



Effect of shear stress and carbon surface roughness on bioregeneration and performance of suspended versus attached biomass in metoprolol-loaded biological activated carbon systems

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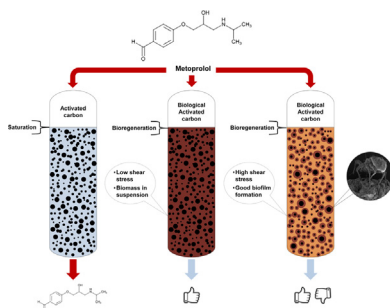
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HIGHLIGHTS

- Pharmaceutical metoprolol was >99% removed by biologically activated carbon.
- Before reaching full biodegradation metoprolol was removed by adsorption.
- The saturated activated carbon was regenerated during the biodegradation phase.
- Biofilm formation on activated carbon granules with a smooth surface was poor.
- However, the performance was not hampered due to the presence of suspended biomass.

GRAPHICAL ABSTRACT



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ABSTRACT

The bioregeneration of activated carbon (AC) in biological activated carbon (BAC) systems is limited by sorption-desorption hysteresis and transport between the adsorbent and biomass. In this study, we investigated these limitations and whether a biofilm covering the AC surface is required. Consequently, BAC reactors were operated at different shear stress and AC surface smoothness, since this may affect biofilm formation. The experiments were carried out in BAC and blank reactors treating synthetic wastewater containing the pharmaceutical metoprolol.

After start-up, all reactors removed metoprolol completely; however, after 840 h the removal dropped due to saturation of the AC. In the blank reactors, the removal dropped to 0% while in the BAC reactors removal recovered to >99%, due to increased biological activity. During the initial phase, the metoprolol was adsorbed, rather than biodegraded. At the end, the AC from the BAC reactors had higher pore volume and sorption capacity than from the blank reactors, showing that the AC had been bioregenerated.

At high shear ($G = 25 \text{ s}^{-1}$), the rough AC granules ($R_a = 13 \text{ }\mu\text{m}$) were covered with a 50–400 μm thick biofilm and the total protein content of the biofilm was 2.6 mg/gAC, while at lower shear ($G = 8.8 \text{ s}^{-1}$) the rough AC granules were only partly covered. The biofilm formation at lower shear ($G = 8.8 \text{ s}^{-1}$) on

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smooth AC granules ($R_a = 1.6 \mu\text{m}$) was negligible. However, due to the presence of suspended biomass the reactor performance or bioregeneration were not reduced. This showed that direct contact between the AC and biomass was not essential in mixed BAC systems. The microbial analyses of the suspended biomass and the biofilm on AC surface indicated that metoprolol was mainly biodegraded in suspension.

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1. Introduction

In Biological Activated Carbon (BAC) systems, activated carbon (AC) treatment is combined with biological treatment. BAC is generally applied for post-treatment of wastewater or pre-treatment of potable water production [1–3]. The AC ensures a high effluent quality, while the biodegradation limits the rate at which the AC becomes saturated. To maximize the AC lifetime, the biomass should not only prevent the AC from becoming saturated, but also regenerate it after a period of net adsorption [4,5]. Net adsorption takes place when the adsorbate concentration in the liquid exceeds the equilibrium concentration at a given AC saturation. However, Abromaitis et al. [6] showed that active BAC biomass is able to reduce the adsorbate concentration to such a level that adsorption does not take place. Thus, net adsorption only takes place when the biomass concentration or specific activity is too low to cope with the persistent organics load. This can be expected to occur during reactor start-up, peak loads, and inhibition of the biodegradation (e.g. due to the exposure to low temperature or toxic compounds) [7]. Thus, the main advantage of BAC systems over pure biological systems for removal of biodegradable pollutants is that the pollutants are retained when the biodegradation capacity is temporary insufficient. This is especially relevant for compounds that are harmful and recalcitrant to biodegradation, such as pharmaceuticals [8,9]. The average removal efficiency of pharmaceuticals (e.g. the β -blocker metoprolol) in wastewater treatment plants usually is around 50% [10,11].

