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ABSTRACT

SYNTHESIS AND APPLICATION OF CARBON-HALLOYSITE COMPOSITES FOR PARACETAMOL AND CHOSEN NSAIDS ADSORPTION FROM WATER

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In this PhD thesis the adsorption of non-steroidal anti-inflammatory drugs (NSAIDs), i.e. diclofenac, ketoprofen, naproxen and paracetamol on carbon-halloysite adsorbents and non-modified halloysite, from water, was investigated.

The carbon-halloysite nanocomposites were obtained through the impregnation of halloysite (H) as the template and saccharose solution as the carbon precursor at different concentration (5%, 10%, 20%, 30% wt.%) and the carbonization of the obtained materials at the temperature of 800°C in the nitrogen atmosphere. The obtained adsorbents were marked as follows: 5C/H, 10C/H, 20C/H and 30C/H.

The physicochemical properties of the obtained composites were characterised using the following methods: scanning electron microscopy (SEM), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), X-ray photoelectron spectroscopy (XPS), and Inverse gas chromatography (IGC).The total carbon (TC) was analyzed and the parameters of porous structure were determined, using the low-temperature nitrogen adsorption method.

In the experimental part of the thesis referring to the adsorption in the static system, some aspects were studied: the influence of contact time, the initial concentration of the adsorbents, pH solution and mass of the adsorbents H and 5C/H, 10C/H, 20C/H and 30C/H on the adsorption process. Based on the obtained results, it was confirmed that composite 30C/H has the highest adsorption capacity for all studied adsorbates. Thus, the suceeding experiments were conducted with the usage of this adsorbent. The adsorbent H was used as the reference system. Adsorption mechanism was found to fit pseudo-second order and intra-particle diffusion models for all chosen NSAIDs and paracetamol on 30C/H and H adsorbents. The

adsorption constant rates decreased in the following order: diclofenac > paracetamol > ketoprofen > naproxen, for the 30C/H adsorbent, and paracetamol > diclofenac > ketoprofen > naproxen for the H adsorbent.

The best fit of the experimental data was obtained for the adsorption Langmuir multi-center model. The adsorption constants for the 30 C/H adsorbent were greater in the case of all the NSAIDs and paracetamol in comparison to the H adsorbent. These values decreased in the following order: paracetamol > ketoprofen > diclofenac > naproxen.

The adsorption experiments were also carried out in the dynamic system with the usage of the Inverse Liquid Chromatography method (ILC): Peak Division (PD) and Breakthrough Curve (BC) methods. It was proved that the adsorption of all adsorbates occurs according to the Langmuir model on "*n*" active centres (PD ILC method). The adsorption capacity of the carbon-halloysite adsorbents was obtained by the BC ILC method. The use of both methods of inversion liquid chromatography (PD and BC ILC methods) can replace the traditional measurement of the adsorption in the static system.

The obtained results confirmed that carbon-halloysite composites are suitable adsorbents for all tested pharmaceuticals in comparison with unmodified halloysite.

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