Removal of Specific Pharmaceuticals from Water using Activated Carbon

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Abstract. Many types of pharmaceutical substances have been detected with significant concentrations through various advanced instrumental techniques in surface water, ground water, partially treated water (with concentration typically less than 0.1 ug/L), drinking water (concentration below 0.05 ug/L) domestic wastewater, municipal wastewater and industrial effluents (concentration approximately 1 ug/L). Pharmaceutical compounds are found at much lower concentrations in drinking water sources than the normally prescribed doses, but there is concern that chronic exposure to numerous compounds could cause serious health problems and that compounds can act synergistically to cause adverse health effects. The effectiveness of removal the specific pharmaceuticals (paracetamol, carbamazepine, metronidazole and caffeine) from drinking water with adsorption using two types of granular activated carbon (Filtrasorb 400 and WG12) was monitored. Paracetamol is a medication used to treat fever and mild to moderate pain. Paracetamol significantly relieves pain in acute migraine and headache. Carbamazepine is an anticonvulsant medication used primarily in the treatment of epilepsy and neuropathic pain. It is used in schizophrenia along with other medications and as a second-line agent in bipolar disorder. Metronidazole is an antibiotic and antiprotozoal medication. It is used either alone or with other antibiotics to treat inflammatory disease. Caffeine is a central nervous system (CNS) stimulant of the methylxanthine class. It is the world's most widely consumed psychoactive drug.

The concentrations of caffeine in surface waters were detected in the range 0.1 - 6.9 µg/L. The experiments were performed in laboratory conditions with varying values of pH (7, 8, or 6.5), sample temperature, and dose of adsorbent. The experiments were performed in the glass bottles with the volume of 400 mL stock model water (drinking water spiked with pharmaceutical standard) with concentration approximately 0.05 µg/L. On the analytical scales weighed out 400 mg GAC was used and then it was added to the bottles. Subsequently these bottles were regularly stirred at 400 rpm. Samples were taken at 30, 60, 120 and 240 minutes, after which they were analyzed. Analyses of target pharmaceutical were performed in laboratories of ALS Czech Republic in Prague. LC-MS methodology (method was validated according to ISO 17025 system) was used to determine pharmaceuticals in water samples. The adsorption efficiency of pharmaceutical removal and the adsorption capacity of granular activated carbon depends on the time of contact of water with the material. Adsorption efficiency for two types of granular activated carbon varies from 13 to more than 90%.

1. Introduction
In the last decade, traces of pharmaceuticals, typically at levels in the nanograms to low micrograms per litre range, have been reported in the water cycle, including surface waters, wastewater, groundwater and, to a lesser extent, drinking-water. Advances in analytical technology have been a key factor driving...
their increased detection. Pharmaceuticals are synthetic or natural chemicals that can be found in prescription medicines, over-the-counter therapeutic drugs and veterinary drugs [1-4].

Their presence in water, even at these very low concentrations is a potential risk not only to human health from exposure to traces of pharmaceuticals via drinking-water, but also to aquatic organisms and other components of the environment.

Pharmaceuticals enter the environment through many routes, including human or animal excreta, wastewater effluent, treated sewage sludge, industrial waste, medical waste from health-care and veterinary facilities, landfill leachate and biosolids.

The published literature and national studies have shown that concentrations of pharmaceuticals in surface water and groundwater sources impacted by wastewater discharges are typically less than 0.1 μg/l (or 100 ng/l), and concentrations in treated drinking-water are usually well below 0.05 μg/l (or 50 ng/l) [5,6].

On the basis of monitoring the occurrence of selected pharmaceuticals, drugs and their metabolites in surface waters in Slovakia, it was found that the most common to occur pharmaceuticals such as valsartan, venlafaxine, telmisartan, metoprolol, tramadol, clindamycin, erythromycin, carbamazepine and diclofenac. Among legal drugs, it was mainly caffeine. Pharmaceuticals such as erythromycin, diclofenac, telmisartan, carbamazepine or the caffeine stimulant have also been found, for example, in Tatras mountain lakes [7].

1.1. Methods for removal of pharmaceuticals from water

Conventional wastewater treatment facilities typically have biological degradation using the activated sludge process, whereas advanced facilities have tertiary treatment processes, such as reverse osmosis, ozonation and advanced oxidation technologies can generally achieve higher removal rates (up to 100%) for pharmaceuticals compared with conventional processes [8-13].

Pharmaceuticals are a diverse group of chemicals, with varying physical and chemical properties. Treatment efficacy depends on these physical and chemical characteristics (e.g. hydrophobicity), their reactivity towards different treatment processes and process control, such as solids retention time, temperature and hydraulic retention time.