Bioregeneration is the simultaneous desorption of pollutants that are adsorbed to the AC, diffusion of those pollutants from the pores to the biofilm, and biodegradation [5,12–14]. Abromaitis et al. [6] showed that despite considerable hysteresis between adsorption and desorption, the threshold concentration of the BAC biomass is low enough to allow for desorption and thus bioregeneration. However, the rate of the bioregeneration is dependent on the type of AC being used [15–17], reactor configuration, and operational parameters such as hydraulic retention time, temperature, and mixing [5]. One important AC characteristic is the pore size distribution: a high fraction of mesopores usually enhances desorption and thus, bioregeneration because of better intra-particle diffusion. In contrast, a higher fraction of micropores promotes faster adsorption [18–20]. A less investigated influence of the type of AC on BAC performance is its suitability for biofilm formation as granules with a smooth surface might hinder microbial attachment compared to the adsorbents with a rough surface.

Reactor configuration and operation will also affect the biofilm formation. Biofilm formation on AC particles is a slow process and the time needed for colonization can vary from several weeks to months [21,22]. Biofilms in packed bed AC filters are typically thick and fluffy, while in stirred tank reactors the biofilm is denser and thinner due to exposure to shear stress [23,24]. Biofilm formation on the AC surface can lead to pore blockage; macro- and mesopores can be clogged with bacterial biomass or extracellular polymeric substances. This blockage reduces the transportation rate of the adsorbate through the pores and therefore, hampers the adsorption and desorption [5,25,26]. On the other hand, a dense biofilm might be needed to sufficiently reduce the substrate concentration at the AC surface to allow for desorption.

To enhance our understanding of biofilm formation in BAC systems and its role in AC bioregeneration under specific conditions, we assessed the removal of the pharmaceutical metoprolol during start-up of BAC and abiotic AC (blank) reactors containing AC granules with different surface roughness and operated at different shear stress levels. In addition, the adsorption characteristics of the AC granules and the growth, activity, morphology, and phylogenetic composition of the biomass were monitored.

2. Material and methods

2.1. Buffer and medium solution

The adsorption experiments and biomass washing were carried out in phosphate buffered saline (PBS); composition is provided in [Table S1 of the Supplementary material](#). The pH was checked at the start, during, and at the end of each experiment; in all experiments the pH remained between 6.7 and 7.4.

The biomass, used as inoculum, was submersed in PBS supplemented with nutrients (quantities are provided in [Tables S2 and S3 of the Supplementary material](#)). For the reactor experiments, the PBS with nutrients was supplemented with 10 mg/L metoprolol and 137 mg/L sodium acetate (equivalent to 100 mg/L acetate). Acetate was added as a model compound for the easily degradable organics that micro-pollutants such as metoprolol typically accompany. It has been shown that the easily degradable organics influence the biodegradation of micro-pollutants [6]. The applied ratio of 1:10 (metoprolol-acetate) was based on the ratio encountered in the secondary effluent of Dutch wastewater treatment plants. This effluent typically contains <4 mg BOD/L (the BOD is an indicator for the amount of easily biodegradable organic matter) [27]. Whereas, the sum of the organic compounds resistant to biodegradation, including pharmaceuticals, varies in the range of tens to hundreds $\mu\text{g/L}$ [28]. The circa 100 fold higher concentrations used in this study were chosen to be able to monitor the fate of the metoprolol, as this research aims to understand the interaction between biomass and AC, rather than to optimize full-scale BAC systems.

To prevent aerobic growth, the PBS with nutrients, metoprolol, and acetate were fed to the reactors from a vessel which was maintained anaerobic by continuously sparging N_2 gas. The acetate and metoprolol concentrations in the dosing vessel were monitored throughout the experiment and remained constant.

2.2. Inoculation

The biomass used to inoculate the BAC reactors was obtained from the full-scale BAC filter at the “Puurwaterfabriek” factory (Nieuw Amsterdam, the Netherlands). The “Puurwaterfabriek” produces ultrapure water from municipal wastewater treatment plant effluent for oil extraction. The Puurwaterfabriek comprises the following treatment steps: sieving, ultrafiltration, primary BAC filter, polishing BAC filter, reverse osmosis, and electro deionization. The main function of the BAC filters is to remove fouling precursors for reserve osmosis membrane filtration. In 2010, when the Puurwaterfabriek was started, both BAC filters were filled with

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