Naphthalene Removal From Water by Novel Mesoporous Carbon Nitride Adsorbent

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In the present work, well ordered, mesoporous carbon nitride (MCN) sorbent with uniform mesoporous wall, high surface area and pore volume has been fabricated using the simple polymerization reaction between ethylene diamine and carbon tetrachloride in mesoporous silica media. The structural order and textural properties of nanostructured materials were studied by XRD, elemental analysis, and nitrogen Adsorption–desorption experiment. Adsorption of naphthalene over various porous adsorbents such as mesoporous carbon (MC), mesoporous carbon nitride (MCN), and amino modified mesoporous carbon (AMC) was studied from solutions with different concentration at ambient temperature (28 ± 2 °C) and pH 7.5 in polar (water) solvent. The adsorption isotherms of naphthalene were in agreement with a Langmuir model, moreover, the uptake capacity of naphthalene over MCN was much higher than amino modified mesoporous carbon (AMC) and pristine mesoporous carbon adsorbent.

Key words:
Naphthalene, mesoporous carbon nitride, adsorption, amino modification

Introduction

Polycyclic aromatics hydrocarbons are chemical species with two to six fused benzene rings and are well-known toxic hazardous pollutants and highly potent carcinogens that can cause tumors in some organisms.[1] In recent years, naphthalene contamination in water systems has drawn increasing attention. Naphthalene originates from natural and anthropogenic sources. Anthropogenic sources include engine exhaust, industrial processes, crude oil, urban run-off, domestic heating systems, incinerators and smoke. Natural sources include terrestrial coal deposits, volcanic eruptions and forest fires. The main sources of naphthalene in surface water are atmospheric deposition, runoff from contaminated soils and deposition from sewage discharges.[2] Most naphthalenes are hydrophobic with high boiling and melting points and electrochemical stability. Therefore, they can exist and be accumulated in soils or water for a long time.[3]

Adsorption treatment provides a simple and universal approach to effectively removing organic pollutants from the aquatic environment. The removal of toxic organic pollutants from water is a problem, particularly when they are present in low concentrations. Several studies have focused on the fate and transport of these pollutants and the application of remedial technologies to manage them.[4–10] Activated carbons present an outstanding adsorption capacity that stems from their high surface area, pore structure and surface chemical properties. These materials are effective adsorbents for priority pollutants, therefore being suitable for the decontamination of water and wastewater. These porous carbons are generally microporous and the preparation of carbon materials with well-ordered mesoporous structure would offer many application possibilities not only in the adsorption and in separation of large molecules whose molecular sizes are too large to enter micropores but also in electrical double layer capacitors, gas separation, catalysis, water and air purification and energy storage. Recently, Ryoo et al. prepared ordered mesoporous carbons from mesoporous silica templates such as MCM-48, SBA-1 and SBA-15 using sucrose as the carbon source.[11–14]

Adsorption plays an important role in these processes. Therefore, the interactions of such compounds with the mesoporous carbon surface must be studied in detail. The mesoporous carbon materials adsorption capacity depends on quite different factors. Obviously, it depends on the mesoporous carbon's characteristics: texture (surface area, pore size distributions), surface chemistry (surface functional groups).[15–17] It also depends on adsorptive characteristics: molecular weight, polarity, pK_a, mo-

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lecular size, and functional groups. The influence of the texture and surface chemistry of nanoporous carbons in the adsorption of organic compounds has been studied for years, and many references on this topic can be found in the literature. The adsorption of organic molecules from aqueous solutions on nanoporous carbons has been recently reviewed,[18,19] and it was shown that the specific mechanism by which the adsorption of many organic compounds takes place on this adsorbent is still ambiguous. Since the pioneer works of Coughlin and Ezra [20] and Mattson et al. [21] to other published more recently,[22–24] it was found that the adsorption capacity is significantly affected by the carbonaceous adsorbent surface chemistry.

