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Studies on the adsorption of phenol on dried sewage sludge and solid gasification by-products

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ABSTRACT

Dried sewage sludge and solid gasification byproducts (ash) were evaluated in terms of their application to the removal of phenol from aqueous solutions by adsorption. The adsorption was carried out under static conditions at 20˚C. The following properties of the tested unconventional adsorbent materials were determined: susceptibility of phenol to adsorption, the equilibration time and the adsorption isotherms based on the Langmuir and Freundlich equations. Additionally, leaching of primary inorganic and organic substances from adsorption materials was studied including the determination of a toxic effect of the solution (without the addition of phenol). It was found that the gasification ash adsorbs phenol better than dried sewage sludge. The results obtained for the adsorption of phenol on the ash were described by both isotherms with great accuracy. In the case of dried sludge, the Freundlich isotherm made it possible to obtain a better correlation coefficient. It was observed that organic and inorganic substances are leached from the adsorption materials in deionized water. The intensity of this phenomenon was greater for dried sludge than for the ash. It was observed that the toxic effect of this solution increased. The efficiency of adsorption of phenol on conventional and unconventional adsorbents (including those analyzed in detail) was also determined in this study.

Keywords: Phenol; Adsorption; Dried sewage sludge; Gasification; Gasification by-products; Ash; Adsorption isotherms

1. Introduction

The use of adsorption methods in wastewater treatment allows one to achieve a high quality level of the streams discharged into the environment, particularly, when taking into account the presence of different toxic and hazardous organic and inorganic substances in the wastewater [1–7]. This problem applies to both municipal wastewater after mechanical–biological treatment [1–4] as well as to industrial wastewater [5–7]. Active carbon is the most commonly used for this purpose [7,8]. However, some new

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opportunities are being examined, including, for example, the use of waste (with appropriate physico-chemical characteristics) as adsorbents [5,7–10].

One still unresolved problem regarding disposal of sewage sludges is examining the possibility of using them as unconventional adsorbents [1–13]. Sewage sludges have been also considered as a raw material for the production of new adsorbents [14]. This is justified by the fact that there is a basic similarity between the elemental composition of sewage sludge and the raw materials used in the production of carbon adsorbents, such as wood, peat, lignite, bituminous coal, and anthracite [12,14].

In the literature, the adsorption of phenol and its derivatives on active carbon [12,15–18] is often addressed due to a widespread use of this method in order to eliminate biologically active compounds. Description of the process of adsorption is customary based on the analysis of kinetic and static factors, as well as dynamics of the process [18]. The efficiency of that process depends on many factors [12,15–18]. These factors include the characteristics of the adsorbent, including the porous structure, chemical properties of the surface, and also the properties of the compound, such as solubility, the particle size, the type and location of the substituent, and the properties of the solution (e.g. pH, temperature, and ionic strength).

The study presented in this paper is related to the process of adsorption of the phenol from water on dried sewage sludge and solid gasification byproducts (ash). Gasification is a promising method of thermal management of combustible organic substances, however, the management of solid waste gasification byproducts remains an unresolved issue. The part of the study dedicated to the statics of the process determined the susceptibility of phenol to adsorption and the equilibration time for phenol adsorbed on the materials tested. The effect of the initial concentration of phenol on its adsorption was also studied. Langmuir and Freundlich equations were used for the mathematical description of adsorption. The scope of this work included also an assessment of the leaching of primary inorganic and organic substances from adsorption materials and the determination of a toxic effect of the solution using indirect methods. These studies were conducted with no addition of phenol to an aqueous solution. An additional task of this study was to compare the efficiency of adsorption of phenol on conventional and unconventional adsorbents based on various literature data.

