

FILTERS FOR ACETONE REMOVAL FROM AIR STREAM AT PPM AND PPB LEVELS

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Abstract

The presence of volatile carbonyl containing contaminants, such as acetone, in indoor and cabin air environments is of great concern due to their hazardous health effects. The removal of acetone at ppm and ppb levels is still a challenge to many researchers around the world. Several samples of zeolites and activated carbons adsorbents of various origins were used in this study. The initial samples were modified by impregnation and chemical modifications using several approaches. The surface of the carbon samples was characterized using sorption of nitrogen and Boehm titrations. Acetone breakthrough curves were collected at typical high purity concentrations using flame ionization detectors (FID). The curves were converted into efficiency as well as capacity curves where the total capacity of each adsorbent was calculated. Activated carbons showed higher efficiency than zeolites for acetone. The volume of small micropores ($<10\text{\AA}$) determined the capacity of adsorbents toward acetone removal where the adsorption potential is the strongest. Although impregnants were expected to enhance acetone uptake, they showed the reverse effect. By modifying the surface chemistry, enhanced uptake was achieved by increasing the hydrogen bonding characteristics of the adsorbent surface.

Introduction

Adsorption of volatile organic contaminants (VOC) from the environment is of great importance to the scientific community even at trace levels due to their effects on the ecosystem, their noxious odor and toxicity, and their potential health impacts. In recent years, growing concern over the harmful effects of various VOC has led government agencies to set firm regulations for clean air. Acetone is considered to be an undesirable VOC molecule. Although a significant amount of work has been reported in the literature studying acetone removal, very little addresses its adsorption on activated carbons and zeolites under high flow dynamic conditions (Cal et al, 1997; Journet et al 2005; Kwon, 2002; Mangun et al, 1999; Mangun et al, 1999; Panov et al, 1998; Pires et al, 2003; Ramirez et al, 2005; Zaki et al, 2000; Madey et al, 1988).

Adsorption phenomena have been used for a long time to perform various separation or purification processes. The success or failure of an adsorption process depends on how the solid performs in both equilibrium and kinetic conditions (Duong, 1994). The first controls the adsorptive capacity while the second is related to the time required for an adsorbate molecule to be transported to specific adsorption sites or pores.

Removal of pollutants and contaminants from air and water on activated carbons and zeolites is a well-known method used to clean the environment and prevent its pollution (Abassi and Streat, 1994; Bansal, 1988; Gregg et al, 1982; Puri, 1970). At present, activated carbon is one of the most promising solid adsorbents used to remove such compounds, owing to its commercial availability, high adsorptive capacity, surface chemistry and structural features including a high surface area and the presence of small pores, similar in sizes to the molecules to be removed (Abassi and Streat, 1994; Bansal, 1988; Puri, 1970). In order to study the adsorption of acetone on activated carbons, characterizations of surface chemistry and structural parameters (pore size distribution, pore volume and surface area) of the activated carbon sorbents is of importance.

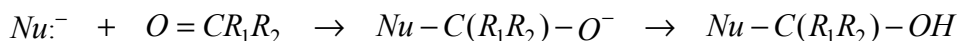
The goal of this work is to study the performance and efficiency of various activated carbons and zeolites for the adsorption of acetone from an air stream. A comparison is done with our standard activated carbon for such application. In order to provide optimum chemical filters for acetone removal, it is very important to understand the mechanism by which it is adsorbed. In this paper, the effect of chemical modification and impregnation for the removal of acetone is investigated.

Theoretical

Both surface chemistry and pore structure of adsorbents are expected to play a role in the adsorption process. It is believed that acetone can be adsorbed through several dispersive and specific (selective) interactions with the adsorbent surface. The presence of small pores similar in size to acetone molecule is expected to enhance the dispersive interactions while polar and nucleophilic groups aid specific interactions.