Different studies have been done on interaction between surface functional groups and the adsorption of organic compounds in aqueous solution. There are two types of interactions between the adsorbate and the carbonaceous adsorbent: electrostatic and dispersive.[17–19] The former appears when the adsorptive is dissociated under the experimental conditions used; for the latter, three mechanisms are proposed: π–π dispersion interaction mechanism, the hydrogen bonding formation mechanism, and the electron donor–acceptor complex mechanism. The π–π dispersion interaction mechanism is the most widely accepted.[17,18] The surface chemistry and solution pH are the most important factors controlling the adsorption process.[25,26] Most aromatic pollutants are found in water solution in the molecular state for a broad range of pH values. In this case, dispersive interactions are predominant, mainly because of the attraction between the π orbitals on the carbon basal planes and the electronic density in the adsorbate aromatic rings (π–π interactions). However, when the solution pH is very high or very low, ions may be present, so electrostatic interaction between them and charged functional groups containing electronegative elements (like nitrogen) on the carbon surface could be significant.[17]

In this work, the influence of the texture and surface chemistry of nanoporous carbon adsorbent were systematically studied in the adsorption of naphthalene. For this purpose, mesoporous carbon nitride, amino modified mesoporous carbon and unmodified mesoporous carbon with quite different surface properties were selected in order to analyze the influence of the textural parameters. Interestingly, it has been found that the adsorption capability of MCN for naphthalene is much higher compared to that of other adsorbents. Langmuir and Freundlich adsorption isotherms were studied to explain the sorption mechanism.

Experimental

Materials

The reactants used in this study were tetraethyl orthosilicate (TEOS) as a silica source, cetyltrimethylammonium bromide (CTAB) as a surfactant, sodium hydroxide (NaOH), sodium fluoride (NaF), and deionized water for Synthesis of mesoporous silica (MCM-48), sucrose as a carbon source, and sulfuric acid as a catalyst for synthesis of mesoporous carbon, EDA (Ethylene Diamine) and CTC (carbon tetrachloride) for Synthesis of mesoporous silica (MCM-48), acetic anhydride, and concentrated sulfuric acid for amino modification of mesoporous carbon and naphthalene. All the chemicals were analytical grade and purchased from Merck.

Experimental procedure

Synthesis of mesoporous silica (MCM-48)

MCM-48 was prepared according to the synthesis procedure described by Yaofeng Shao.[24] In a representative synthesis, the MCM-48 molecular sieve was prepared as follows: 10 mL of tetraethyl orthosilicate (TEOS) was mixed with 50 mL of deionized water, and the mixture was vigorously stirred for 40 min at 35 °C, then 0.9 g of NaOH was added into mixture, and at the same time, 0.19 g of NaF was added into the mixture. After the NaF was added completely, the required content of sources, respectively, were added. After another 60 minutes of vigorous stirring, 10.61 g of cetyltrimethylammonium bromide (CTAB) was added to the mixture, and stirring continued for 60 minutes. The mixture was heated for 24 h at 393 K in an autoclave under static conditions, and the resulting product was filtered, washed with distilled water, and dried at 373 K. The as-synthesized samples were then calcined in air for 4 h at 550 °C, increasing the temperature to 550 °C at 1 °C min^{-1} of the heating rate.

Synthesis of mesoporous carbon (MC)

