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Evaluation of the use of regenerated activated carbons for the adsorption of phenol from a river

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Abstract: The aim of the study was to use regenerated activated carbon to adsorb phenol from a river. Coconut shell activated carbon was derived from used tap water filter cartridges. The activated carbon was carbonised and then activated with KOH at 200°C, under a nitrogen atmosphere. The resulting adsorbent was characterised on the basis of nitrogen adsorption by Brunauer–Emmett–Teller (BET), scanning electron microscopy (SEM) analysis and point of zero charge (pH PZC). The study of periodic adsorption included kinetic and equilibrium modelling, determined the effect of solution pH on efficiency and the possibility of regeneration and reuse of the adsorbent. The efficiency of phenol removal from model water was evaluated, followed by the possibility of their adsorption from a polluted river in Silesia Province. Phenol adsorption followed pseudo-second-order kinetics. The adsorbents showed high adsorption abilities, as determined by the Langmuir isotherm model. The model fits the experimental data well. The concentration of phenol in the river was in the range of 0.45–0.77 mg·dm^{-3}, which means that its value was at least five times higher than the standard values. The use of regenerated activated carbon from waste filter cartridges removed phenol from the river by 78% using optimal test parameters.

Keywords: adsorbent regeneration, organic and inorganic contaminants, phenol removal, surface water

INTRODUCTION

The adsorption process using activated carbon is a frequently used method for removing various types of pollutants and toxins from the environment (Sultana *et al.*, 2022). Unfortunately, the adsorbent after a period of active use becomes waste and thus a troublesome problem. The solution to this issue may become the regeneration of the adsorbent, which can then be reused to remove contaminants. Activated carbon was derived from used water filter cartridges used in filter pitchers, very common in our households. The aim of research on reusing used adsorbents is to strive to minimise the environmental burden. It is also related to the implementation of the so-called "closed-loop economy", i.e. increasing the level of recycling and reuse of materials. According to current literature reports, so far there has been no research into the reuse of waste cartridges from water filter pitchers. Therefore, it was decided to test the possibility of using regenerated activated carbon for the adsorption of pollutants found in a river in the Silesian province.

Rapid industrialisation and urban development have led to the introduction of large amounts of pollutants into rivers, decreasing water quality and negatively affecting the ecosystem (Fadlillah *et al.*, 2023). River pollution is often associated with the presence of toxic elements, which is a serious problem worldwide (Jaskuła and Sojka, 2022). Rivers are often polluted with heavy metals (Ciazela, Siepak and Wojtowicz, 2018; Talbi and Kachi, 2019; Kadriu *et al.* 2021; Jabłońska-Czapla and Grygoyć, 2022) as well as sulphates, sodium and chlorides, which is mainly due to upstream discharge of mine water (Nádudvari and Fabiańska, 2015). For example, studies conducted on the Vistula showed that the river's water quality has deteriorated over the past 40 years. In addition to heavy metals, the river is also contaminated with phenols, which come from infiltration of surface pollution and from tanneries (Guéguen *et al.*, 2004). Phenol is also widely used in petrochemicals, pharmaceuticals, refineries, coal conversion, wood products, polymers, paints, pesticides and the paper industry. Wastewater from these industries can contain high concentrations of phenol (Silva da *et al.*, 2022). Phenol is

a dangerous pollutant, so its permanent removal from the environment is required (Dehmani *et al.*, 2022).

Adsorption is one of the extremely efficient and inexpensive methods (Cho *et al.*, 2022; Abdel-Gawwad *et al.*, 2023; He *et al.*, 2023). The application of waste materials becomes an additional advantage of the process (Mittal *et al.*, 2009; Li *et al.*, 2022) for example, Ghasemi *et al.* (2016b) used *Sargassum hystrix* algae as a biosorbent to remove Fe(II) or manganese from aqueous solutions collected along the Gulf coast (Ghasemi *et al.*, 2016a; Ghasemi *et al.*, 2016b). Another study looked at the assessment of the biosorption capacity of *Padina sanctae crucisalgi* in removing fluorides from the aqueous phase carried out by Dobaradaran *et al.* (2016). In turn, Lua (2022) used waste from oil palm shells to produce activated carbon, which was used for the adsorption of phenol. The biosorbent showed a high adsorption capacity of 275.5 mg phenol per g of activated carbon (Lua, 2022). Lütke *et al.* (2019) used activated carbon from black acacia bark waste to remove phenol from an aqueous solution. Based on the literature, a growing trend can be observed towards the use of waste materials for the adsorption of various pollutants.

