, these two materials were initially selected as the basis for this project, with the corresponding unactivated Starbons[®] (S800 and A800) and commercial, Norit activated carbon (AC) being used as control samples.

X-ray photoelectron spectroscopy (XPS) analysis of S800, A800, S800K2 and A800K2 showed that they were all composed of 85–95% carbon, with the remainder (5–14%) being almost entirely oxygen. Combustion analysis showed that the hydrogen content of the materials was 0–0.2%. Deconvolution of the C1s and O1s peaks indicated that the carbon was predominantly sp² hybridised consistent with a graphitic type structure and the oxygen was present as ethers, ketones, and esters/lactones.^[22] SEM imaging of the materials showed that the rough, nodular surface of S800 and A800 was transformed into a much smoother surface in S800K2 and A800K2 as a result of the activation process.^[22]

Surface area and pore volume data for these materials are given in Table 1. The activation process results in a two–three fold increase in both the surface area and micropore volumes of S800K2 and A800K2 compared to S800 and A800. In contrast,

Material	BET surface area [m ² g ⁻¹]	Micropore volume [cm ³ g ⁻¹] ^[a]	Mesopore volume [cm ³ g ⁻¹] ^[b]	Total pore volume [cm ³ g ⁻¹] ^[c]
S800	632	0.22	0.14	0.31
A800	485	0.16	0.15	0.25
AC	812	0.34	0.40	0.74
S800K2	1347	0.40	0.18	0.55
A800K1	975	0.32	0.27	0.45
A800K2	1202	0.49	0.07	0.43
A800K3	1477	0.49	0.08	0.49
A800K4	1706	0.47	0.1	0.56
A800K5	1271	0.20	0.15	0.46

[a] Determined using the t-plot method. [b] Determined using the BJH = Barrett Joyner Halenda method. [c] Determined using the HK = Horvath Kawazoe method at $P/P_0 = 0.99$.

the mesopore volumes increased only slightly so that the total pore volumes also approximately double compared to the unactivated analogues and the activated materials become hierarchically porous. TEM imaging confirmed the presence of mesopores and micropores in the materials.^[22]

29 VOCs varying in size, molecular weight, boiling point and functionality were selected for study (Table 2). These are all commonly used solvents, with some being substances of concern (e.g., pyridine, chloroform, hexane) whilst others are considered greener solvents (e.g., ethanol, dimethyl carbonate, 2-methyl-THF). The VOCs included in Table 2 include those present in household products (e.g., acetone, esters), foodstuffs (e.g., ethanol, 2-propanol, acetic acid), painting products (dichloromethane), vehicle exhausts (toluene) as well as those with medical applications (e.g., diethyl ether, chloroform) and those used as solvents in the industrial synthesis of fine chemicals and pharmaceuticals.

To measure the saturated adsorption capacities, a beaker containing a large excess of the solvent and sample tubes containing weighed amounts of adsorbent were placed in a sealed container and left to equilibrate at 21 °C. Control experiments using toluene showed that equilibrium was reached in under 20 h, so an exposure time of 21 h was used for all solvents except where indicated in Figure 1. The container was then opened, the tubes quickly sealed and then weighed to determine the amount of solvent adsorbed. All adsorption measurements were carried out in triplicate and the resulting

Table 2. VOCs used in this work and their properties.

VOC	Boiling point [°C]	Number of non-H atoms	Molecular weight	Functionality
Diethyl ether	35	5	74	ether
Pentane	36	5	72	hydrocarbon
Dichloromethane	40	3	85	haloalkane
Petroleum ether	40–60			hydrocarbon
Acetone	56	4	58	ketone
Chloroform	61	4	119	haloalkane
Methanol	65	2	32	alcohol
THF	66	5	72	ether
Diisopropyl ether	69	7	102	ether
Hexane	69	6	86	hydrocarbon
Ethyl acetate	77	6	88	ester
Ethanol	78	3	46	alcohol
Butanone	80	5	72	ketone
2-Methyl-THF	80	6	86	ether
Cyclohexane	81	6	84	hydrocarbon
Acetonitrile	82	3	41	nitrile
<i>Tert</i> -butanol	82	5	74	alcohol
2-Propanol	83	4	60	alcohol
Isopropyl acetate	89	7	102	ester
Dimethyl carbonate	90	6	90	carbonate
Heptane	98	7	100	hydrocarbon
Cyclopentyl methyl ether	106	7	100	ether
Toluene	111	7	92	aromatic
Pyridine	115	6	79	aromatic
Acetic acid	118	4	60	acid
1-Butanol	118	5	74	alcohol
Butyl acetate	126	8	116	ester
Butyl isobutyrate	156	10	144	ester
Butyl butyrate	166	10	144	Ester

