

Activated Carbons from *Miscanthus* Straw for Cleaning Water Bodies in Kazakhstan

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Abstract

Pollution of water bodies by heavy metals is an acute problem in Kazakhstan. Hence, search for and implementation of sustainable environmental technologies for water purification is of high priority. Activated carbon appears as an appropriate material as reflected in a number of previous studies. Due to a growing interest in low-cost active coals from renewable, rapidly recovering raw materials, especially for the purification of drinking water and wastewater, special attention has been in recent years been paid to the preparation activated carbons from some types of agricultural by-products. Along these lines a technology for producing new carbon-containing sorbents, i.e., activated carbon from *Miscanthus* straw (*Miscanthus × giganteus*) is reported. The method of preparation, type of reactor, experimental conditions and the influence of the selected initial material on the properties of the resulting activated carbons are discussed. The activation of the primarily carbonized material in super-heated steam creates an activated coal material with a specific surface area of 541.9 m²/g and a specific pore volume of 0.232 cm³/g. The possible application for purification of water bodies from selected heavy metals was studied by estimation of the sorption capacity of the generated active coal material towards selected metal ions. The adsorption capacity for copper, zinc and lead ions is 90, 100, 500 mg/L, which allows to consider obtained by this method AC as an attractive alternative to traditional coal sorbents.

1. Introduction

Freshwater resources are limited, and in Kazakhstan the water quality of most surface water bodies is unsatisfactory.

One of the possible solutions to this acute problem of pollution of water bodies in Kazakhstan with heavy metals (HM) is the treatment of wastewater from enterprises of mining and smelting complexes, chemical industries, agriculture and household waste using activated carbons. Obviously, the search for and development of new economically attractive and sustainable technologies for the process of cleaning water bodies from HM

has attracted increasing attention [1–4]. The need to further develop this area of research is emphasized by the United Nations' Sustainability Development Goals, specifically goal No. 6 that states the need to improve water quality by 2030, as well as to ensure the protection and restoration of water-related ecosystems by 2020.

For the purification of aqueous media, sorption purification methods are widely used, in particular applying activated carbon (AC) with a highly developed porous structure as sorbent, which is capable of sorbing gaseous as well as dissolved substances [5].

Thus, carbon black has long and effectively been used to solve environmental problems in metallurgical, oil and gas industries, agriculture, etc. [6, 7].

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Obviously, the application field of AC is very broad. Thus, there is a need to develop new sources of carbon-containing materials with specific physico-chemical properties [8–11], which include high carbon content, the presence of a porous structure or the possibility of their formation, as well as a low mineral content. As the demand for AC materials increases, a simultaneous increase in prices is seen calling for alternative low-cost processes, which are at the same time both environmentally friendly and sustainable.

On the industrial scale, ACs are typically produced from wood, oil waste or coal. Further methods for obtaining activated carbon from industrial food waste have recently been proposed (coconut or walnut shells, etc.) [12–14]. However, it appears mandatory to search for alternatives to the traditional raw materials in order to reduce the cost of obtained coal, as well as the conservation of the woodland and natural resources. Renewable sources of low-cost lignocellulose materials with a high carbon and low ash content [15, 16] constitute in this respect as interesting option.

Thus, the purpose of this work is to obtain activated carbon from cheap non-traditional raw materials of plant, to study its physico-chemical properties, and its further use in the purification of water bodies.

For agrarian regions, obtaining activated carbon from agricultural waste for the purpose of purifying drinking water constitutes an important and promising alternative. Thus, the simultaneous realization of two specific tasks, i.e., minimizing agricultural waste and generating the desired carbon material appears highly attractive [17].