Adsorption on activated carbon is the most advantageous method due to its large specific surface area, well-developed internal pore structure and surface chemical functional groups located on the outer and inner surfaces (Kumar and Jena, 2016).

The innovative point of this research was to conduct tests according to the possibility of regenerating and reusing using waste materials from our households and to determine the efficiency of phenol removal from river water by static adsorption. The research focused on the characterisation of adsorbents (SEM, BET, pH PZC), adsorption isotherms and kinetics, and the effect of pH on the adsorption process.

MATERIALS AND METHODS

STUDY MATERIALS

The filter cartridges were cleaned of contaminants and dirt with distilled water, dried and then the ion exchanger was separated from the activated carbon. The resulting activated carbon was sieved in a pneumatic sifter, obtaining granules of 0.5 to 1.0 mm in size. The activated carbon was then subjected to a carbonisation process in a muffle furnace at 500°C for 2 h. The next step was the activation of the adsorbent with KOH. In the activation process, the ratio of adsorbent to KOH was 1:1, then the material was kept in the furnace for 6 h at 200°C under a nitrogen atmosphere. After that, the adsorbent was washed with distilled water to pH 6.5 and dried in a laboratory dryer at 110°C.

The surface morphology of the studied minerals was investigated by scanning electron microscopy (SEM). The scanning electron microscope of model JSM 6360LA was used. The structural properties of the adsorbents were determined by measuring the surface area and pore size distribution using the low-temperature nitrogen adsorption and desorption technique according to the Brunauer–Emmett–Teller (BET) method. Measurement of the zero charge point (pH PZC) was performed using the suspension method. For this purpose, 100 mg of the adsorbent was added to 100 cm^3 of distilled water with a different pH (from 2 to 10) and stirred at 25°C for 24 h. The final pH *f* of the solutions was then measured. The pH PZC value is the point at which the ΔpH (pH *f* – pH *i)* curve as a function of pH *i* intersects the zero line. The pH was adjusted using 0.1 mol·dm^{-3} HNO_3 and NaOH solution (Marszałek, 2022).

STUDY METHODS

The effect of contact time and dose on adsorption capacity was investigated. Adsorption was carried out in 100 cm^3 glass flasks in a shaker with an incubator at 300 rpm. The effect of the adsorbent dose on phenol removal was determined. Preliminary studies were conducted on model water prepared by dissolving phenol in deionised water. The volume of the solution was 50 cm^3, and the weight of the sorbents varied from 0.1 g to 0.5 g per 50 cm^3. Kinetic experiments were carried out as follows: 0.2 g of adsorbent was added to 50 cm^3 of model water in glass flasks, and the flask was shaken at 300 rpm for the following times: 10, 20, 30, 40, 60, 90, 120 min. The pH of the solution was kept constant. Samples of adsorbent were separated from the liquid for analysis at fixed intervals. Samples of the adsorbent were separated from the liquid for analysis at fixed intervals. Experiments were also conducted to study the effect of solution pH; pH was controlled with a 0.1 mol·dm^{-3} HNO_3 and NaOH solution. In these experiments, an adsorbent dose of 0.2 g per 50 cm^3 was used, and the contact time was 80 min. Next, specific tests were conducted on river water.

The adsorption process in the studied adsorbent-adsorbate systems was determined according to the equation:

$$A\% = \frac{C_0 - C_t}{C_0} 100 \quad (1)$$

where: *A*% = degree of removal of phenol from solution, C_0 = initial concentration of the metal (mg·dm^{-3}), C_t = concentration of phenol in solution after time *t* (mg·dm^{-3}).

The amount of adsorbed metal ions was determined as:

$$q = (C_0 - C_t)\frac{V}{m} \quad (2)$$

where: *q* = quantity of adsorbed (mg·g^{-1}), C_0 = initial concentration of phenol (mg·dm^{-3}), C_t = concentration of phenol in solution after time *t* (mg·dm^{-3}), *V* = volume of solution (dm^3), *m* = mass of adsorbent (g).