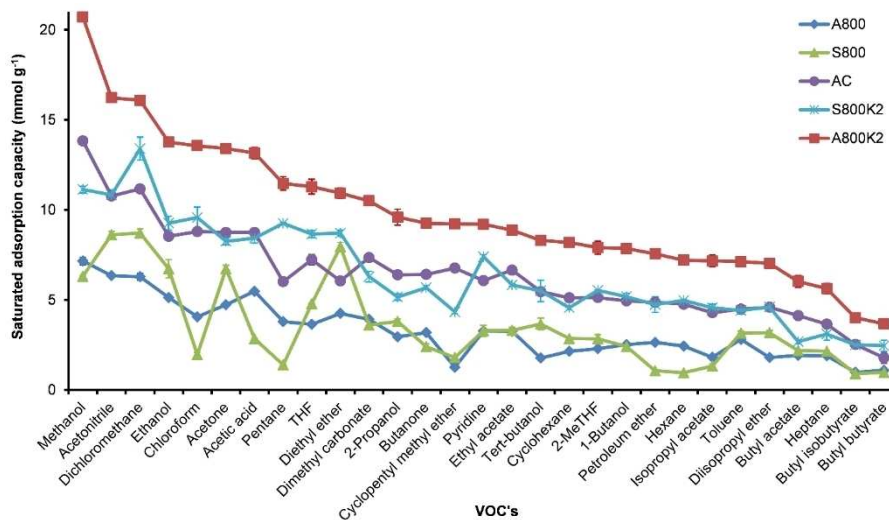


Figure 1. Saturated adsorption capacities for 29 VOC's. All data after an equilibration time of 21 h except for 1-butanol, butyl acetate, butyl butyrate and butyl isobutyrate which were equilibrated for 67 h. Data points are the average of three measurements with error bars showing \pm one standard deviation.

adsorption data is shown in Figure 1. This data is tabulated and shown as separate plots for each adsorbent in the Supporting Information.

The data shown in Figure 1 show that the five adsorbents fall into three categories. The unactivated Starbons® (S800 and A800) generally have similar saturated adsorption capacities for all 29 solvents and, except for diethyl ether adsorbed onto S800, these are the least effective adsorbents. Activated carbon (AC) and activated starch derived Starbon (S800K2) always have similar saturated adsorption capacities and activated alginate derived Starbon (A800K2) has the highest saturated adsorption capacity for all 29 VOCs. These results correlate well with the micropore volumes of the materials given in Table 1 for which $A800K2 > S800K2 > AC > S800 > A800$. The results for the most effective adsorbents (the activated Starbons) do not correlate with the BET surface areas for which $S800K2 > A800K2$ and there is no correlation with the mesopore or total pore volumes of the materials. This suggests that (at least for the most effective adsorbents), adsorption of VOC's occurs primarily within the micropores of the adsorbent.

To investigate the factors that influence the adsorption of these VOCs onto the five adsorbents, the variation of saturated adsorption capacities amongst VOCs which contain the same functional group was studied. Figures 2–4 show the saturated adsorption capacities for five hydrocarbons (Figure 2), five alcohols (Figure 3) and five esters (Figure 4). In each case, for adsorption onto A800K2, S800K2 and AC the saturated adsorption capacity was found to decrease as the size of the VOC increased, with this being particularly apparent for adsorption onto A800K2. For adsorption onto unactivated Starbons® (S800 and A800), this effect is less apparent. These results again directly correlate to the micropore volumes of the adsorbents (Table 1) and suggest that for A800K2, S800K2 and AC, the adsorption is largely occurring in micropores (diameters $< 2 \text{ nm}^{[24]}$) where the size of the VOC will affect its ability to