MC was prepared according to the synthesis procedure described by Riong Ryoo.[25] In a representative synthesis, the MC molecular sieve was prepared as follows: 1.25 g sucrose and 0.14 g H_2SO_4 in 5.0 g H_2O was dissolved, and this solution were added to 1 g MCM-48. The sucrose solution corresponded approximately to the maximum amount of sucrose and sulfuric acid that could be contained in the pores of 1 g MCM-48. The resultant mixture was dried in an oven at 373 K, and subsequently, the oven temperature was increased to 433 K. After 6 h at 433 K, the MCM-48 silica containing the partially carbonizing organic masses was added with an aqueous solution consisting of 0.75 g sucrose, 0.08 g H_2SO_4 and 5.0 g H_2O. The resultant mixture was dried again at 373 K, and subsequently the oven temperature was increased to 433 K. The color of the sample turned very dark brown or nearly black. This powder sample was heated to 1173 K under vacuum using a fused quartz reactor equipped with a fritted disk. The carbon ± silica composite thus obtained was washed with 1 M NaOH solution of 50 % ethanol- 50 % H_2O twice at 363 K, in order to dissolve the silica template completely. The carbon samples obtained after the silica removal were filtered, washed with ethanol and dried at 393 K.

Surface modification of mesoporous carbon

AMC was prepared using MC, according to Abe et al.[20] Briefly, Nitration was allowed to proceed in a 1000 mL three-neck flask containing MC, acetic anhydride, and concentrated sulfuric acid with dropwise addition of fuming nitric acid in 5 h while keeping the temperature below 5 °C . The reaction was completed after 19 h of stirring at room temperature. Modified MC thus obtained was thoroughly washed with deionized water until the electroconductivity of filtrate attained a value nearly the same as that of the water and vacuum-dried for 24 h at 110 °C. Reduction of the nitrated mesoporous carbon was permitted to proceed in a 1000 mL flask containing deionized water, 28 % aqueous ammonia, sodium hydrosulfite, and the carbon with stirring for 24 h in nitrogen atmosphere at room temperature. The aminated mesoporous carbon thus obtained was vacuum-dried at 110 °C after being washed with deionized water until the electroconductivity of filtrate became nearly the same as that of the water. This carbon sample is hereafter abbreviated as AMC.

Synthesis of mesoporous carbon nitride (MCN)

Mesoporous carbon nitride materials were prepared by using mesoporous silica MCM-48 as template. In a typical synthesis, 0.5 g of calcined MCM-48 was added to a mixture of EDA (1.35 g) and CTC (3 g). The resultant mixture was refluxed and stirred at 90 ºC for 6 hours. Then, the obtained dark brown colored solid mixture was placed in a drying oven for 12 hours, and ground into fine powder. The template-carbon nitride polymer composites were then heat treated in a nitrogen flow of 50 mL per minute at 600 °C with the heating rate of 3.0 °C min^{-1} and kept under these conditions for 5 h to carbonize the polymer. The mesoporous carbon nitride was recovered after dissolution of the silica framework in 5 wt % hydrofluoric acid, by filtration, washed several times with ethanol and dried at 100 °C.

Characterization

The X-ray powder diffraction patterns were recorded on a Philips 1830 diffractometer using Cu Ka radiation. The diffractograms were recorded in the 2θ range of 0.8–10 with a 2θ step size of 0.01θ and a step time of one second.

The adsorption-desorption isotherm of the as-synthesized sample was measured at 77K on micromeritics model ASAP 2010 sorptometer to determine an average pore diameter. Pore-size distribution was calculated by the Barrett-Joyner-Halenda (BJH) method while Brunauer-Emmett-Teller (BET) measured surface area of the sample.

Adsorption studies

A stock solution of 30 mg L^{-1} of naphthalene was prepared by dissolving an appropriate amount of naphthalene in ultra-pure water (18 MΩ cm) derived from a Milli-Q plus 185 water purifier. Batch adsorption isotherms were performed by shaking 500 mL amber Winchester bottles containing the required concentration of the naphthalene in a Gallenkamp incubator shaker. The shaker was set at a temperature of 25 ± 1 °C and a speed of 150 rotations per minute (rpm), corresponding to Reynold's numbers of 39400, which were estimated by using computational fluid dynamics modeling. Initial solution concentration of 0.02–0.14 mmol L^{-1} was prepared by pipetting out the required amounts of the naphthalene from the stock solution. The volume of solution in each bottle was maintained at 500 mL and the solutions were adjusted to pH 5 (pH values were measured by an Orion 720 A *pH-meter*). About 0.01 mg of each adsorbent was weighed accurately on aluminum foils using a Sartorious (Model BP 201D) analytical balance. The adsorbents were transferred carefully into the bottles using 50 mL solutions from the bottles. The bottles were shaken vigorously before shaking for different times in the incubator shaker (New Brunswick Scientific C25 Model).