The experimental data were then analysed using a pseudo-second-order kinetics model, as described in Marszałek, Kamińska and Fathy (2022). The experimental data were also fitted using three popular adsorption isotherm models: the Langmuir model and the Freundlich model. All the calculations used in the article were described in a previous paper by Marszałek et. all (2022)

STUDY SUBJECT

The subject of this study was river water from the Silesian province. Many years of underground coal mining and the consequent subsidence of the land surface have contributed to the disruption of water relations on the river's surface. Deformations of the riverbed and the formation of counter-slopes, disrupting the conditions of gravitational water flow, are also consequences of the mining (Czajkowska, 2017).

Water was collected three times over a period of five weeks. In each week, the average of the measurements was calculated. Samples for testing were taken in the spring period (April–May 2023). The river's treatment efficiency was evaluated by monitoring conductivity, pH, colour, and sulphate and phenol concentrations.

WATER QUALITY ANALYSIS

Concentrations of chemical oxygen demand (COD), nitrate nitrogen, ammonium nitrogen, phosphate phosphorus, sulphate, phenol and heavy metals were determined spectrophotometrically using Merck test kits (Spectroquant® Pharo 300, Merck). Total organic carbon (TOC) was measured using a TOC-L series analyser (Shimadzu). Values of pH and conductivity were monitored with a CPC-505 multifunction analyser (Elmetron). Phenol, KOH, NaOH and HNO_3 were purchased from Sigma Aldrich.

RESULTS AND DISCUSSION

PHYSICOCHEMICAL CHARACTERISTICS OF THE RIVER

The evaluation of water quality in typical rivers and their pollution characteristics are important issues in the regulation and management of water reservoirs. Table 1 shows the results obtained from the physicochemical analysis.

High concentrations of phenol were observed in river water, exceeding five times the permissible values set by the Regulation (Rozporządzenie, 2019). Among heavy metals, high concentrations of zinc were noted, exceeding standards over two times. Also, the high salinity of the river was noted based on measurements of conductivity which were of up to 9.43 mS·dm^{-3}. As well as high concentrations of sulphate were noted in the river. River pollution is caused by the rapid economic development of urban areas, and the scale of cities is increasing. The amount of wastewater discharged is increasing significantly, causing a high pollutant load, resulting in the deterioration of surface water quality (Xue *et al.*, 2023). Phenol and heavy metals found in surface waters are associated with the discharge of wastewater from various industries in the metropolitan area (Raj *et al.*, 2023).

MATERIAL STRUCTURE

The first step in the adsorption study was to find out the structure and morphology of the adsorbents used and evaluate the regeneration. The surface morphologies of the adsorbents were observed using SEM (Fig. 1).

Figure 1 shows SEM images of spent activated carbon (a), carbon after carbonisation (b) and KOH-regenerated carbon (c) with magnifications of 5000 times. Activated carbon after KOH activation has a rough and irregular surface structure, as shown in the figure, which is favourable for phenol adsorption (Silva da *et al.*, 2022). The regeneration was also followed by the presence of circular cavities. In the raw carbon, rather elongated cavities were seen. Regeneration resulted in a well-developed pore structure, which can be clearly observed. It is clearly visible that the process of carbonisation, and reactivation with KOH expansion in the atmosphere of nitrogen in high temperature affected the development of pores which was caused by the

Table 1. Physical and chemical characteristics of river water and limit values acc. to Rozporządzenie (2019)