Removal of pharmaceuticals during drinking water treatment is largely dependent on their physical and chemical properties, and treatment processes can therefore achieve some level of removal. For example, biodegradation on slow sand filters and/or sorption to particles removed by coagulation may help reduce the levels of some pharmaceuticals present in drinking-water sources; granular activated carbon (GAC) and powdered activated carbon (PAC) are increasingly adopted in drinking water treatment to remove pesticides and improve taste and odour, and these processes may remove some pharmaceuticals by sorption (or biodegradation on GAC). Groundwater sources that are used for drinking-water typically have low particulate matter and organic matter content. Therefore, drinking-water treatment is mostly single-stage disinfection, without multiple treatment barriers [14-22].

Membrane treatment is highly effective in removing chemicals from water, and removal efficacy is a function of physical and chemical properties, such as molecular weight, hydrophobicity, polarity, chemical nature and pore size of the membranes [23,24].

1.2. Pharmaceuticals used in this research

Paracetamol is a medication used to treat fever and mild to moderate pain. Paracetamol significantly relieves pain in acute migraine and headache. Carbamazepine is an anticonvulsant medication used primarily in the treatment of epilepsy and neuropathic pain. It is used in schizophrenia along with other
medications and as a second-line agent in bipolar disorder. Metronidazole is an antibiotic and antiprotozoal medication. It is used either alone or with other antibiotics to treat inflammatory disease. Caffeine is a central nervous system (CNS) stimulant of the methylxanthine class. It is the world's most widely consumed psychoactive drug. Basic characteristics of selected pharmaceuticals are in table 1.

| Pharmaceutical  | Chemical formula | CAS     | IUPAC                        | Molecular formula | Medication                              |
|------------------|------------------|---------|------------------------------|-------------------|-----------------------------------------|
| Paracetamol      |                  | 103-90-2| N-(4-hydroxy-fenyl)acetamide | C₈H₉NO₂            | analgesic, antipyretic                  |
| Carbamazepine    |                  | 298-46-4| benzo[b][1] benzazepine-11-carboxamide | C₁₅H₁₂N₂O         | anticonvulsant (antiepileptic drugs)    |
| Metronidazole    |                  | 443-48-1| 2-(2-methyl-5-nitroimidazol-1-yl)ethanol | C₆H₅N₃O₃          | antibiotic (antiprotozoal agent)        |
| Caffeine         |                  | 58-08-2 | 1,3,7-trimethylpurine-2,6-dione | C₄H₁₀N₄O₂         | alkaloid (psychoactive drug) central nervous system (CNS) stimulant |

2. Material and Methods
Pharmaceutical’s standard was purchased from company ALS Czech Republic, which also provided us with the sample vials and analysis of pharmaceuticals in samples. From the wide range of pharmaceuticals were chosen paracetamol, carbamazepine, metronidazole and caffeine. Granular activated carbon WG12 was purchased from ENVI-PUR and Filtrasorb 400 (F400) was purchased from Jako Ltd. Basic properties of used granular activated carbon are in table 2.

Stock solution of pharmaceuticals, with initial concentration of approximately 0.5 µg/L, was prepared by mixing 50 mL of the pharmaceutical’s standards with approximately 5 L of drinking water. This solution was then properly mixed and was used in the experiments. Experiments were performed in the Erlenmeyer’s flasks with the initial volume of 400 mL model solution. On the analytical scales was weighed out 400 mg of the adsorbent, which was added to the flasks. These flasks were then stirred for the duration of 4 hours by using OHAUS Orbital Shaker at 400 rpm. During this time samples were taken from the flasks at specific times of 0, 30, 60, 120 and 240 minutes. The samples were taken to glass vials with 40 mL of volume, in which a preservation substance was put (0.32 ml 1% sodium tiosulfate). The vials were stored in a refrigerator and subsequently transferred to the ALS laboratory for analysis.
Table 2. Properties of activated carbons WG12 and F400 [27,28]

|                          | WG12     | F400     |
|--------------------------|----------|----------|
| Iodine number [mg/g]     | min. 1000| min. 1050|
| Particle size [mm]       | 1.0-1.5  | 0.42-1.68|
| Specific surface area (BET) [m²/g] | min. 1000| min. 1100|
| Operating density [g/cm³] | 450±30   | 425      |
| Uniformity coefficient   | max. 1.3 | max. 1.9 |
| Hardness [-]             | min. 95  | min. 95  |
| Abrasion [-]             | min. 85  | min. 75  |
| Moisture by weight [%]   | max. 2   | max. 2   |

Analyses of target pharmaceutical were performed in laboratories of ALS Czech Republic in Prague. Analytical standards of pharmaceuticals were obtained by Neochema. Concentration of target compounds in examined water samples were obtained by accredited LC-MS methodology (method was validated according to ISO 17025 system). Due to the sensitivity of applied UPLC-MS/MS instrumentation, examined water samples (10 ml of each) were only centrifuged and mikro-filtrated prior to their direct injection into the chromatographic system.