2. Materials and methods

2.1. Adsorbents

Dried sewage sludge and ash produced during the gasification process were used in this study. The dried sewage sludge originated from wastewater treatment plant operated as mechanical–biological system located in western Poland. The sludge generated in this wastewater treatment plant is anaerobically digested and then dewatered and dried in a cylindrical dryer on the shelves heated to 260˚C. Finally, that dried sludge is in the form of granules (Fig. 1(a)). The sludge was gasified in a fixed bed reactor using air as a gasifying agent at a temperature in the range of 298–523 K, and using the amount of a gasifying agent corresponding to the excess air ratio (λ) from 0.12 to 0.27. These studies were part of a larger research program, which aimed at determining the effects of gasification parameters on the parameters of the process gas, and in particular, its composition and the calorific value as discussed, for example, in [19,20]. Ash and char were formed as the main solid byproducts of the gasification process. The ash generated during the gasification of sewage sludge (Fig. 1(b)) was selected for this study. In this particular case, the gasification process was operated at constant process conditions (temperature of the gasifying agent 298 K, $\lambda = 0.18$).

Table 1 shows the degree of contamination of dried sewage sludge and the ash produced during the gasification by organic and inorganic materials, which was the subject of our previous study [21]. It was determined that dried sludge was contaminated by both toxic and hazardous organic substances (mainly polycyclic aromatic hydrocarbons) and inorganic substances (nine different heavy metals were identified among other contaminants). Noteworthy, the solid gasification byproducts (ash and char coal) were found to contain no organic substances which occurred primarily in the sewage sludge. The solid products were contaminated mainly by inorganic substances including heavy metals. This information is relevant to the assessment of risk resulting from the practical application of unconventional adsorbents, and associated with the release of these contaminants in the process.

Prior to the process of adsorption, the adsorption materials had been washed with deionized water, dried at 60˚C, and pulverized according to the procedure outlined in [11]. This is the usual procedure used while preparing the adsorbent in this kind of research.

2.2. Adsorbate

The adsorbate used in the study was phenol. A standard phenol reagent was purchased from Merck

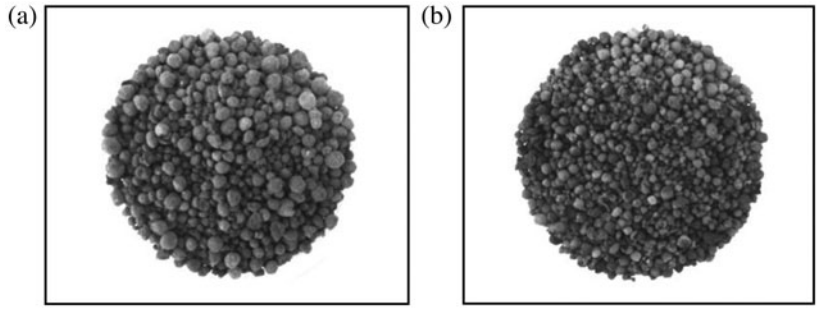

Fig. 1. Samples of dried sewage sludge (a) and ash (b).

Table 1
Concentration of organic and inorganic compounds in sewage sludge and ash [21]

	Concentration, μg/kg dry basis	
Group of organic compounds	Sewage sludge	Ash
Sum of PAHs[1]	2,433.40	n.d.
Sum of pesticides[2]	18.85	n.d.
Sum of PCBs[3]	66.86	n.d.
Heavy metals	Concentration, mg/kg dry basis	
Zn	920.90	2,727.00
Cu	495.30	1,224.00
Pb	119.30	183.00
Ni	103.67	294.00
Cr	180.53	456.00
Cd	6.47	12.30
As	4.19	7.25
Hg	0.99	0.00
Se	9.84	0.01
Sum	1,841.19	4,903.56

[1]Phenanthrene, anthracene, benzo (a) fluoranthene, pyrene, chrysene, benzo (b) fluoranthene, dibenzo (a,h) anthracene, benzo (g,h,i) perylene, indeno (1,2,3-cd) pyrene.
[2]Heptachlor, aldrin, endrin.
[3]2,2′,5,5′-PCB, 2,2′,4,5,5′-PCB, 2,2′,4,4′,5,5′-PCB; n.d.—not detected.