We have considered some types of raw materials for the production of AC and selected the high-yielding triploid, perennial cereal of the type C4 *Miscanthus* × *giganteus* obtained by crossing diploid *Miscanthus sinensis* Anders with triploid *Miscanthus sacchari florus* Hack has received special interest [18]. This plant grows well in waterlogged areas, and is further adapted to cold winter conditions. Worldwide interest in *Miscanthus* × *giganteus* has increased due to its ability to accumulate HM ions in roots, reduce organic xenobiotics in the rhizosphere, while maintaining high yielding [19, 20]. The aboveground biomass that is not contaminated with HM can be used for various purposes [21–23], in particular, for the manufacture of activated carbons [24, 25]. It's known that *Miscanthus* previously has been used as a source for AC production [26]. This method applies activation in an

nitrogen atmosphere, whereas in the present study activation at air has been applied. Further, as will appear, the present study reports improved physico-chemical characteristics of the final product as is the sorption characteristics towards heavy metals.

Technologies are being developed for using it as a bioenergy fuel, due to its high yielding nature that allows a harvest from one sowing to be collected for approx. 15–30 years in amounts of up to 15 t per hectare [27].

Studies using *Miscanthus* × *giganteus* for cleaning heavy metal contaminated military sites in Kazakhstan [28] as well as studies on the possibility of using *Miscanthus* for the production of cellulose and lignin as a substitute for cotton are ongoing.

Of the present study focus on developing methods for obtaining AC from the above-ground mass of *Miscanthus* straw for potential use in purification technologies for polluted water media.

2. Methods and materials

2.1. Sample preparation

Samples of *Miscanthus* straw were obtained from the Institute of Plant Biology and Biotechnology [28]. The samples were grinded in a rotary knife mill PM 120 ("Vibrotekhnik", St. Petersburg, Russia) to a piece size of 1–5 cm, at a rotation frequency of 1500 rpm. Portions of 30 g of the grinded sample were washed with distilled water. The sample was dried for 2 h at 60–65 °C, and subsequently at 100–105 °C to constant weight.

2.2. Carbonization

Carbonization of the dry, grinded *Miscanthus* straw sample was carried out in a muffle furnace in a nitrogen atmosphere with a temperature rise rate of 1–5 °C/min. The calcination temperature was in the range from 300 to 600 °C. It was found that an increase in the temperature to above 600–650 °C caused a decrease in the yield of the carbonization product in agreement with previous studies by Zanzi et al. [29].

Time ranged from 30 to 60 min. Upon completion of the carbonization process, the reactor was cooled and the carbonizate was subsequently transferred to the activation furnace.

2.3. Activation

Activation of the obtained carbonizate was carried out with superheated steam at 800–850 °C, the steam consumption being 100–200 g per 50 g of carbonized product. The activation time was 60–120 min [29].

The ash content and humidity of the obtained coal samples were determined according to GOST 33625-2015 [30].

2.4. Characterization of the activated coal samples

The study of the surface- and structure characteristics of the activated material, as well as elemental analysis were carried out using a FEI Quanta 3 D200i electron scanning microscope (FEI Company, Hillsboro, Oregon, USA) with energy dispersive X-Ray fluorescence detector (AM-TEK EDAX, Mahwah, New Jersey, USA). The microscope was tuned to an accelerating voltage of 15 kV, the working distance was 9.8 mm. Micrographs were taken at a magnification of 1000 to 30000 times to obtain a detailed information of the surface morphology. Elemental analysis was performed at an accelerating voltage of 15 kV, the working distance was 15 mm. The scanning surface of working material area was 19200 μm^2 .

Specific surface area and specific pore volume was determined applying a Sorbtometer M (Katakona, Novosibirsk, Russia). The specific surface area was calculated on the basis of the Brunauer-Emmett-Teller (BET) equation by a single point method [31] using a nitrogen adsorbate gas at 77 K.

The sorption capacity of the activated coal samples was determined by iodine titrimetry according to GOST 4453-74 [32].

The methylene blue sorption capacity was determined according to the GOST 4453-74 standard [36]. The optical density was determined on a Lambda 35 UV spectrometer (Perkin Elmer, Germany) using a blue light filter ($\lambda = 400 \text{ nm}$) in 10 mm cuvettes. Distilled water was used as a control solution. Slit width was 1 nm.