Target analytes were separated and detected by liquid chromatography (Acquity UPLC I-Class, Waters, USA) coupled to tandem mass spectrometry (XEVO TQ-MS, Waters, USA). InfinityLab Poroshell 10 EC-C18 (3.0 x 150 mm; 2.7 µm, Agilent Technologies) chromatographic column was used for separation of compounds, applied mobile phase consisted of 0.01% HCOOH in water (A) and MeOH (B). Analytes were identified by specific MRM transitions detected in exact retention time of analysis. The UHPLC-MS/MS system was controlled by MassLynx software, measured data were evaluated using TargetLynx quantifier software. Quantification of analytes was done by the method of isotopically labeled internal standards addition.

3. Results and discussions
The goal of this study was to examine and compare the adsorption efficiency of different types of granular activated carbon (WG12, F400) for the selected pharmaceutical removal from water. The efficiency of the sorption materials used was monitored at pH 7.8 and 6.5, at ambient temperature (22-23 °C) and with an input each pharmaceuticals concentration of 0.44-0.55 µg/l and a water – sorbent contact time 30 to 240 minutes. The results of static tests are given in tables 3 and 4. In these tables you can see the concentration of each pharmaceutical before and after sorption and the comparison of selected pharmaceutical at the different pH values.

The adsorption efficiency (in %) and immediate adsorption capacity (in µg/g) of activated carbons WG12 and F400 were calculated for the individual pharmaceuticals depending on the water – material contact time on the base of the measured concentrations of the individual pharmaceuticals. Based on the defined values, the efficiency of pharmaceuticals removal \( \eta \) [%] and immediate adsorption capacity of selected sorption materials – \( a_t \) [µg/g] were calculated.

\[
a_t = \frac{(c_0 - c_m)V}{m} \quad [\mu g/g] \tag{1}
\]
\[
\eta = \frac{(c_0 - c_m)100}{c_0} \quad [%] \tag{2}
\]

where \( a_t \) is the immediate adsorption capacity in µg/g, \( \eta \) is the adsorption efficiency [%], \( c_0 \) is the concentration of pharmaceuticals before the adsorption, \( c_m \) is the concentration of pharmaceuticals after the adsorption at the time \( t \) [µg/L], \( V \) is the volume of water solution of 0.4 litre, \( m \) is the weight of sorption material, 0.4 g.
Table 3. Concentration of pharmaceutical [µg/L] before and after filtration with WG12

| Time [min] | 0   | 30  | 60  | 120 | 240 | 30  | 60  | 120 | 240 |
|------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Pharmaceutical pH = 7.8 | pH = 6.5 |
| Paracetamol    | 0.444 | 0.386 | 0.316 | 0.136 | 0.011 | 0.365 | 0.297 | 0.196 | 0.034 |
| Carbamazepine | 0.505 | 0.385 | 0.334 | 0.143 | 0.019 | 0.385 | 0.313 | 0.209 | 0.046 |
| Metronidazole  | 0.552 | 0.430 | 0.409 | 0.175 | 0.020 | 0.435 | 0.346 | 0.207 | 0.063 |
| Caffeine       | 0.479 | 0.401 | 0.335 | 0.144 | 0.010 | 0.448 | 0.326 | 0.221 | 0.033 |

Table 4. Concentration of pharmaceutical [µg/L] before and after filtration with F400

| Time [min] | 0   | 30  | 60  | 120 | 240 | 30  | 60  | 120 | 240 |
|------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Pharmaceutical pH = 7.8 | pH = 6.5 |
| Paracetamol    | 0.444 | 0.373 | 0.260 | 0.079 | 0.010 | 0.346 | 0.250 | 0.125 | 0.010 |
| Carbamazepine | 0.505 | 0.260 | 0.180 | 0.058 | 0.012 | 0.278 | 0.196 | 0.112 | 0.012 |
| Metronidazole  | 0.552 | 0.442 | 0.355 | 0.096 | 0.013 | 0.424 | 0.322 | 0.220 | 0.015 |
| Caffeine       | 0.479 | 0.365 | 0.269 | 0.082 | 0.010 | 0.375 | 0.259 | 0.139 | 0.010 |

Results of the adsorption efficiency (in %) and the immediate adsorption capacity (in µg/g) for sorption materials WG12 and Filtrasorb F400 with initial pharmaceutical concentration of approximately 0.5 µg/L are shown in figures 1 and 2.
Figure 1. The adsorption efficiency of pharmaceuticals removal using WG12 or F400 at different pH value

Figure 2. The adsorption capacity of pharmaceuticals removal using WG12 or F400 at different pH value
As it is obvious from the values in the tables 3 and 4, both F400 and WG12 have great adsorption efficiency for all used pharmaceutical. There are little differences between value of pH, but very significant.