(Warsaw, Poland). A phenol solution was prepared by dissolving the compound directly in deionized water. The study was conducted at pH 7.0. The pH was adjusted using HCl 0.1 mol/L or NaOH 0.2 mol/L. The phenol concentration was determined spectrometrically with 4-aminoantipyrine at a wavelength of 454 nm.

2.3. Adsorption studies

The study of the adsorption process was carried out under static conditions, in Erlenmeyer flasks. To a solution of the adsorbate ($V = 100$ mL, pH 7.0) at a concentration of 60–90 mg/L an adsorbent material (200 mg/L) was added. The process was carried out at a constant temperature equal to 20°C. The samples were shaken during 1 h on a shaker produced by Labor System (Wroclaw, Poland) in a circular motion at a speed of 300 rpm. Before being marked the sample had been filtered through a membrane filter with a pore size of 0.22 μm Merck (Warsaw, Poland) to remove the adsorbent material.

The results are given as the units of adsorbed phenol quantity per gram of sorbent at any time and at equilibrium q_t and q_{eq} (mg/g), respectively; unadsorbed phenol concentration in solution at any time and at equilibrium C_t and C_{eq} (mg/dm^3), respectively; and adsorption yield (adsorption (%) = $100 \times (C_0 - C_t)/C_0$).

Equilibrium data can be analyzed using commonly known adsorption isotherms, which provide the basis for the design of adsorption systems. The most widely used isotherm equation for modeling of the adsorption data is the Langmuir equation, which is valid for monolayer sorption onto a surface with a finite number of identical sites and is given by Eq. (1).

$$q_{eq} = \frac{q_{max} \cdot K \cdot C_{eq}}{1 + K \cdot C_{eq}} \quad (1)$$

where K is the adsorption equilibrium constant including the affinity of binding sites (mg/dm^3) and q_{max} is the maximum amount of phenol per unit weight of sewage sludge or ash to form a complete monolayer on the surface (mg/g). It represents a practical limiting adsorption capacity when the surface is fully covered with phenol. q_{max} and K can be determined from the linear plot of $1/q_{eq}$ vs. $1/C_{eq}$:

$$\frac{1}{q_{eq}} = \frac{1}{q_{max} \cdot K} \cdot \left(\frac{1}{C_{eq}}\right) + \frac{1}{q_{max}} \quad (2)$$

The Freundlich model is an empirical equation based on adsorption on a heterogeneous surface. It is given as:

$$q_{eq} = K_f \cdot C_{eq}^{1/n} \quad (3)$$

where K_f and n are the Freundlich constants that indicate relative capacity and adsorption intensity, respectively. The Freundlich equation can be linearized by taking logarithms (Eq. (4)) and constants can be determined.

$$\log q_{eq} = \log K_f + \frac{1}{n} \log C_{eq} \quad (4)$$

2.4. Assessment of the leaching of inorganic and organic substances from adsorption materials and the determination of a toxic effect of the solution

The scope of this work included also an assessment of leaching primary inorganic and organic substances from adsorption materials and the determination of a toxic effect of the solution using indirect methods. These studies were conducted without the addition of phenol to the aqueous solution. The content of inorganic and organic substances was determined indirectly by determining the conductivity and total organic carbon (TOC), respectively. Conductivity was measured using the laboratory multi-parameter meter inoLab® 740 manufactured by WTW (Wrocław, Poland). This device was also used to measure the general parameters of the solutions, i.e. pH and temperature. The concentration of TOC was determined by the HiPerTOC analyzer manufactured by Thermo Electron (Gliwice, Poland). The toxic effect of the solution was evaluated based on bioluminescence inhibition of the marine bacteria *Vibrio fischeri*. The exposure of the bacteria to toxic substances results in changes in metabolic processes, which simultaneously causes some changes in the intensity of the light emitted by the micro-organisms. The tests were performed using the Microtox Omni system in the Microtox 500 analyser purchased from Tigret Ltd. (Warsaw, Poland) serving as both an incubator and a photometer. The principle of the assay consist of adding a suspension of rehydrated bacteria to the sample solution. After 5–15 min of exposure bioluminescence inhibition percent was measured against the control sample (2% NaCl).