The amount of naphthalene adsorbed was calculated by subtracting the amount found in the supernatant liquid after adsorption from the amount of naphthalene present before addition of the adsorbent by UV absorption at the λ_{max} of naphthalene, 275.5 nm. Calibration experiments were done separately before each set of measurements with naphthalene's solution of different concentrations. Centrifugation prior to the analysis was used to avoid potential interference from suspended scattering particles in the UV–Vis analysis.

The adsorption capacities were calculated based on the differences of the concentrations of solutes before and after the experiment according to the equation 1.

$$q_e = (C_o\text{-}C_e)\ V/W \qquad (1)$$

Where q_e is the concentration of the adsorbed solute (mmol g^{-1}); C_o and C_e are the initial and final (equilibrium) concentrations of the solute in solution (mmol L^{-1}); V (mL) is the volume of the solution and W (g) is the mass of the adsorbent.

Adsorption kinetics of naphthalene

For the measurement of the time resolved uptake of naphthalene onto adsorbents, 15 mL of solvent was mixed with 60 mg of MC in a 500 mL flask for about 10 minutes. 285 mL of Naphthalene solution was quickly introduced into the flask (keeping the initial concentrations of the resulting solutions at 30 ppm) and stirred continuously at 20 °C. Samplings were taken by fast filtration at different time intervals. The concentration of residual naphthalene in the solution was determined and the adsorption amount q(t) was calculated according to equation 2:

$$q(t) = (C(0)\text{-}C(t))V/W \qquad (2)$$

Where *q(t)* is the adsorption amount at time *t, C(0)* is the initial concentration of naphthalene solution, *C(t)* is the concentration of naphthalene solution at time *t and V* is the volume of naphthalene solution and *m* is the mass of MC, AMC or MCN.

Result and discussion

Characterization

Adsorption–desorption analysis

Besides PXRD nitrogen physisorption is the method of choice to gain knowledge about mesoporous materials. This method gives information on the specific surface area and the pore diameter. Calculating pore diameters of mesoporous materials using the BJH method is common.[26] Former studies show that the application of the BJH theory gives appropriate qualitative results, which allow a direct comparison of relative changes between different mesoporous materials.[27–30]

Fig. 1 shows nitrogen adsorption and desorption isotherms measured at 77 K by using a Micromeritics ASAP 2010 automatic analyzer. BET surface areas and the pore size determined by BJH (Barrett, Joyner, and Halenda) method for the synthesized mesoporous sorbents (MC, AMC and MCN) were determined by the adsorption branches of the isotherms. All mesoporous materials yield a type IV isotherm. The isotherm of MC is reversible and in good agreement with that previously reported. Table 1 summarizes the important physical properties of mesoporous sorbents. The surface areas of MC, AMC and MCN samples were 1432, 1321 and 1280 m^2 g^{-1}, and their average pore size were 4.6, 3.5 and 4.1 nm, respectively. This result mainly depends on the pore size and shape for all nanostructured carbon materials.