Parameter	Unit	Value in river water in week					Limit value
		1	2	3	4	5	
pH	–	8.2	7.7	7.9	7.9	7.8	6.5–9.0
Conductivity	mS·cm^{-1}	6.60	7.50	8.86	9.43	7.50	–
Colour	mg Pt·dm^{-3}	40	59	50	41	59	–
COD	mg O_2·dm^{-3}	71	63	59	78	64	125
TOC	mg·dm^{-3}	6.27	3.28	3.20	3.80	3.50	30
N-NH_4^+	mg·dm^{-3}	2.3	2.1	2.6	2.5	2.4	10
N-NO_3^-	mg·dm^{-3}	2.8	2.8	3.0	3.2	2.5	30
P-PO_4^{3-}	mg·dm^{-3}	0.1	0.4	0.1	0.2	0.2	2.0
SO_4^{3-}	mg·dm^{-3}	342	438	461	429	456	250
Iron	mg·dm^{-3}	0.65	0.50	0.40	0.50	0.60	10
Zinc	mg·dm^{-3}	0.35	5.10	4.38	4.41	3.90	2.0
Copper	mg·dm^{-3}	0.21	0	0	0	0	0.5
Nickel	mg·dm^{-3}	0.30	0.20	0.23	0.24	0.37	0.5
Lead	mg·dm^{-3}	0.65	0	0	0	0	0.5
Cadmium	mg·dm^{-3}	0.05	0	0	0	0	0.4
Phenol	mg·dm^{-3}	0.48	0.31	0.40	0.44	0.50	0.1

Explanations: COD = chemical oxygen demand, TOC = total organic carbon.
Source: own study and Rozporządzenie (2019).

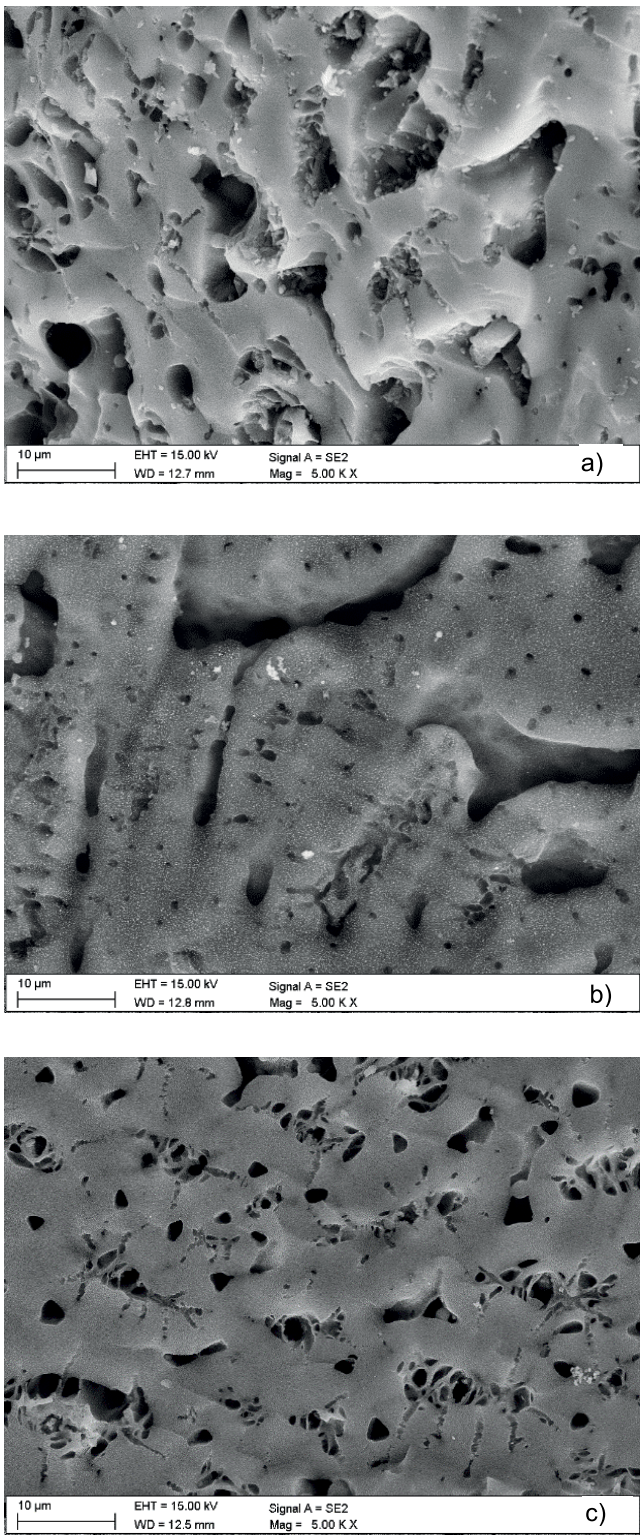

Fig. 1. Scanning electron microscopic images of: a) spent activated carbon, b) carbonised carbon, c) KOH regenerated carbon; source: own study

decomposition of part of the material (Ahmad, Puad and Bello, 2014). The reaction rate between the activator, which is KOH, and carbon also increases when the precursor is exposed to a high activation temperature (Ahmad, Puad and Bello, 2014). The BET surface area of the adsorbent used was 779 $m^2 \cdot g^{-1}$ and for the one activated with KOH, it was 863 $m^2 \cdot g^{-1}$. This shows that activation affected the specific surface area of the adsorbent.