To determine the sorption capacity for copper, zinc and lead ions, standard solutions of CuSO_4 , ZnSO_4 , $\text{Pb}(\text{NO}_3)_2$, respectively, containing 1 to 500 mg/L were prepared in distilled water. A sample ($m = 0.2 \text{ g}$) was placed in a solution (20 mL) for 60 min. Sorption was carried out in 50 mL conical flasks under agitation at 100 rpm on a Sky Line laboratory orbital shaker S-3.20 L Sky Line (ELMI, Latvia) with a rotation amplitude of 20 mm. The equilibrium concentration of the metals was determined applying a Analyst-400 atomic absorption spectrometer (Perkin Elmer, Germany) with acetylene/air flame atomization at flow rates of 2.5 L/min acetylene and 10 L/min for air using electrodeless lamps and a hollow cathode lamp ($\lambda = 324.75, 283.31$ and 213.86 nm for copper, lead and zinc, respectively).

The sorption of copper, zinc and lead ions by the coal material was studied using Spectroscan Max-GV X-ray fluorescence spectrometer ("Technical Equipment" LLC, St. Petersburg, Russia) working at the operating voltage: 40 kV, current 0.5 to 3 mA depending on the analyzer crystal. Four types of crystals were used to obtain the spectra: LiF200, C002, PET, KAP.

The physico-chemical studies described above were conducted at the "Center of physical-chemical methods of Research and Analysis", "National Nanotechnology Laboratory of Open Type" and "Laboratory of Physical Chemical Methods of Research" of the Faculty of Chemistry and Chemical Technology of Al-Farabi Kazakh National University.

2.5. Regeneration of AC samples

The regeneration of AC from *Miscanthus* straw samples after the sorption process of HM was carried out using a 10% solution of nitric acid at a ratio of 1:50 mass of dry coal: acid solution; the contact time being 60 min while stirring at 100 rpm. Subsequently the sample was washed with distilled water and air dried.

3. Results and discussion

3.1. Investigation of samples of *Miscanthus* straw from first and second year of cultivation

Samples of *Miscanthus* straw, prepared as described above, were carbonized under nitrogen at different temperatures. In Table 1 the elemental composition of the carbonized material following different calcination temperature and time is shown.

The results of the experiments showed (vide supra) that the optimal calcination temperature for the carbonization process was 400–500 °C. A further increase in temperature above 600–650 °C caused burning of the sample where as applying temperatures lower than 400 °C caused incomplete charring of the straw material. With incomplete carbonization of the sample, a high content of impurities is observed due to incomplete removal of the organic component of straw biomass of *Miscanthus* containing O, Mg, P, K, Ca. At higher temperatures these impurities are removed in the form of tar and gases. At 300–350 °C the carbon content was only 57.1%, where as an increase of the calcination temperature 400–500 °C, the sample was completely carbonized in 30 min, and the carbon content in the sample was 78.3–81.1%. A further increase in carbonization time did not result in an increased yield.

Table 1
Results of elemental analysis of coal based on straw of *Miscanthus* of the first (Coal 1) and second (Coal 2) years of cultivation, obtained at different calcination conditions

Sample	Carbonization conditions	Mass fraction of an element, %						
		C	O	Mg	P	Cl	K	Ca
Coal 1	T – 300–350 °C, T – 60 min	57.1	21.8	2.5	3.0	5.0	8.5	2.1
	T – 400–450 °C, T – 60 min	81.1	6.5	0.49	0.53	3.0	7.7	1.1
	T – 400–450 °C, T – 30 min	80.5	7.1	0.45	0.70	3.1	7.0	1.1
	T – 500–550 °C, T – 30 min	79.8	7.6	0.48	0.71	3.2	7.2	1.0
Coal 2	T – 400 °C, T – 30 min	78.5	20.3	-	-	-	1.2	-
	T – 500 °C, T – 30 min	78.3	9.2	0.31	0.69	1.3	8.0	2.2

The mass loss during carbonization under optimal conditions amounted to 70–72% of the initial sample mass. The content of these components was studied in the work of Minkova et al. [33].