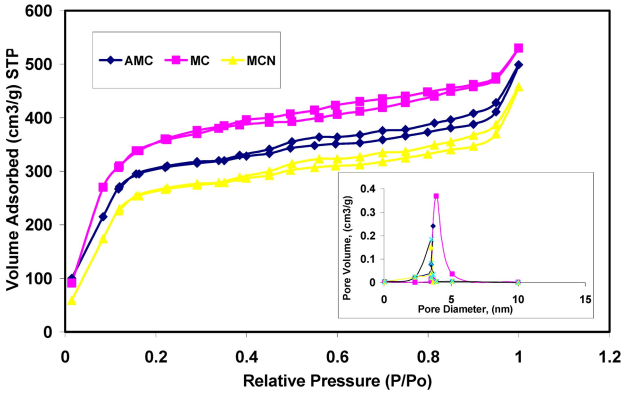

Fig. 1 – *Adsorption-desorption isotherms of nitrogen at 77 K on (a) MC, (b) AMC (c) MCN. The insert shows the BJH pore size distribution calculated from the desorption branch of the isotherm.*

Table 1 – *Textural parameters of the MC, AMC and MCN employed in this study*

Adsorbent	d spacing (nm)	A_{BET} (m^2 g^{-1})	V_p (cm^3 g^{-1})
MC	4.6	1432	0.74
AMC	3.5	1321	0.69
MCN	4.1	1280	0.72

XRD analysis

The XRD patterns of MC showed the two characteristic (211) and (220) reflections in the 2θ range from 0.8° to 10°, indicating a well-ordered cubic pore (Fig. 2). Like MC, the XRD patterns of AMC and MCN showed three diffraction peaks that can be indexed to (110), (210), and (220) in the 2θ range from 0.8° to 10°, representing well-ordered cubic

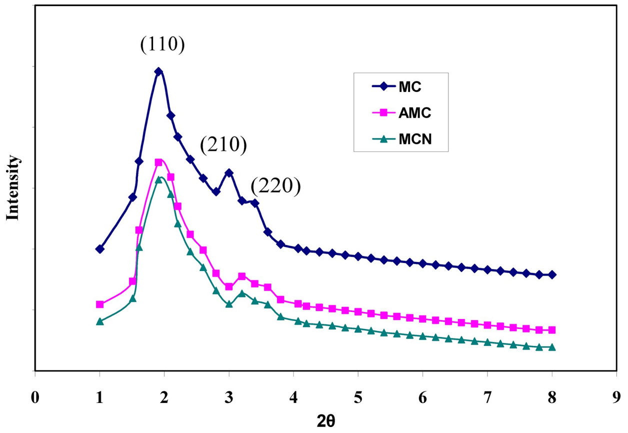

Fig. 2 – *XRD pattern of (a) MC (b) AMC (c) MCN*

I4132 pores. The observation data from the original samples of all nanostructured carbons are in good agreement with that previously reported.[25]

Elemental analysis

In Table 2 are shown the results of elemental analysis performed to check if nitrogen containing groups have really been introduced to the mesoporous carbon adsorbents. Since 2.1 % and 17.5 % of nitrogen was detected for AMC and MCN though no nitrogen was detected for MC, the results in the table demonstrate the presence of nitrogen-containing functional groups in both modified mesoporous carbon sorbents.

Table 2 – *Elemental Analyses of Mesoporous Carbons*

Sample	% C	% H	% N
MC	93.5	0.49	0
AMC	89.8	1.48	2.1
MCN	75.8	3.2	17.5

Adsorption studies

Effect of agitation speed

The effect of agitation speed on removal efficiency of naphthalene on mesoporous carbon sorbents (MC, AMC and MCN) was studied by varying the speed of agitation from 0 (without shaking) to 250 rpm, while keeping the analyte concentration, contact time, pH, temperature and dose of the adsorbent as constant. As it is demonstrated in Fig. 3, the naphthalene removal efficient generally increased with increasing agitation speed from 0 rpm to 250 rpm and the adsorption capacity of all adsorbents remained constant for agitation rates greater than 150 rpm. The mechanism of naphthalene removal from water involved four steps: i) migration of naphthalene molecules from the bulk solution to the surface of the adsorbent; ii) diffusion through the boundary layer to the surface of the adsorbent; iii) adsorption at a site; iv) intraparticle diffusion into the interior of the adsorbent. Increase in amount adsorbed with increase in agitation speed is due to change in the boundary layer resistance of the system. The boundary layer resistance will be affected by the rate of adsorption and increasing the degree of agitation will reduce this resistance and increase the mobility of the system, due to this the adsorbate molecules are forced towards the surface of the adsorbate. Similar results were obtained by McKay and Poots[31] and McKay.[32]