For selective chemical adsorption of acetone, understanding the surface chemistry of adsorbents is of great importance. It is understood that the chemical nature of the activated carbon surface is heterogeneous and more complex than its pore network. This heterogeneity results from the presence of heteroatoms other than carbon that exist in the form of organic functional groups at the edges of the graphene layers in the activated carbon matrix (Boehm, 1966). The origin of these heteroatoms is in the nature of the organic precursors (nitrogen, oxygen and sulfur), in the chemistry of activation processes (oxygen and phosphorus), in the storage conditions (oxygen), and in further modification processes (oxygen and nitrogen) (Leon and Radovic, 1992). The presence of such functional groups on the surface is the primary contributor to specific interactions with acetone. Such interactions are usually in the form of dipole-dipole and hydrogen bonding. On the other hand, for adsorption under humid conditions, these groups are known to strongly adsorb water molecules present in the air stream. Thus competition for the polar adsorption sites exists and the results indicate that water is the winner. Those water molecules adsorbed on modified samples block the pore entrances and thus limit the accessibility of acetone to small pores where it can be strongly adsorbed via dispersive forces.

Moreover, acetone can be adsorbed via nucleophilic attack-based reactions. Hence, these types of reactions have typically been used for the removal of carbonyl containing VOCs. Ketones and aldehydes are expected to react with nucleophiles in presence of slightly acidic medium. The nucleophilic reaction is best described as follows:



Where Nu: can be sulfanilic acid, amine, water, alcohol, etc. R1 is an alkyl group and R2 can be either a hydrogen atom (aldehyde) or an alkyl group (ketone).

Applying this chemical approach, Hayashi and co-workers (Hayashi et al, 2005) studied acetaldehyde adsorption capacities on activated carbons impregnated with various amines. They found that the adsorption capacity of impregnated activated carbons is influenced by the steric structure of impregnants as well as their acidity. The activated carbons impregnated with sulfanilic acid showed different adsorption mechanism for acetaldehyde than those impregnated with other amines. The adsorption capacities of activated carbons for acetaldehyde increased after impregnation with Sulfanilic acid.

Although sulfanilic acid is assumed to be a good nucleophile, steric hindrance may arise preventing the initiation of nucleophilic reaction with carbonyl containing compounds. Additional problem with this compound will be blocking some of the micropores and hence decreasing the amount of acetone being adsorbed via dispersive interaction. However, there are surface modification steps that have the potential to avoid these issues. For example, impregnation of activated carbon with urea followed by heat treatment at high temperature can eliminate those problems by introducing amine groups into the edges of the graphene layers in the form of pyridine and pyrrole.

In this paper, the role of surface chemistry and porosity on the removal of acetone is investigated. Amines are introduced at the edges of the graphene layers of activated carbons by urea modification into the surface and by impregnation with sulfanilic acid and amino compounds. The effect of different chemical modification and impregnations for the removal of acetone on activated carbons is studied under dynamic conditions.

Experimental

1. Materials

Eight activated carbon samples of different origins and methods of activation were chosen for this study in addition to three zeolite samples. STD is a standard activated carbon for acetone removal. The other seven activated carbons are named as G, H, I, J, K, L and M. These materials were selected based on their diversity in surface chemical and structural properties or because they have been used in the past in similar applications. Zeolite samples are referred to as X, Y and Z. The latter two are potassium permanganate impregnated zeolites. The initial carbons were modified using several methods which are given in detail below.

1.1 Urea Modification and Thermal Modification

One group of carbons STD, G, H and I were impregnated with urea (saturated solution). The suspension of activated carbon and urea solution were stirred overnight and then heated at 1223 K for 1 hour in nitrogen atmosphere at the rate of 10 K/min in order to introduce nitrogen groups (Elsayed and Bandosz, 2002). For comparison purposes, new carbon samples of STD, H and I were thermally modified by just heating at 1223 K for 1 hour in nitrogen atmosphere at a rate of 10 K/min. After modification, the samples were washed in a soxhlet apparatus to remove any excess of urea products and any water-soluble compounds. After this process the samples are referred to as STD-UT, G-UT, H-UT, I-UT, STD-T, H-T and I-T. UT and T represent urea modified and thermally modified respectively.