To increase the active surface area of the carbonized material and activate the pores, the obtained samples were activated for 120 min at 800 °C, using superheated water vapor. The weight reduction of the carbonizate during the activation process amounted to 10–12%. The effect of activation was elucidated by the BET analyses. In Table 2, the data for specific surface area and specific pore volume are given.

It was disclosed (Table 2) that the activation of coals using superheated water vapor makes it possible to obtain AC from *Miscanthus* straw with a specific surface area of up to 541.9 m²/g and a pore volume of 0.232 cm³/g. In 2016 Hodgson et al. [25] described the technology for producing coal from *Miscanthus × giganteus* and coal from other types of plant raw materials. The technology of coal production presented in the present study differs from the Hodgson study [25] by the additional activation of the obtained coal material leading to a significant increase in the surface area and

pore volume. Further the present study shows that an increased size range of straw material, from 0.5 to 5 cm, can be used. In work [26], the activation of the obtained coal from *Miscanthus* straw was carried out in a nitrogen atmosphere, which, in comparison with the activation method used in our work, makes it possible to obtain AC with a surface area of only up to 322 m²/g. In 2006 Michel et al. [24] considered *Miscanthus* for the production of briquettes as well as the possibility of preparing coal from pellets of *Miscanthus* straw. However, the activated carbons, obtained from straw [24] had lower specific surface area, compared to coals obtained from granules. Thus, the surface area of the coal strongly depends on the actual processing method for the straw [24].

Activated carbon was obtained from straw of *Miscanthus* samples of the first and second years of cultivation. According to the results of the analysis, an increase in the activation time up to two hours increases the surface area of a sample of coal obtained from the *Miscanthus* straw of the first year of cultivation by 1.63 times. An increase in the activation time did not significantly affect the change in the surface area of the coal from the straw of the

Table 2
Characteristics of the porous structure of coal based on *Miscanthus* straw of the first (Coal 1, AC 1) and second (Coal 2, AC 2) years of cultivation (T_{fir.} – 400–450 °C, t_{fir.} – 30 min, T_{act} – 800–850 °C)

Characteristics of the coal structure	Coal 1	AC 1, t _{act} – 60 min	AC 1, t _{act} – 120 min	Coal 2	AC 2, t _{act} – 60 min	AC 2, t _{act} – 120 min
Specific surface area, m ² /g	27.2	251.1	408.8	64.5	541.9	520.0
Specific pore volume, cm ³ /g	0.012	0.108	0.175	0.028	0.232	0.223

second year of cultivation; thus, the full activation of the carbonized product apparently takes place within a period of 60 min.

An examination of the surface of the obtained AC confirming the presence of a porous structure can clearly be seen from the micrographs shown in Fig. 1.

The surface of activated carbon samples is represented by pores of various size sarranged in order and have the oval shape (Fig. 1). In Table 3 the elemental composition of the carbonized material after activation is shown.

Table 3

Elemental composition of carbons obtained from *Miscanthus* straw before and after activation
($T_{\text{fir.}} = 400\text{--}450\text{ }^{\circ}\text{C}$, $t_{\text{fir.}} = 30\text{ min}$,
 $T_{\text{act}} = 800\text{--}850\text{ }^{\circ}\text{C}$, $t_{\text{act}} = 120\text{ min}$)

Sample	Mass fraction of an element, %						
	C	O	Mg	P	Cl	K	Ca
Coal 1	79.8	7.6	0.48	0.71	3.2	7.2	1.0
AC 1	89.9	7.4	0.39	-	0.42	1.8	-
Coal 2	78.5	20.3	-	-	-	1.2	-
AC 2	85.2	9.1	-	0.45	-	2.4	-
Coal 2	78.3	9.6	0.31	0.69	1.3	8.0	2.2
AC 2	84.2	12.5	-	-	-	3.3	-