The applied indirect methods were selected since both the dried sludge and the ash produced during the gasification are very complex matrices containing various toxic and hazardous organic and inorganic substances. Full identification of these substances would require a costly and time-consuming multi-chemical analysis.

3. Results and discussion

3.1. The efficiency and equilibration time of the phenol adsorption

Fig. 2 shows the efficiency of the adsorption (as a function of time) of phenol on the sewage sludge and the ash produced during gasification of the sewage sludge. It was determined that the efficiency of adsorption of phenol on the ash was greater than the adsorption on the sewage sludge. For example, after 10 min contact time, in the case of ash ca. 72% of phenol was adsorbed ($q_{10} = 22$ mg/g), and only 21% on the sewage sludge ($q_{10} = 6$ mg/g).

An important parameter which must be taken into account when assessing the adsorbent is the equilibration time. The equilibration time for the phenol adsorption depended on the type of the absorbent material. This parameter was 10 min for the ash and 30 min for the dried sludge. Further extension of the contact time to 60 min did not affect the efficiency of adsorption of phenol in both cases. The adsorption process on the dried sewage sludge was significantly slower than on the ash. This is probably due to the difference in the values of a specific surface area between the studied sorption materials. According to the literature the specific surface area of the dried sewage sludge is ca. 45 m^2/g [22] and for the ash it is ca. 124 m^2/g [23]. Obviously, these values are much lower than those observed in the case of active carbons [24]. It should also be noted that the specific surface area of the ash depends to a large extent on the conditions of the heat treatment of the sludge. It was presented in [23] that the surface area of the ash produced during the pyrolysis of the sludge increased together with the increasing temperature. The authors

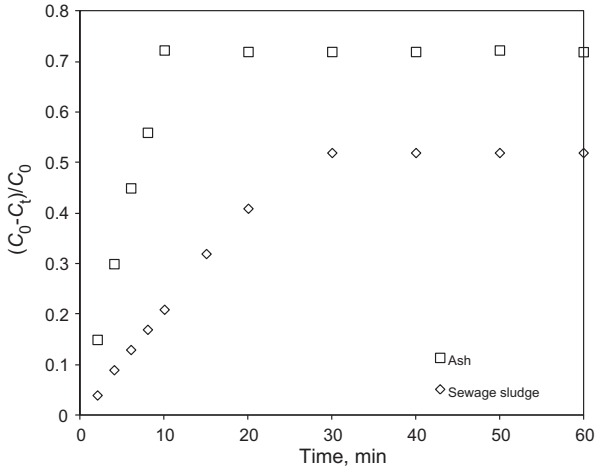

Fig. 2. Equilibration time for the adsorption of phenol on the sewage sludge and ash ($C_0 = 60$ mg/L).

studied the pyrolysis in the context of the preparation of adsorbents, and not as a process of waste disposal.

3.2. The influence of the initial concentration of phenol on the adsorption process

Fig. 3 shows the effect of the initial phenol concentration on the efficiency of adsorption on the tested adsorption materials.

It was found that the phenol adsorption on a sorbent material was increasing with increasing the initial concentration of phenol. This phenomenon was observed for the phenol adsorption on both the dried sewage sludge and the ash. According to the authors of [25], the initial concentration of the adsorbed substance is an important driving force against the mass transport resistance. An increase in adsorption of phenol with higher initial concentration may also be caused by greater interaction between the compound (to be removed) and the adsorption material. Additional information regarding this problem can be obtained from the analysis of the value of parameter n (determining the heterogeneity of the adsorbent) in the Freundlich isotherm, which will be discussed in the next section of this article. Given that any adsorbent material is characterized by a finite number of active sites, it can be stated that the saturation of its surface will occur more rapidly as higher the concentration of phenol will be [13].