Effects of dose, time and solution ph on phenol adsorption

The second stage of the work was to find out the effect of adsorbent dose, process time, and solution pH. It can be seen from the study that the adsorption ability of phenol, was at a high level. The obtained test results are shown in Figure 2.

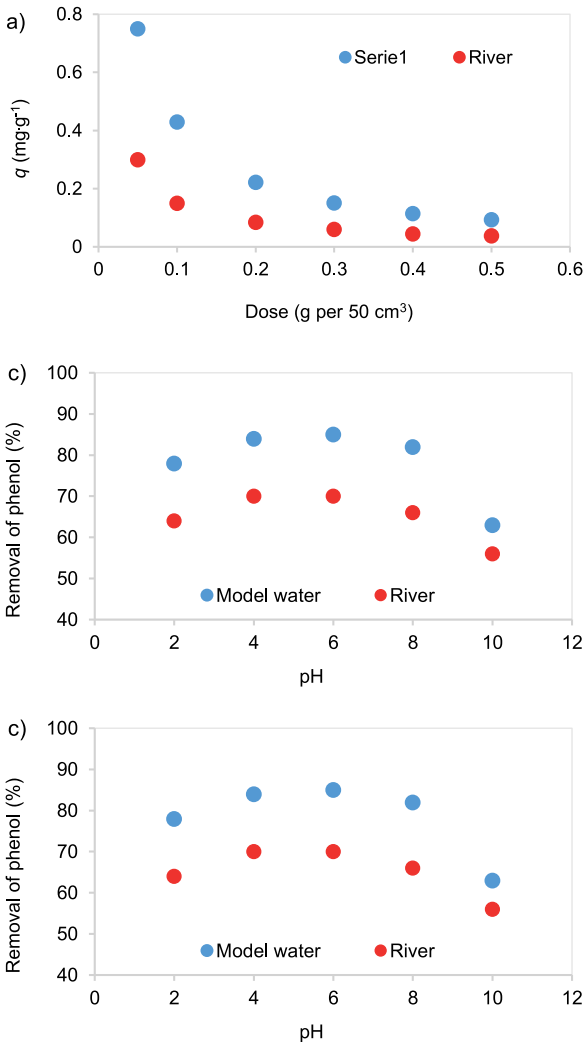

Fig. 2. The amount of adsorbed phenol depending on: a) dose, b) time, c) solution pH; q = quantity of adsorbed metal ions ($mg \cdot g^{-1}$); source: own study

The study showed that the sorption kinetics has two areas, i.e. an area of fast kinetics during the first 30 min of contact and an area of slower kinetics after 30 min. The speed of the adsorption process in the initial phase was related to the presence of a high gradient of adsorbate concentration in the solution. This applies to both model water and river water. In turn, Lv *et al.* (2020) reached the adsorption equilibrium of phenol to activated carbon within 12 min. However, in addition to regeneration, KOH also used EDTA-4Na modifications. As presented by the results obtained, one can see the great influence of the impurities present in the river water on the efficiency of phenol removal. Both in the case of the dose of adsorbent, the time of conducting the process and during the determination of pH. It was also found that as the weight of the adsorbent increased, the amount of

adsorbed phenol decreased. The dose of 3 g·dm^{-3} may be sufficient for phenol adsorption. At this dose, the phenol removal rate was 91% (C = 0.09 mg·dm^{-3}) in model water. In contrast, the adsorption efficiency of phenol from the river was reduced for this dose by 19%. Silva da *et al.* (2022) prepared activated carbons from Brazil nut shells. They used a dosage of 5 g·dm^{-3} and observed an adsorption capacity of 43.96% for phenol from industrial wastewater. Lower adsorption capacity can be caused by a higher amount of pollutants in wastewater compared to a river. In turn, Dehmani *et al.* (2022) demonstrated the adsorption efficiency of phenol from an aqueous solution in the range of 81.64 to 98.77% for acid-activated carbons and 91.48% for commercial activated carbon. Cho *et al.* (2022) achieved >90% phenol reduction using a dose of 10 g·dm^{-3} activated carbon made from the fast-growing kenaf plant. Kumar and Jena (2016) used activated carbon from fox nut shells, and they also obtained a high phenol adsorption efficiency, i.e. 99.19%.