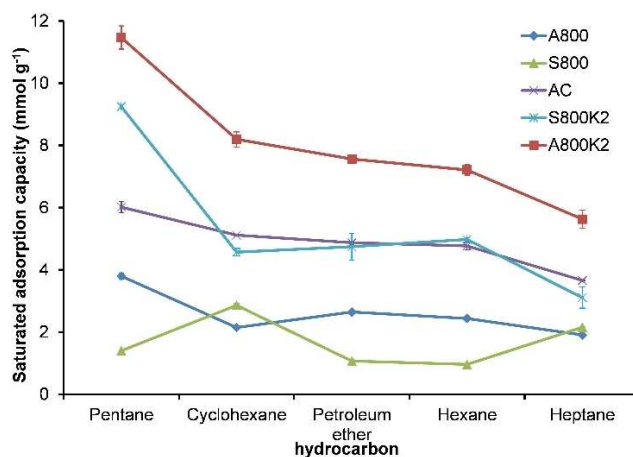


Figure 2. Influence of adsorbate size on the adsorption of hydrocarbons.

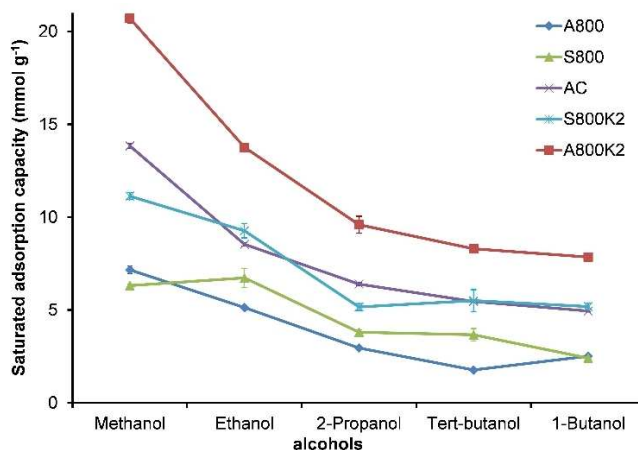


Figure 3. Influence of adsorbate size on the adsorption of alcohols.

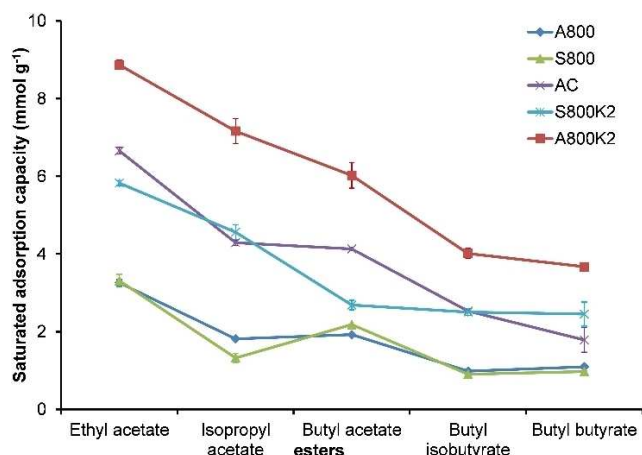


Figure 4. Influence of adsorbate size on the adsorption of esters.

enter the pore. In contrast, for S800 and A800, the adsorption will largely be occurring into mesopores (diameters 2–50 nm^[24]) or onto the surface of the adsorbent where the size of the VOC will be less important. A plot of saturated adsorption capacity on A800K2 versus molar volume of the VOC was also constructed (data in Supporting Information) and showed that the saturated adsorption capacity decreased as the molar volume of the VOC increased. This indicates that size is more important than functionality in determining the saturated adsorption capacity.