3.3. Adsorption isotherms

The Langmuir (Fig. 4) and Freundlich (Fig. 5) isotherms in their linear form were created taking into account the obtained results of the adsorption study. This enabled to determine the coefficients of these isotherms (Table 2). For each system, the coefficient of determination R^2 was determined, which shows that theoretical curve corresponds with experimental data.

The results of the adsorption of phenol on the ash corresponded markedly to both isotherms. The coefficients of determination R^2 were similar. In the case of the dried sludge, a better coefficient R^2 was obtained for the Freundlich isotherm.

The coefficient K_f in the Freundlich isotherm is associated with an adsorption capacity, whereas the constant n depends on the strength of adsorption. Low values indicate a weak binding of the adsorbate to the adsorbent. For the Freundlich isotherm, the lowest K_f value was obtained for the dried sludge. The parameter q_{max} in the Langmuir isotherm equation corresponds to the monolayer capacity and maximum possible adsorption, which may occur with an increasing equilibration concentration. The largest value of $q_{max} \cdot K$ was obtained for the ash, which confirms high efficiency of this material in the adsorption of phenol from solution. Furthermore, the high value of the parameter K for this absorbent material indicates high affinity of the adsorbate to the adsorbent and relatively great adsorption.

3.4. Comparison of adsorption of phenol on conventional and unconventional adsorbents

Table 3 presents the comparison of the maximum adsorption capacity of the phenol monolayer on different adsorbents. Based on the presented data, it can be concluded that the efficiency of phenol adsorption on the adsorbents tested in this study was greater than

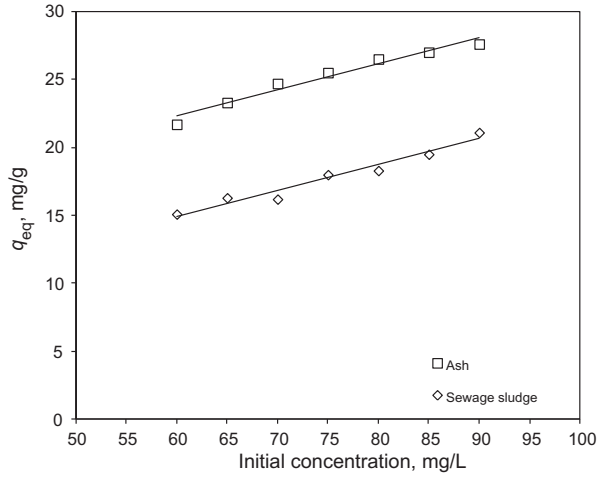

Fig. 3. The effect of the initial concentration of phenol on the adsorption process.

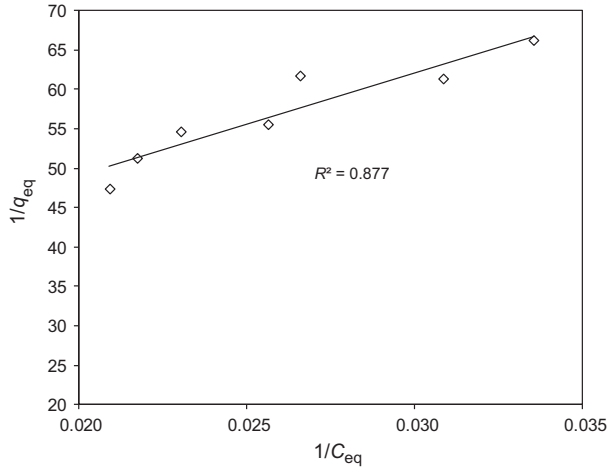

Fig. 4. The linearized Langmuir adsorption isotherm of phenol for sewage sludge.