The effect of pH on the sorption of phenol by activated carbon was tested in the range from 4 to 10. The pH value of the water solution impacts both the surface charges of the adsorbent and the degree of ionisation of the pollutants. Therefore, it is a variable affecting the adsorption effect. The figure shows that the degree of phenol removal changed over the pH range studied. In order to determine the effect of pH on adsorption, surface landing (pH PZC) studies of the regenerated adsorbent were also performed (Fig. 3). Based on these results, the pH PZC of the regenerated activated carbon was 5.8. When the pH of the solution was <pH PZC, the adsorbents reacted as positive surface charges, while when the pH was >pH PZC – with a negative surface charge (Garmia, Zaghouane-Boudiaf and Viseras Ibbora, 2018). The pH of the river water was 7.8 while that of the model water was 7.2. Both solutions had a pH higher than the surface charge of the adsorbent. The pH of the solution plays a very important role in the adsorption of phenol by activated carbon, since pH has a significant effect on the existing form of phenol and the chemical characteristics of the activated carbon surface. The highest efficiency was observed at pH 6, and then the amount of adsorbed phenol began to decrease. Dehmani *et al.* (2022) obtained high adsorption efficiency of phenol on activated carbon at pH 4. Kumar and Jena (2016) achieved the maximum rate of phenol removal at pH 7. At higher pH, phenol exists mainly in the phenolic anion, and the activated carbon surface is negatively charged, which reduces the affinity of activated carbon for phenol due to its electrostatic repulsion (Lv *et al.*, 2020).

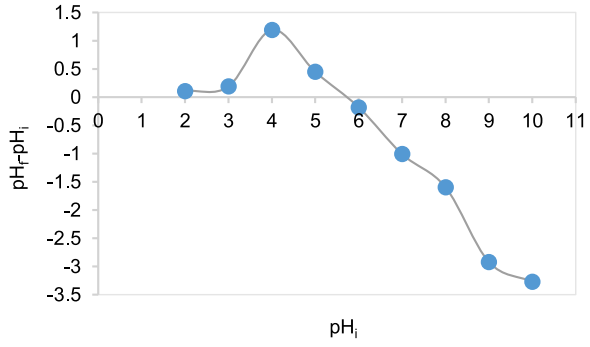

Fig. 3. The point of zero charge (pHPZC) of the adsorbent; source: own study

Isotherms and kinetics of phenol adsorption

Analysing the mathematical description of the sorption equilibrium, it was found that the best fit was given by the Langmuir equation (Fig. 4a). In the case of the description of sorption kinetics, a pseudo-second-order equation was chosen to give the correct fit (Fig. 4b).

According to Table 2, the adsorption behaviour of activated carbon for phenol is more consistent with the Langmuir model,