Paracetamol had adsorption capacity with WG12 for pH of 7.8 in range from 17 to 92%, the adsorption capacity was an interval 0.058 to 0.453 µg/g and for slightly acidic pH of 6.5 in rage from 13 to 97%, the adsorption capacity was an interval 0.079 to 0.410 µg/g, depending on the contact time. The efficiency with F400 sorption materials for pH of 7.8 and slightly acidic pH of 6.5 was in range from 16 to 98 % and from 24.07 to 98 % respectively. The adsorption capacity of paracetamol removal was in range 0.071 to 0.434 µg/g and 0.098 to 0.434 µg/g, respectively.

Carbamazepine had adsorption capacity with WG12 for pH of 7.8 in range from 23 to 96%, the adsorption capacity was an interval 0.12 to 0.486 µg/g and for slightly acidic pH of 6.5 in rage from 23 to 91 %, the adsorption capacity was an interval 0.12 to 0.459 µg/g, depending on the contact time. The efficiency with F400 sorption materials for pH of 7.8 and slightly acidic pH of 6.5 was in range from 48 to 98% and from 45 to 97% respectively. The adsorption capacity of carbamazepine removal was in range 0.245 to 0.493 µg/g and 0.227 to 0.493 µg/g, respectively.

Metronidazole had adsorption capacity with WG12 for pH of 7.8 in range from 22 to 96%, the adsorption capacity was an interval 0.12 to 0.532 µg/g and for slightly acidic pH of 6.5 in rage from 21 to 88 %, the adsorption capacity was an interval 0.12 to 0.489 µg/g, depending on the contact time. The efficiency with F400 sorption materials for pH of 7.8 and slightly acidic pH of 6.5 was in range from 20 to 98% and from 23 to 97% respectively. The adsorption capacity of metronidazole removal was in range 0.11 to 0.539 µg/g and 0.13 to 0.537 µg/g, respectively.

Caffeine had adsorption capacity with WG12 for pH of 7.8 in range from 16 to 98%, the adsorption capacity was an interval 0.08 to 0.469 µg/g and for slightly acidic pH of 6.5 in rage from 6.5 to 93 %, the adsorption capacity was an interval 0.03 to 0.446 µg/g, depending on the contact time. The efficiency with F400 sorption materials for pH of 7.8 and slightly acidic pH of 6.5 was in range from 24 to 98% and from 22 to 98% respectively. The adsorption capacity of caffeine removal was in range 0.11 to 0.469 µg/g and 0.10 to 0.469 µg/g, respectively.

Reaction kinetics was also studied for the adsorption of these pharmaceuticals. Reaction kinetics of 0th, 1st, 2nd and 3rd order were studied (Figure 3-6).
Figure 4. Reaction kinetics of 0th (left) and 1st (right) order for carbamazepine.

Figure 5. Reaction kinetics of 0th (left) and 1st (right) order for metronidazole.

Figure 6. Reaction kinetics of 0th (left) and 1st (right) order for caffeine.
As the figures 3 to 6 presents, 0th order kinetics has been shown to best describe adsorption process for adsorbent WG12. For the adsorbent F400, the 1st order kinetics is the best describe the process of adsorption. $R^2$ factor represents how accurate and precise kinetic equation is. The closer the number is to 1, the better kinetic equation describes the process. These equations are acquired by linearization of the kinetic equations.

4. Conclusions
In our article, the efficiency and adsorption capacity of the adsorption materials used differed only slightly. Filtrasorb F400 proved to be a better sorption material than WG12 but the difference is very small, as both adsorbents removed more than 90% of pharmaceuticals from water solution after 4 hours. There are little differences between value of pH, but pH value had effect for removal of pharmaceuticals from drinking water using adsorption with activated carbon WG12 and F400.

The adsorption efficiency (%) and immediate adsorption capacity (µg/g) for the individual pharmaceuticals depending on the water – material contact time, pH value of water, and concentrations of the individual pharmaceuticals in water.

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