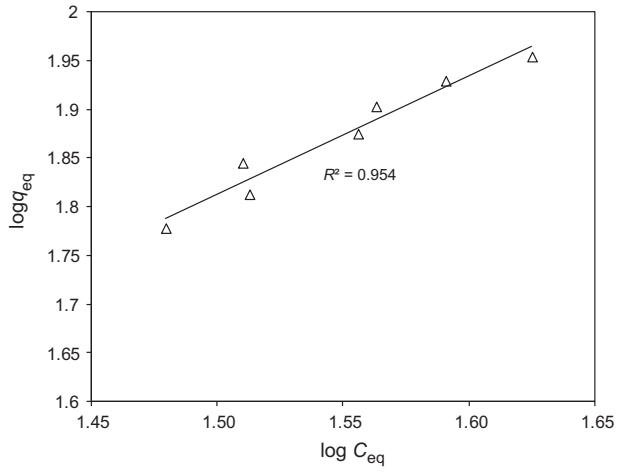

Fig. 5. The linearized Freundlich adsorption isotherm of phenol for sewage sludge.

for the other unconventional adsorbents (bagasse fly ash, neutralized red mud, and olive pomace) only in the case of ash. The adsorption of phenol was found for commercially available activated carbons and activated carbons derived from waste materials such as a beet pulp or a rice husk.

3.5. Leaching of primary organic and inorganic substances from the adsorption materials under study—toxic effect of the solution

The studies showed that tested adsorbent materials in contact with deionized water release primary inorganic and organic substances. This is confirmed by an increase in the conductivity (Fig. 6(a)) and the TOC (Fig. 6(b)). The intensity of this phenomenon was greater for the dried sludge than for the ash. In the case of the dried sludge, it was also observed that the toxic effect of the solution increased, which was determined on the basis of the inhibition of bioluminescence (Fig. 6(c)). The exposure time of the samples (5 and 15 min) had no effect on the inhibition of bioluminescence. According to the toxicity classification scheme based on the observed effect using indicator organisms, [33,34] it was found that deionized water solution after 15 min contact time with the dried sludge showed low toxicity. In the case of the ash the resulting solution was not toxic.

The obtained results regarding the toxic effect of the solutions are difficult to interpret, because both the dried sewage sludge and the ash generated during the gasification of dried sewage sludge are contaminated with various toxic and hazardous organic and inorganic materials. To some extent they have been identified in the prior studies in this field [21], as shown in Table 1. The concentration of heavy metals (including chromium) in the ash was higher than in the dried sludge; however, the solution after given contact time with the ash was not toxic. Elucidation of the phenomena occurring during the contact of water with an unconventional adsorbent needs further investigation. It cannot be excluded that other types of contaminants are present in these samples.

Table 2
Langmuir and Freundlich parameters for phenol adsorption onto the sewage sludge and ash

	Langmuir parameters			Freundlich parameters		
	q_{max} (mg/g)	K (mg/dm^3)	R^2	K_f (mg/g)	$1/n$	R^2
Sewage sludge	0.047	0.015	0.877	14.568	0.859	0.954
Ash	0.038	0.086	0.959	42.218	0.615	0.970

Table 3
Comparison of the maximum monolayer adsorption capacity of phenol onto various adsorbents

Adsorbents	q (mg/g)	References
Activated carbon fiber	110.20	[25]
Beet pulp carbon	90.61	[26]
Commercial activated carbon	49.72	[27]
Rice husk carbon	22.00	[28]
Chemically modified green macro alga	20.00	[29]
Baggase fly ash	12.00–13.00	[30]
Neutralized red mud	5.13	[31]
Olive pomace	4.00–5.00	[32]