1.2 Impregnation with 1% *p*-Aniline Sulfonic Acid and 2.5% Amino Compound Solutions

Activated carbon samples STD and H were mixed in volume proportions with 1% *p*-anilinesulfonic acid (sulfanilic acid) solution. Another sample of each carbon was mixed with 2.5% of amino compound solution. The suspensions were shaken so as to allow for homogeneous mixing followed by drying at 393K. After impregnation the samples are referred to as STD-S and H-S, STD-N and H-N. S and N indicate *p*-anilinesulfonic acid and amino compound impregnation, respectively.

2. Study of Surface Chemistry of Carbon

2.1 Boehm Titration

One gram of carbon sample was added to each of the 50 ml of 0.05N solutions: sodium hydroxide, sodium carbonate, sodium bicarbonate and hydrochloric acid. The vials were sealed and shaken for 24 h. The samples were filtered. 10 mL of each filtrate was pipetted out and the excess of base or acid was titrated with 0.1N HCl or NaOH, respectively. The number of acidic sites of various types were calculated under the assumption that NaOH neutralizes carboxyl, phenolic, and lactonic groups; Na₂CO₃ carboxyl and lactonic; and NaHCO₃ only carboxyl groups (Boehm, 1966; Elsayed and Bandosz, 2002). The total number of acidic and basic groups is calculated based on the amounts of sodium hydroxide and hydrochloric acid reacted.

2.2 pH Measurement

0.4 g of dry carbon powder was added to 20 ml of water and the suspension was stirred overnight to reach equilibrium. The samples were filtered and the pH of the filtrates was measured.

3. Characterization of the Pore Structure

3.1 Sorption of Nitrogen

An ASAP 2010 (Micromeritics, Norcross, GA, USA) was used to measure nitrogen adsorption isotherms at 77 K. Before the experiment, the samples were heated at 333 K and outgassed under a vacuum to constant pressure of 10⁻⁵ torr. Characterization of pore sizes and pore structure were accomplished using Density Functional Theory (DFT) (Lastokie et al 1993). DFT works under the assumption that the experimental isotherm can be expressed as the sum of the convolution of a kernel function which represents the isotherm of an ideal homoporous adsorbent with a frequency distribution of pore sizes. The convolution is done by fitting the experimental isotherms into combinations of pre-existing isotherms for various pore sizes until the best fit is obtained. Using DFT, specific surface areas, total pore volume, micropore volume, volume of pores less than 10Å ($V_{<10\text{\AA}}$) were calculated (Lastokie et al 1993). Moreover, the surface areas, SBET, were also calculated using the BET method.

4. Adsorption of Acetone

4.1 Break through Measurements

Acetone breakthrough tests were carried out under dynamic conditions on all carbons and zeolites at low concentration using packed beds, 1 inch in depth (Dallas et al, 2005). The service life of the activated carbon and zeolite filters was predicted from the adsorbate breakthrough time and the test methods relative to the source application. The experiments were carried out at room temperature with acetone inlet concentration of 50 ppmv. A 10% breakthrough concentration of 5 ppmv was chosen for material performance comparisons. The air flow was adjusted to 30 L/min with a relative humidity of 50%. The bed diameter was 1.5 inches while the activate carbon particle diameter is 12X30 mesh. FID detector was used to monitor acetone concentration.

4.2 Capacity Curves

From the breakthrough curves, the capacity of all activated carbons and zeolites for acetone can be calculated using the following equation:

$$q = \sum_{i=1}^t \frac{(C_s - C_o) * F}{m}$$

Where q is the capacity of activated carbons and zeolites in (g acetone/g of adsorbent), C_s is the outlet concentration (g/L) at the end of breakthrough curve, C_o the outlet concentration (g/L) at time i , F is the flow rate (L/min) and m is the mass (g) of the activated carbon or zeolite sample in the packed bed. When the outlet concentration reaches the challenge concentration (C_s), the total capacity can be calculated and is referred to as W_c . At this point, W_c is equal to q.

Results and Discussion

The results obtained from Boehm titrations for initial carbons along with their surface pH values are presented in Table 1. The data reported indicates that the initial carbons differ in their acidity as a result of the activation method and the organic precursor (Bansal et al, 1988). The total number of groups and their distributions within various categories result in different values of the surface pH. Although STD, G, H and K carbons have average surface pH values close to neutral, carbon G is the most acidic. In the case of STD, H and K carbons, while the total numbers of groups are similar, the distributions of species are different. Carbons H and K contain more carboxylic and phenolic groups than STD carbon. The presence of large amount of basic groups on the surface of carbons I, J, L and M is reflected in their higher pH values located in the basic region. Even though carbon M has larger amount of basic groups than carbon J, carbon J has a higher pH value indicating the presence of stronger basic groups on its surface.