This also indicates that a shaking rate in the range 150–250 rpm is sufficient to assure that the maximum available sites of adsorbent existing in the pores of adsorbents are made readily available for naphthalene uptakes. For convenience, agitation speed of 150 rpm was selected as the optimum speed for all the adsorption experiments.

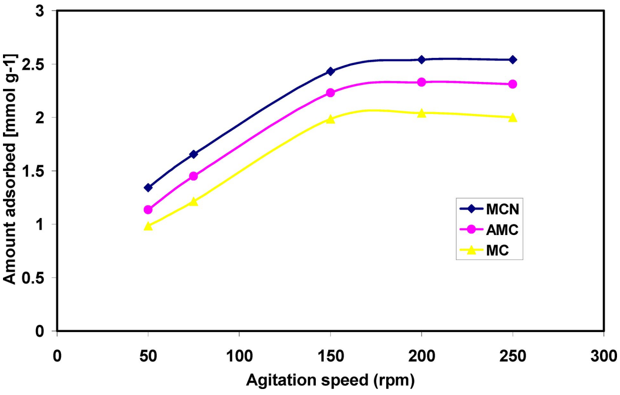

Fig. 3 – *Effect of agitation speed on the adsorption capacity of adsorbents (adsorbents dosage = 0.2 g L^{-1}, [naphthalene] = 30 ppm, contact time = 3 h, T = 298 K, solutions' volume = 100 mL and pH = 7)*

Effect of contact time and concentration

In order to establish equilibration time for maximum uptake and to know the kinetics of adsorption process, the adsorption of naphthalene on carbonaceous adsorbent was studied as a function of contact time and results are shown in Fig. 4. It is seen that the rate of uptake of the naphthalene is rapid in the beginning and 50 % adsorption is completed within an hour. Fig. 4 also indicates that the time required for equilibrium adsorption is 3 h. Thus, for all equilibrium adsorption studies, the equilibration period was kept 3 h. The effect of concentration on the equilibration time was also investigated on mesoporous carbon sorbent as a function of initial naphthalene concentration and the results are shown in Fig. 5. It was found that time of equilibrium as well as time required to achieve a definite fraction of

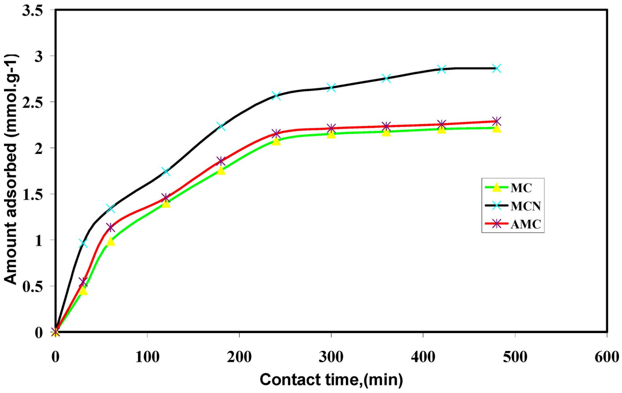

Fig. 4 – *Effect of contact time on removal of naphthalene ([naphthalene] = 30 mg L^{-1}, agitation speed = 150 (rpm), adsorbent dosage = 0.2 g L^{-1})*