Steam activation affects on the specific surface areas, pore volumes and pore size distribution and might increase O content [34]. In addition, the yield of activated carbon depends on the activation time. Carbon material is partially combusted in oxygen, which is present in steam. Thus, longer activation times, may cause increased burning of organic compounds Obviously this leads to some consumption of the carbonation product, but results in a material with rather high specific surface and pore volume [35]. During the activation process volatiles are removed with the steam causing a decrease in the amounts of impurities associated with the elements, such as Mg, P, Cl, K and Ca. The difference in the content of trace elements in the coal can be explained by the fact that the material of *Miscanthus* straw may have a different composition depending on the time of the year the material is collected, as well as the soil and the age of the plant [36]. Hence, the activation of the coal samples caused an increase in the mass fraction of carbon to 85–90%. During the activation of coal, containing a large amount of oxygen, part of the oxygen is removed, and the mass fraction of impurities in relation to carbon may increase, which is observed in the second pair of coal before and after activation.

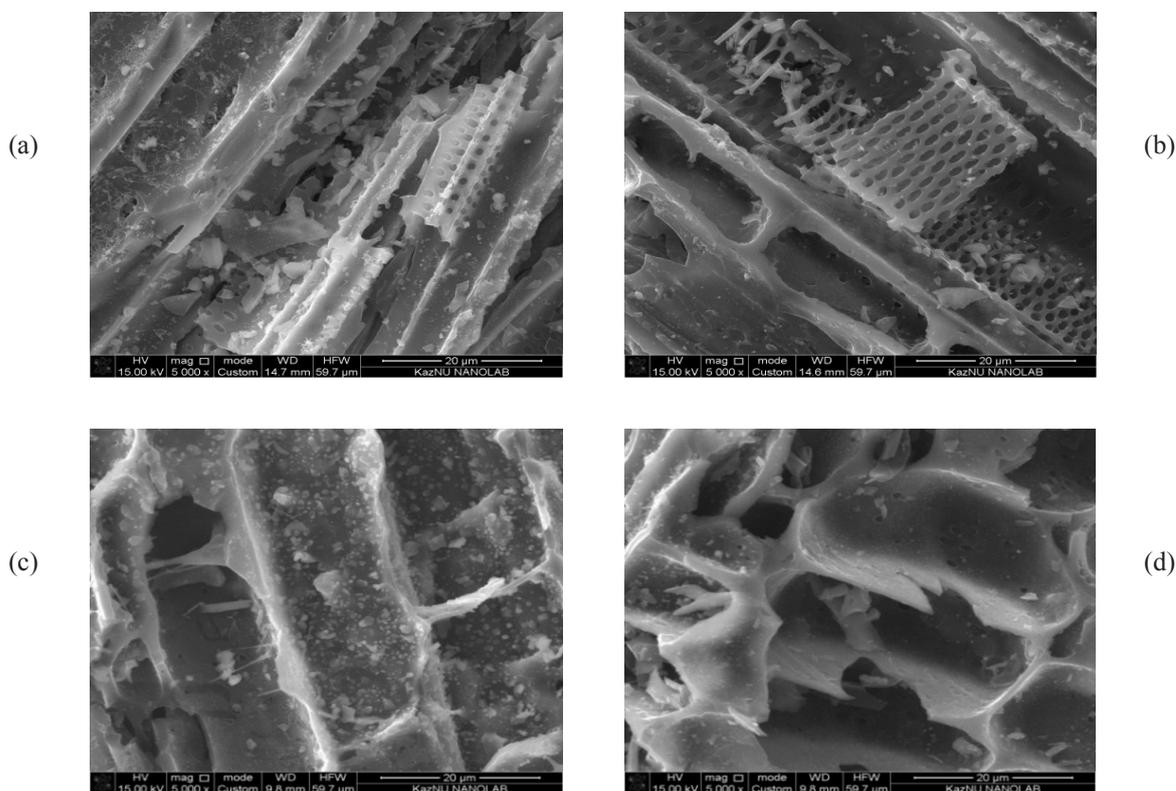


Fig. 1. Micrographs of activated carbon from *Miscanthus* straw ($T_{\text{fir.}} = 400\text{ }^{\circ}\text{C}$, $t_{\text{fir.}} = 30\text{ min}$, $T_{\text{act}} = 800\text{ }^{\circ}\text{C}$, $t_{\text{act}} = 120\text{ min}$): (a) – Coal 1 before activation; (b) – Coal 1 after activation; (c) – Coal 2 before activation; (d) – Coal 2 after activation.

Table 4
Properties of AC from *Miscanthus* straw material

Sample	Carbonization conditions	Iodine adsorption activity, %	Methylene blue adsorption activity, mg/g	Moisture content, %	Ash content, %
Coal	$T_{\text{fir.}} - 500\text{ }^{\circ}\text{C}$, $t_{\text{fir.}} - 30\text{ min}$	215	0	4.5	15.0