Table 1. Results of Boehm Titration (mmol/g).

Sample	pH	Carboxylic	Lactonic	Phenolic	Acidic	Basic
STD	7.56	0.0250	0.0754	0.1504	0.2508	0.3813
G	6.55	0.2550	0.1400	0.3670	0.7630	0.3630
H	7.29	0.1003	0.0252	0.2759	0.4014	0.3813
I	9.44	0.0000	0.0000	0.2006	0.2006	0.5338
J	10.8	0.0000	0.0000	0.0000	0.0000	0.7881
K	7.12	0.1254	0.0252	0.3010	0.4516	0.3287
L	8.56	0.0000	0.0000	0.2006	0.2006	0.5836
M	10.2	0.0000	0.0250	0.1254	0.1504	1.1938

Surface structure is another important parameter in studying the adsorption on activated carbon when trace concentrations of small molecules, such as acetone, are to be removed. The carbons used in this study are of different origins. They are also prepared by different activation methods and thus expected to have different pore structure. The structural parameters such as surface area, SBET, and the volume of pores smaller than 10 Å ($V_{<10\text{Å}}$) were calculated from the nitrogen isotherms for the initial and modified activated carbons, as well as zeolites. These results are summarized in Table 2.

Table 2. Structural Parameters Calculated from Nitrogen Adsorption at 77 K

Sample	$V_{<10\text{Å}}$ (cm^3/g)	V_t (cm^3/g)	S_{BET} (m^2/g)	Sample	$V_{<10\text{Å}}$ (cm^3/g)	V_t (cm^3/g)	S_{BET} (m^2/g)
STD	0.261	0.371	866	H-S	0.183	0.371	547
STD-UT	0.259	0.358	865	I	0.263	0.383	1064
STD-T	0.259	0.397	982	I-UT	0.259	0.372	924
STD-N	0.225	0.330	821	I-T	0.279	0.431	1090
STD-S	0.240	0.343	863	J	0.159	0.242	533
G	0.118	1.196	1216	K	0.118	0.322	747
G-UT	0.194	0.852	1116	L	0.235	0.542	873
H	0.197	0.396	761	M	0.148	0.455	934
H-UT	0.195	0.388	751	X	0.067	0.207	309
H-T	0.195	0.354	741	Y	0.000	0.058	23
H-N	0.186	0.343	545	Z	0.043	0.183	199

It is clearly seen that zeolite samples have lower surface area and pore volumes than activated carbons. Carbon G has the highest surface area. This is due to the largest pore volume. In the case of carbon J these parameters are the smallest. Urea modification showed no impact on the volumes of pores for STD and H carbons while for carbons G and I, a slight decrease is observed. It is important to mention that the volume of very small pores ($<10 \text{ Å}$) showed a noticeable increase on carbon G after urea modification. Thermal modification revealed no significant change in the porous structure, mainly for micropores. A slight increase in the volume of mesopores as well in the surface area for STD and I carbon samples is observed. Impregnation of STD and H carbons with sulfanilic acid and amino compounds resulted in a decrease in the total surface area for both activated carbons. This decrease is more pronounced for Carbon I indicating that more pores is blocked with impregnates.

Acetone breakthrough curves obtained at 50% RH are presented in Figure 1. The results indicate that STD activated carbon has the best efficiency for removing acetone followed by carbons H, I and L respectively. Zeolite X showed good performance; however, it is not as good as that of STD sample. The effect of potassium permanganate, a strong oxidant, was also studied. It is expected to oxidize acetone into carbon dioxide and water. Zeolites Y and Z, impregnated commercially with potassium permanganate, were tested. Unfortunately, they showed no efficiency for acetone removal indicating the proposed oxidation reaction is not expected to take place under the applied experimental conditions.