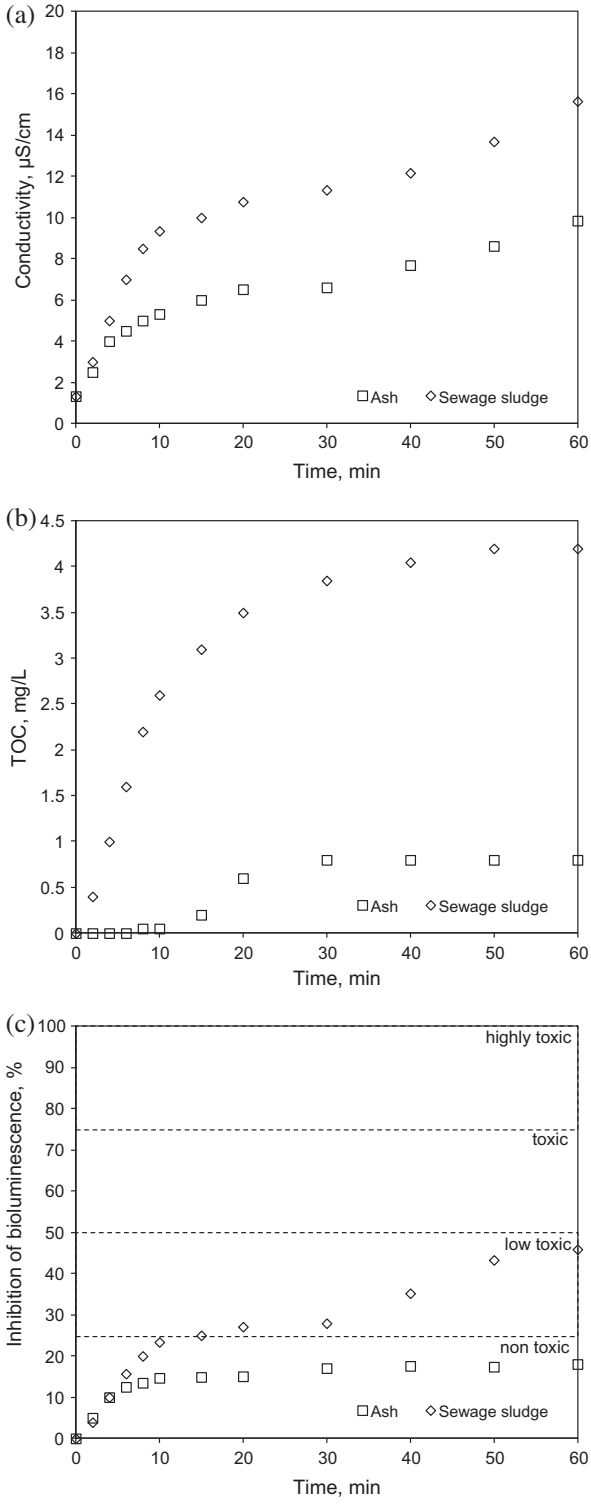

Fig. 6. Changes in conductivity of the solution (a), TOC (b), and the bioluminescence inhibition - exposure time of 5 min (c) in a solution of deionized water containing tested adsorbent materials (process was carried out without the addition of phenol).

4. Conclusions

The following conclusions can be formulated based on the conducted study and the analysis of obtained results:

(1) Phenol adsorption proceeded to a greater extent on the ash produced during the gasification process than on the dried sludge subjected to heat treatment. The adsorption equilibration depended also on the absorbent material. This parameter was 10 min for the ash and 30 min for the dried sludge. Moreover, an increase in the initial phenol concentration corresponded to an increase in phenol adsorption.

(2) The level of correspondence between the theoretical adsorption isotherm (according to the Langmuir or Freundlich equations) and the experimental data in the tested range of phenol concentrations (from 60 to 90 mg/L) depended on the tested adsorbent material. In the case of phenol adsorption on the ash the determination coefficients for both isotherms were similar, and in the case of the dried sludge a better determination coefficient was obtained for the Freundlich isotherm. The analysis of the isotherm coefficients confirmed high adsorption of phenol on the ashes.

(3) Phenol adsorption capacity on the adsorbents tested in this study was greater than for the other unconventional adsorbents only in the case of ash.

(4) The tested adsorbent materials released primary organic and inorganic substances on the contact with deionized water. The intensity of this phenomenon was greater for the dried sludge than for the ash. An increase in the toxic effect of the solution of this material was also observed. These studies, however, require some further research in order to understand this phenomenon better.

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