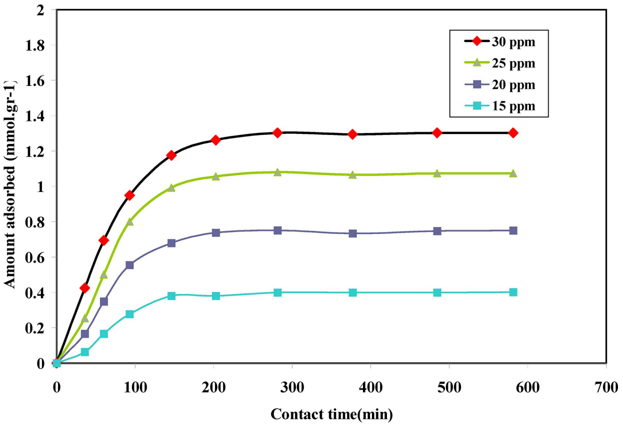

Fig. 5 – *Effect of initial concentration on removal of naphthalene (agitation speed = 150 (rpm), mesoporous carbon sorbent dosage = 0.2 g L^{-1}, room temperature = 25 + 1 °C)*

equilibrium adsorption is independent of initial concentration. These results indicate that the adsorption process is first order.

Adsorption isotherms

In order to evaluate the efficacy of the prepared adsorbents, the equilibrium adsorption of the naphthalene was studied as a function of equilibrium concentration. The adsorption isotherms of naphthalene on adsorbents are shown in Fig. 6. It is seen from Fig. 6 that order of adsorption in terms of amount adsorbed (mmol g^{-1}) on different adsorbents is: MCN >AMC>MC.

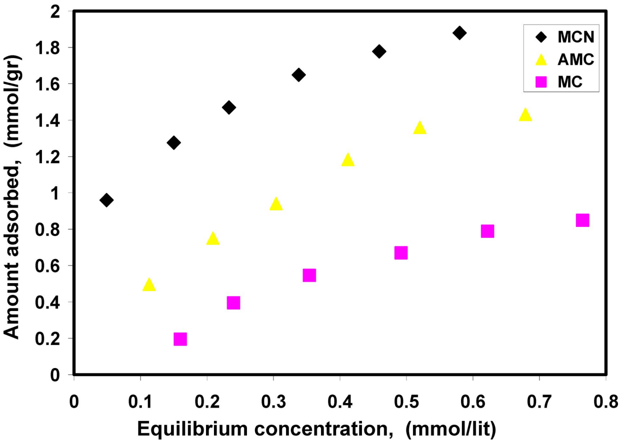

Fig. 6 – *Adsorption isotherm for naphthalene removal on adsorbents. (contact time = 4 h, agitation speed = 150 (rpm), adsorbent dosage = 0.2 g L^{-1})*

This appears due to nitrogen containing carbonaceous adsorbent, which has higher nitrogen content, and much better interaction with naphthalene as compared to pristine mesoporous carbon adsorbent tends to adsorb naphthalene in amounts appreciably larger. The performance of amino modified mesoporous carbon in the adsorption of naphthalene is lower than MCN because of the low content of nitrogen in mesopore surface and not very good molecular state of nitrogen in AMC.

In order to indicate the sorption behavior and estimate adsorption capacity, adsorption isotherms have been studied. The adsorption process of naphthalene was tested with Langmuir and Freundlich isotherm models. Two commonly used empirical adsorption models, Freundlich and Langmuir, which correspond to heterogeneous and homogeneous adsorbent surfaces, respectively, were employed in this study. The Freundlich model is given by the equation 3:

$$\ln qe = \ln K_f + (1/n) \ln Ce \tag{3}$$

Where k_f and n are the Freundlich constants related to adsorption capacity and intensity, respectively.[22,23] In the second model, the Langmuir equation assumes maximum adsorption occurs when the surface is covered by the adsorbate, because the number of identical sites on the surface is finite. The equation 4 (Langmuir equation) is given as follow:

$$C_e/q_e = (1/q_m b) + (1/q_m)\, C_e. \tag{4}$$

Where q_e (mmol g^{-1}) is the amount adsorbed at equilibrium concentration C_e (mmol L^{-1}), q_m (mmol g^{-1}) is the Langmuir constant representing maximum monolayer capacity and b is the Langmuir constant related to energy of adsorption.