The breakthrough curves in Figure 1 give a good general evaluation of the different samples tested and their ability to remove acetone; however, it is worth to mention that the tests were done with a fixed bed volume of each sample rather than fixed sample weight. Since the capacity depends on the weight of the sample, the breakthrough curves were normalized to weight and converted into capacity curves shown in Figure 2.

It is evident that the capacity curves show slightly different trends than the one observed from breakthrough curves. The STD sample has the highest capacity for acetone followed by sample J, L and I, respectively. All zeolite samples, mainly Y and Z, showed very low capacity for acetone.

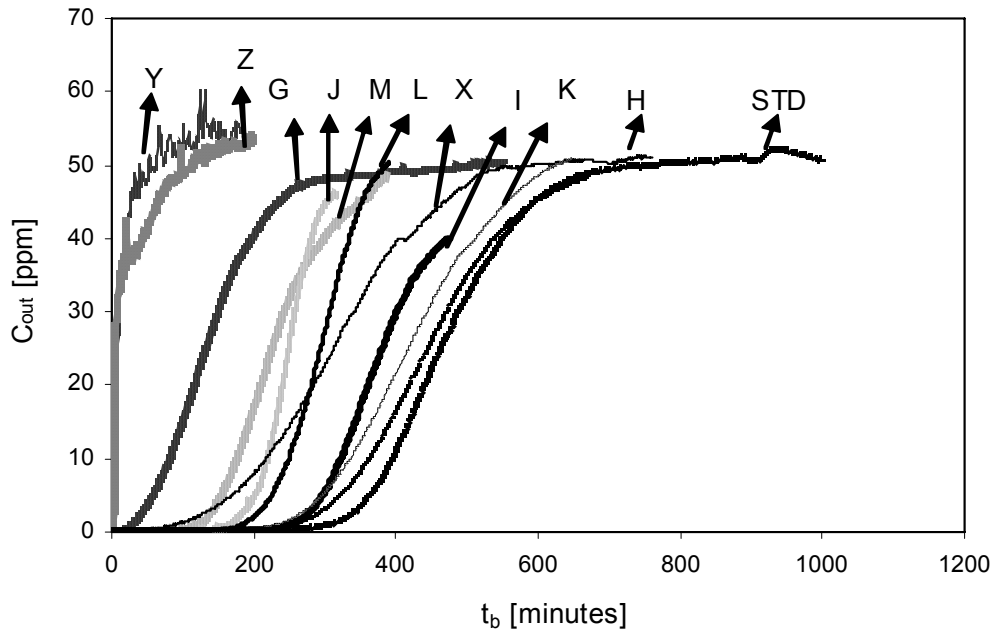


Figure 1. Acetone breakthrough curves on unmodified activated carbon and zeolite samples.