Adsorbates of similar size were then used to investigate the effect of functional groups within the VOC on its saturated adsorption capacity onto A800K2. As a measure of molecular size, the sum of the number of carbon, nitrogen, and oxygen atoms (S_{CNO}) was used. The chlorinated solvents and petroleum ether were omitted from this analysis so that all of the VOCs included were single molecular species composed of only carbon, nitrogen, oxygen, and hydrogen. Figure 5 shows the resulting plot of saturated adsorption capacity onto A800K2 versus S_{CNO} of the VOC where $S_{\text{CNO}}=4$ to 7. This range was

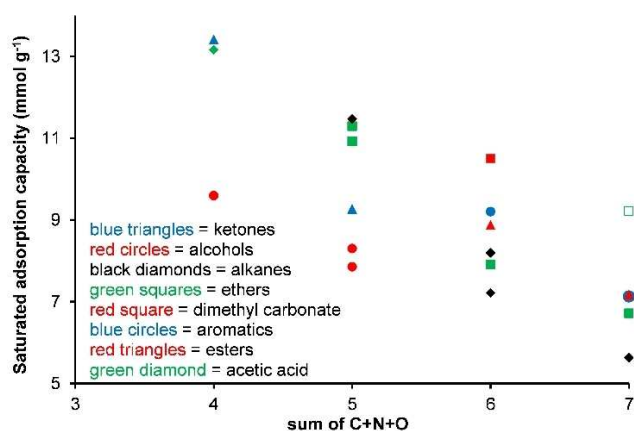


Figure 5. Influence of functional group on the adsorption of VOCs of similar size onto A800K2.

chosen as all the examples in Table 2 with $S_{\text{CNO}} > 7$ are esters, only one VOC in Table 2 (methanol) has $S_{\text{CNO}}=2$ and only two VOCs in Table 2 (ethanol and acetonitrile) have $S_{\text{CNO}}=3$ so these did not give useful information. Each of the four columns in Figure 5, then ranks VOCs of similar size according to their saturated adsorption capacities and allows the effect of functional groups to be elucidated as: dimethyl carbonate > aromatic \approx ester > ether \approx alkane > ketone \approx acetic acid > alcohol. It is notable that alcohols are the least preferred adsorbates, yet methanol has the highest saturated adsorption capacity of any of the VOCs in Figure 1. This is entirely due to the small size of methanol ($S_{\text{CNO}}=2$) which outweighs its unfavourable functional group. The most favourable functional groups are those with delocalisable π -electrons which can interact with the π -electrons present in the polycyclic aromatic rings present in Starbons[®] produced at 800 °C.^[12] Non-polar functional groups are the next most favoured, followed by polar and protic functionalities. This reflects the non-polar and hydrophobic nature of the Starbon materials used. Only one VOC has a saturated adsorption capacity which does not fit the expected value based on its functionality. This is cyclopentyl methyl ether shown by the unfilled green square in Figure 5. The high saturated adsorption capacity of this ether is more consistent with it having $S_{\text{CNO}}=5.5$ rather than 7. This suggests that the interaction between cyclopentyl methyl ether and A800K2 primarily involves the cyclopentyl ring of cyclopentyl methyl ether.

S800K2 and A800K2 were selected for this study on the basis of their carbon dioxide adsorption^[22] which is comparable in size to the smallest VOCs used in this work ($C+N+O=3$). Therefore, use of two mass equivalents of potassium hydroxide to activate A300 might not be optimal for the larger VOC's. To investigate this, A300 was activated with 1–5 mass equivalents of potassium hydroxide to give A800K1–5. The surface areas and porosities of these materials are given in Table 1. Diisopropyl ether was selected as one of the larger VOC's ($C+N+O=7$) and Figure 6 shows its saturated adsorption capacity on each of A800K1–5. There was relatively little difference between the five adsorbents with all saturated adsorption capacities being in the range of 6.3–7.1 mmol g⁻¹, but the highest value was for

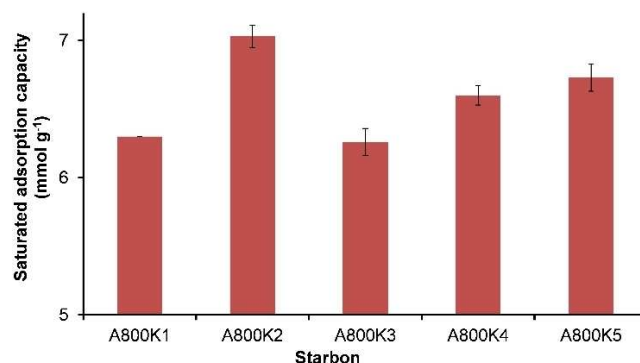


Figure 6. Influence of KOH mass equivalents used to activate A300 on diisopropyl ether adsorption. Data points are the average of three measurements with error bars showing \pm one standard deviation.