The solid phase adsorbate concentrations of naphtalane ($q_{e,meas}$) were determined experimentally using Eq. (1). Theoretical solid phase adsorbate concentrations ($q_{e,calc}$) were calculated using Eqs. (3) and (4). The isotherm constants of each single component isotherm model were determined using the Sum of Squared Error (SSE) analysis. Isotherm constants in each model were optimised by minimising the values of the SSE function, which is represented mathematically as

$$SSE_{20} = \sum_{i=1}^{p} (q_{\mathbf{e,calc}} - q_{\mathbf{e,meas}})_i^2$$

Where p is the total number of equilibrium adsorption isotherm data points.

The isotherm data has linearized using the Langmuir equation. The regression constants are tabulated in Table 3. The high value of correlation coefficient ($R^2 = 0.99$) indicated good agreement between the parameters (C_e and Q_e). The constant q_m, which is a measure of the adsorption capacity to form a monolayer, and the constant b, which denotes adsorption energy are fitted by the Freundlich equation. The regression constants are listed in

Table 3 – *Isotherm constants for adsorption of naphthalene on MCN*

Langmuir constants				Freundlich Constants			
q_m (mmol g^{-1})	B (L $mmol^{-1}$)	R^2	*SSE*	K_F (mmol g^{-1})	*n* (L $mmol^{-1}$)	R^2	*SSE*
3.5263	43.159	0.9989	1.21	5.2328	0.725	0.921	1.52

Table 3. The value of correlation coefficients showed that the data conform well to the Langmuir equation. Moreover, based on the SSE values, the Langmuir isotherm model gives the lowest error value for naphthalene adsorption onto MCN.

Conclusion

In this work, mesoporous carbon nitride was utilized, which was prepared by cubic mesoporous silica MCM-48. The structural order and textural properties of all the materials have also been studied by XRD, elemental analysis and nitrogen adsorption-desorption analysis. Adsorption of naphthalene over various porous adsorbents such as MC, AMC and MCN has been studied. It has been found that the amount of naphthalene adsorption on MCN is higher as compared to the other novel nanoporous carbon adsorbent. The adsorption behavior of naphthalene on the MCN from aqueous solution has been investigated. All adsorption isotherms fitted well with the Langmuir model. Using the Langmuir isotherm the adsorption capacity of naphthalene on MCN was calculated to be 3.53 mmol g^{-1}.

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List of Symbols and abbreviations

q_e – concentration of the adsorbed solute, (mmol g^{-1})
C_o – initial concentration, (mmol L^{-1})
C_e – equilibrium concentration, (mmol L^{-1})
V – volume of the solution, mL
W – mass of the adsorbent, (g)
C(t) – concentration in time (t), (mmol L^{-1})
q(t) – concentration of the adsorbed solute in time (t), (mmol g^{-1})
n – Freundlich constant
K_f – Freundlich constant
q_m – Langmuir constant, (mmol g^{-1})
b – Langmuir constant
$q_{e,meas}$ – concentration of the adsorbed solute were determined experimentally, (mmol g^{-1})
$q_{e,calc}$ – concentration of the adsorbed solute were calculated by Freundlich and Langmuir models, (mmol g^{-1})
t – temperature in reactor, °C
A_{BET} – specific surface area (obtained by a BET N_2 gas adsorption-desorption isotherm)
BET – Brunauer–Emmet–Teller (N_2 gas adsorption desorption isotherm)
MCM – mobile crystalline material
SBA –
PXRD – Powder X-ray Diffraction
SSE – Sum of Squared Error
UV–Vis – ultra violet-visible spectrophotometry
XRD – X-ray diffraction

Greek letters

λ – wavelength, nm

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