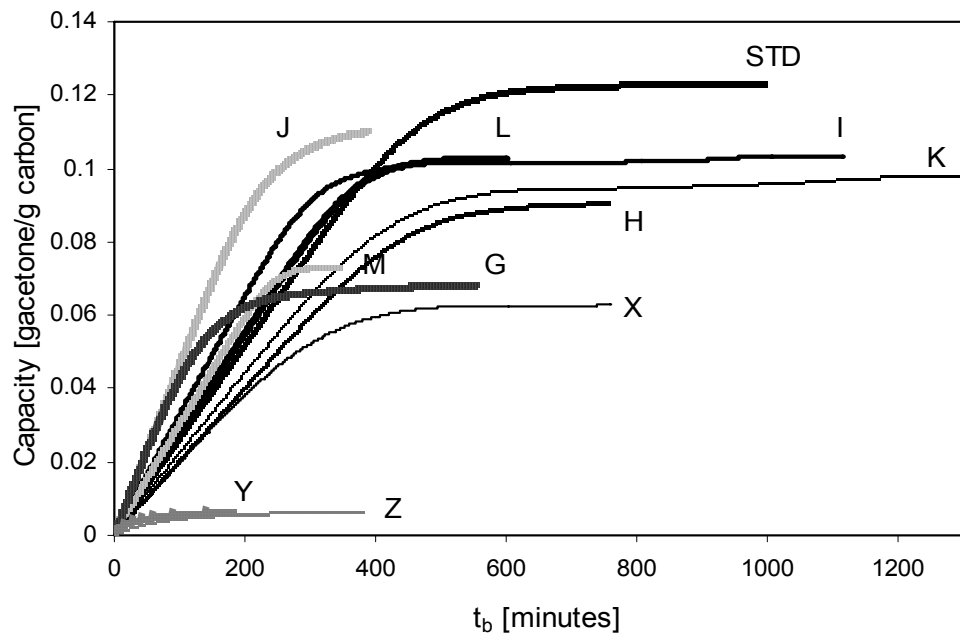


Figure 2. Acetone capacity curves on unmodified activated carbon and zeolite samples.

In an attempt to improve the removal ability of these adsorbents and increase their capacity for acetone, several activated carbon samples were further modified by urea and thermal modification and impregnation with sulfanilic acid and amino compounds as described in the experimental section. The calculated capacities in grams of acetone per gram of adsorbents, along with the 10% breakthrough time, for all modified and initial samples are collected in Table 3.

Table 3. Breakthrough times (10% t_b) and capacities for acetone (W_c) on initial and modified carbons and zeolites.

Sample	10% t_b (min)	Total Capacity, W_c (g/g)	Sample	10% t_b (min)	Total Capacity, W_c (g/g)
STD	360	0.1227	H-S	199	0.0873
STD-UT	274	0.1343	I	300	0.1027
STD-T	281	0.0956	I-UT	312	0.1087
STD-N	263	0.0912	I-T	283	0.0975
STD-S	310	0.1052	J	155	0.1101
G	56	0.0680	K	300	0.1027
G-UT	103	0.0743	L	301	0.0961
H	326	0.0919	M	202	0.0728
H-UT	274	0.1069	X	175	0.0625
H-T	236	0.0952	Y	2	0.0064
H-N	274	0.0949	Z	3	0.0064

Comparison of the results presented in Table 3 demonstrates that STD has the highest breakthrough time as well as capacity for acetone compared to the other activated carbons and zeolites. The effect of each type of modification is also shown. Impregnation of the best performing activated carbons, STD and H, with amino compounds and sulfanilic acid, decreased the breakthrough times and showed no enhancing impact on the overall capacity. This decrease is more likely due to pore blocking resulting in a decrease in the surface area available for acetone adsorption. This is supported by the decrease in the volume of pores smaller than 10Å mainly on the STD sample. Moreover, both impregnation molecules used are large in size. Their chemical reaction with acetone will be sterically hindered hence preventing the initiation of the nucleophilic reaction (Hayashi et al, 2005). This leads us to conclude that impregnation of the DCI sample with amino compounds and sulfanilic acid may not be the best choice for enhancing its efficiency toward acetone removal.

Urea modification of STD, G, H and I samples followed by heat treatment at 1223K, significantly increased their capacity for acetone. As discussed before, urea modification is expected to introduce nitrogen groups into the surface usually in the form of amine, pyrrole and pyridine. The increase in capacity can either be contributed to the nitrogen groups introduced into the surface or to the generation of small pores similar in size to acetone as a result of thermal treatment. To check the effect of thermal treatment and for comparison purposes, STD, H and I samples were heated under the same experimental conditions used during urea modification. Thermal modification of these carbons resulted in a decrease in their capacities. Since the volume of very small pores is similar after both urea and thermal modification methods, the increase in capacity after urea modification is mainly attributed to the presence of nitrogen groups introduced into the surface which are expected to interact with acetone via specific interactions.

Correlating the volume of acetone adsorbed to the various surface chemical and structural parameters showed the importance of pores smaller than 10 Å in diameter in the adsorption process. Figure 3 shows an increasing trend between the volume of acetone adsorbed, calculated at 10% breakthrough time, and the volume of pores <10 Å. Such a trend indicates the predominant effect of those pores as the most energetic sites for adsorption of acetone. This leads to the conclusion that acetone is mainly adsorbed via dispersive interaction at the beginning of the adsorption process.