A800K2. This suggests that activation of A300 with two mass equivalents of potassium hydroxide will give the optimal material for the physisorption of all the VOCs studied.

The adsorption of VOCs by the nine adsorbents correlates well with their micropore volumes (Table 1). In addition, the dependence of the saturated adsorption capacities on the size of the adsorbate (Figures 2–4) indicates that the VOC is being adsorbed within the micropore structure and not just on the surface of the adsorbent.

Since the adsorption of adsorbates by Starbons® is a physisorption process, the adsorbate is only weakly bound to the Starbon. This allowed the Starbon to be regenerated and reused by heating the VOC saturated Starbon at 80 °C in a vacuum oven for 18 h. Regenerated adsorbent displayed the same saturated adsorption capacity for toluene as freshly prepared adsorbent and could also be used to adsorb a different VOC.

Conclusion

Starbons® and especially activated Starbons® are shown to be effective adsorbents for a range of 29 volatile organic compounds (VOCs). In contrast to adsorption of carbon dioxide where starch derived, activated Starbon (S800K2) was the most effective adsorbent, alginic acid derived Starbon (A800K2) is shown to be the most effective adsorbent for all 29 VOCs and this correlates with the higher micropore volume present in A800K2. The saturated adsorption capacities of VOCs containing the same functional group decrease as the size of the VOC increases, as would be expected for physisorption predominantly within micropores. The influence of the functional group within the VOC on its physisorption onto A800K2 was elucidated and the most favourable functional groups were those with delocalisable π -electrons which can interact with the π -electrons present in the polycyclic aromatic rings present in the activated Starbon.

The nine carbonaceous materials used in this study are representative of carbon based adsorbents as a whole as they possess a wide range of structural characteristics (Table 1). Their BET surface areas vary between 485 and 1706 m²g⁻¹, their micropore volumes vary from 0.16 to 0.49 cm³g⁻¹; their mesopore volumes vary from 0.07 to 0.40 cm³g⁻¹ and their total pore volumes vary from 0.25 to 0.56 cm³g⁻¹. The most effective adsorbent for all of the VOCs studied (A800K2) had the equal highest micropore volume and the lowest mesopore volume, indicating that these parameters rather than surface area are important in designing adsorbents for VOCs.

Experimental Section

S800 and A800 are commercially available. Activated Starbon materials were prepared as previously reported.^[22]

Determination of saturated adsorption capacities

A carbon-based adsorbent (100 mg) was added to each of nine sample tubes with the same adsorbent being added to each of three tubes. The filled tubes were fitted with a cap and weighed. A beaker containing a VOC (300 mL) was placed in the centre of a desiccator and the nine sample tubes (with lids removed) were positioned around the beaker. The desiccator was sealed and left at 21 °C for 21 h (unless the VOC was one of those specified in Figure 1, in which case the sealed desiccator was left for 67 h). After the specified time, the desiccator was opened, and each tube was quickly fitted with its cap. The tubes were then weighed to determine the mass of VOC adsorbed by the carbon-based adsorbent, from which the saturated adsorption capacity was calculated.

Regeneration of adsorbent

Used adsorbent was placed in a vacuum oven and heated at 80 °C for 18 h to remove all adsorbed solvent. After cooling to room temperature, the regenerated adsorbent could be used to adsorb another VOC. This process could be repeated at least 30 times.

Conflict of Interests

The authors declare no conflict of interest. <

Data Availability Statement

The data that support the findings of this study are available in the supplementary material of this article.

Keywords: volatile organic compound · biomass derived · Starbon · hierarchically porous · adsorbent

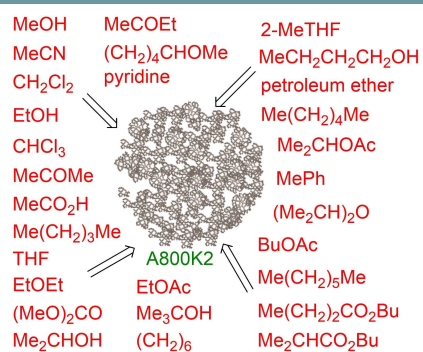
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1 – 7

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