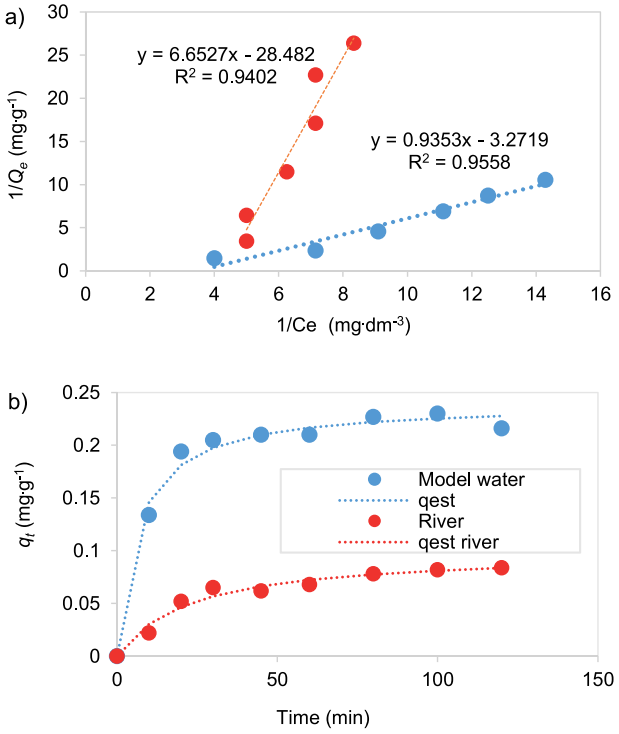

Fig. 4. Fit of: a) equilibrium model, b) sorption kinetics model; Q_e = the equilibrium adsorbed amount, q_t = the amount of phenol adsorbed at time; source: own study

Table 2. Parameters of Freundlich and Langmuir equations, and the correlation coefficients for the adsorption of phenol

Sample	Langmuir			Freundlich		
	Q_m (mg·g^{-1})	K_L (dm^3·mg^{-1})	R^2	K_F ((mg·g^{-1})·dm^3·mg$^{-1/n}$)	n	R^2
Model water	0.305	3.498	0.956	7.461	0.616	0.955
River	0.035	4.281	0.94	66.069	0.278	0.907

Explanations: Q_m = the maximum adsorption capacity, K_L = the Langmuir fitting parameter, K_F = the Freundlich adsorption coefficient, n = correction factor is the number describing surface heterogeneity and sorption intensity, R^2 = determination coefficient.
Source: own study.

with the model water R^2 = 0.956, while for the river R^2 = 0.94. This model suggests the characteristics of single-layer adsorption on a homogeneous adsorbent surface. The maximum adsorption capacity for phenol is 0.3 mg·g^{-1} according to the Langmuir model parameters.

The maximum adsorption capacities and the determination coefficient R^2 calculated from the pseudo-second-order model are shown in Table 3. Phenol adsorption was consistent with the second-order kinetic model, as the R^2 values were close to unity. In addition, the calculated values of $Q_{t(\mathrm{cal})}$ are close to the experimental data values of $Q_{t(\mathrm{exp})}$ – Table 3.

Table 3. Parameters for the pseudo-second-order kinetic model for the adsorption of phenol

Sample	Pseudo-second-order equation parameter					
	K_2 (g·(mg·min)$^{-1}$)	$Q_{t(\mathrm{exp})}$ (mg·g^{-1})	$Q_{t(\mathrm{cal})}$ (mg·g^{-1})	$t_{1/2}$ (min)	h (mg·(g·min)$^{-1}$)	R^2
Model water	0.646	0.239	0.230	0.155	0.04	0.986
River	0.44	0.099	0.084	0.044	0.004	0.968

Explanations: K_2 = the pseudo-second-order rate constant, $Q_{t(\mathrm{exp})}$ = the experimental adsorption capacity (mg·g^{-1}), $Q_{t(cal)}$ = the calculated adsorption capacity, $t_{1/2}$ = he half adsorption time, h = the initial adsorption rate, R^2 = determination coefficient.
Source: own study.

CONCLUSIONS

1. It was found that the surface morphology of the regenerated adsorbent changed under the influence of physicochemical activation. The pores changed their structure from oblong to round. The specific surface area of adsorption increased slightly.
2. Removal of phenol by adsorption on activated carbon derived from used KOH regenerated overflow filters allows to achieve adsorption efficiency of 78% under the following optimal conditions: adsorbent mass 0.2 g, contact time 120 min, phenol concentration in river water 0.5 mg·dm^{-3}.
3. It was found that the high salinity of the river (conductivity from 7.5 mS·cm^{-1} and sulphate concentration 456 mg·dm^{-3}) could affect the adsorption of phenol on the adsorbent used. The efficiency of the phenol adsorption process from model water was 17% higher than from river water.
4. The highest adsorption capacity was observed at pH 6, the rate of phenol removal from the model water was 85% and from the river 70%.
5. The results show that the use of waste materials and their regeneration has great potential due to lower production costs as well.
6. The results of this work open up promising possibilities for the use of an inexpensive and efficient adsorbent that can potentially be used as an adsorbent for effective phenol removal.

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