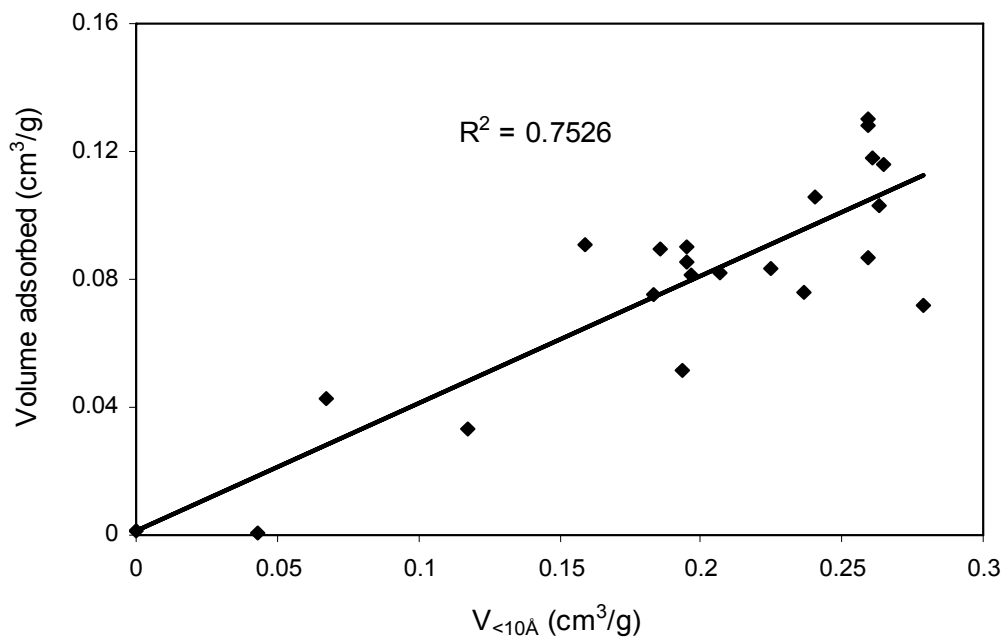


Figure 3. Dependence of the volume of acetone adsorbed at 10% breakthrough time on the volume of pores smaller than 10 Å.

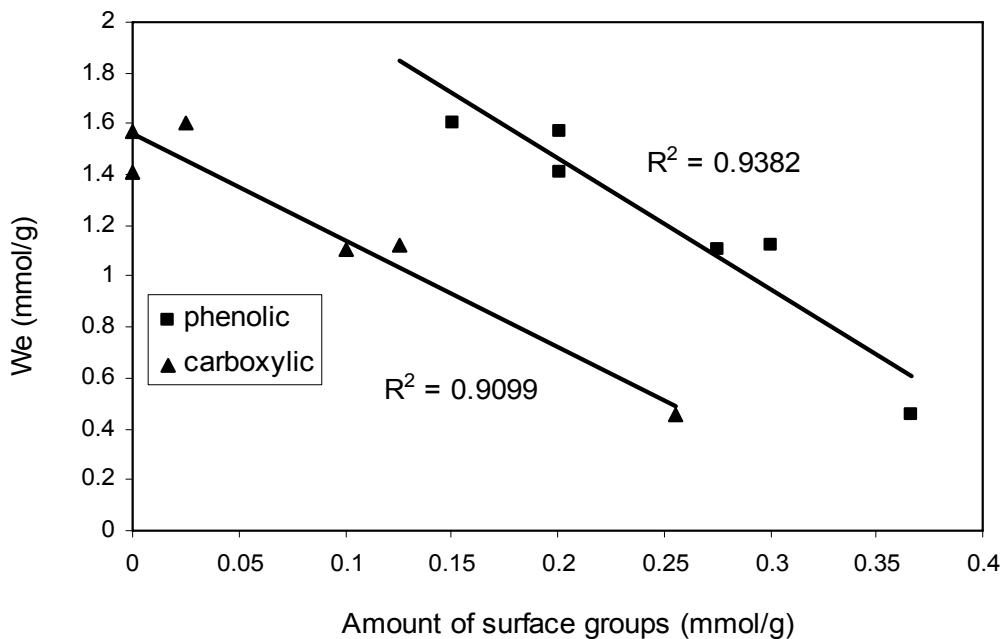


Figure 4. Dependence of the amount of acetone adsorbed on unmodified activated carbon (mmol/g) at 10% breakthrough time on the amount of surface carboxylic and phenolic groups (mmol/g).

It is also important to mention that the amount of acetone adsorbed on unmodified activated carbons at 10% breakthrough time decreased with the amount of surface carboxylic and lactonic groups (Figure 4). The presence of functional groups results in a more hydrophilic surface and hence weaker dispersive interactions with acetone. Moreover, the presence of polar groups provides active sites for water adsorption via hydrogen bonding. Water molecules adsorb more preferably on these sites than acetone. When the small pores are filled, acetone starts to adsorb on the surface via hydrogen bonding. Support for this is evidenced by an increase in the amount of acetone adsorbed at a higher surface coverage of protonated sites (see Figure 5).

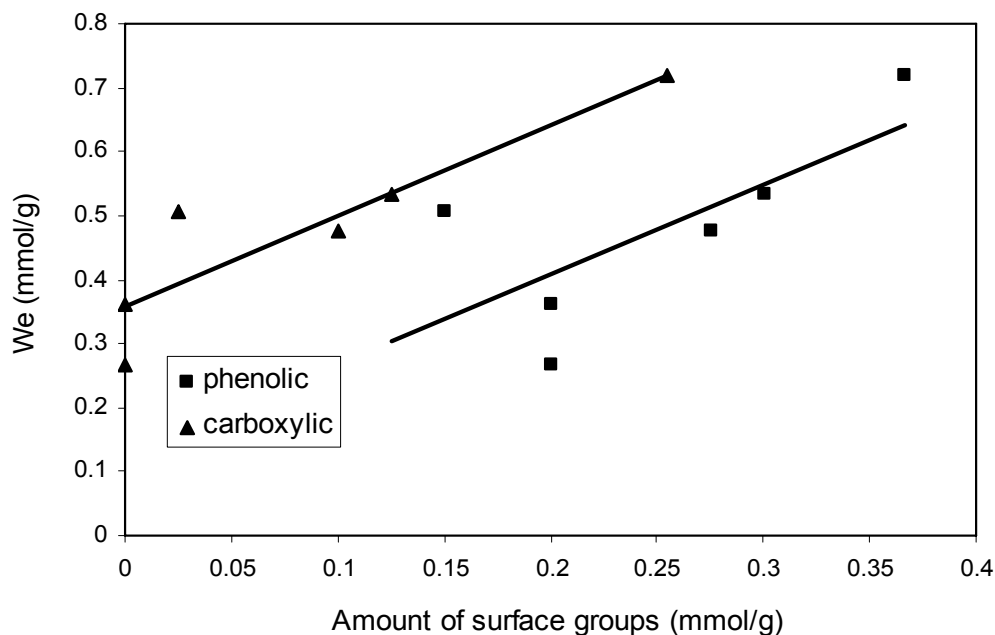


Figure 5. Dependence of the amount of acetone adsorbed on unmodified activated carbon (mmol/g) beyond 10% breakthrough time on the amount of surface carboxylic and phenolic groups (mmol/g).

Conclusions

The standard activated carbon, STD, proved to be the best available activated carbon for acetone removal compared to other commercial activated carbons. Zeolites showed no significant capacity for acetone under the conditions considered in this study even those impregnated with potassium permanganate. Impregnation of activated carbons with amino and sulfanilic acid compounds had no significant impact on improving their capacity for acetone. On the other hand, urea modification of activated carbons resulted in increased acetone capacity. This increase is attributed to the presence of nitrogen groups which were introduced into the surface by the modification process. These groups are expected to interact with acetone via specific interaction. Pores smaller than 10\AA were found to have the most profound impact on the capacity of adsorbents for acetone adsorption. Under the conditions of this study and prior to 10% breakthrough, these small pores govern acetone adsorption in the adsorbents evaluated here. For the surfaces evaluated here, acetone adsorption through specific interactions, mainly through hydrogen bonding with protonated polar surface sites, were observed to have only a minor influence over the 10% breakthrough time and acetone capacity.

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