AC	$T_{\text{fir.}} - 500\text{ }^{\circ}\text{C}$, $T_{\text{fir.}} - 30\text{ min}$ $T_{\text{act}} - 850\text{ }^{\circ}\text{C}$ $T_{\text{fir.}} - 120\text{ min}$	59.5	62.5	5.4	12.5
Birch activated carbon – for adsorption from aqueous media in filter installations [32, 37]		Not less than 70	Not less than 225	Not more than 10	Not more than 10

3.2. Properties of AC from *Miscanthus* straw, comparative characteristics

The obtained ACs from *Miscanthus* straw samples were characterized as described in the sect. 2.4, the resulting data being summarized in Table 4. For comparison, data from birch activated carbon [32, 37] has been included.

Analysis of the physical-chemical properties of coal samples after activation disclosed an increased sorption capacity for methylene blue and for iodine. The obtained data were compared with standard physical-chemical indicators of birch activated carbon (BAC), which are reported among the best adsorbents used in practice [38]. Hence, the adsorption activity of the AC from *Miscanthus* straw with respect to iodine, as well as their moisture and ash content are comparable to those reported for BAC [32]. As methylene blue is a relatively large molecule a low adsorption rate, compared to BAC [32] indicates a smaller pore size of AC from *Miscanthus* straw compared to pores of coal obtained from the wood.

In the work of Minkova et al. [33], activated carbon from *Miscanthus* was obtained by the single-stage method with an iodine number about 28.1%. The two-stage method of carbonization with activation allowed us to get coals with iodine number 2 times larger – 59.5%.

Experimental data on the sorption of HM from aqueous solutions by AC from *Miscanthus* straw are visualized as adsorption isotherms in Fig. 2, shows relatively high sorption capacity for the studied metal ions.

The high porosity of the obtained AC from *Miscanthus* straw obviously contributes to the sorption.

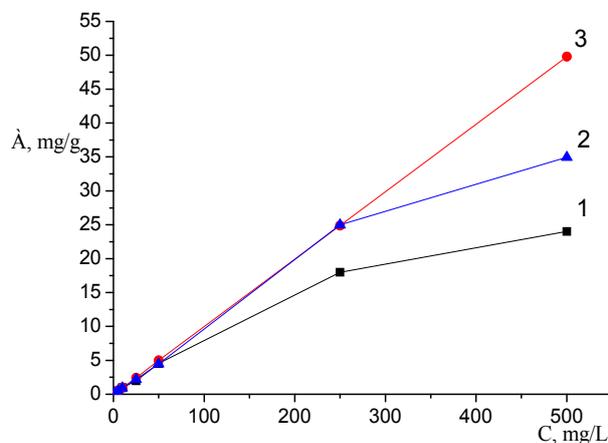


Fig. 2. Sorption isotherms for metal ions on ACM-2: (1) – copper; (2) – zinc; (3) – lead.

In comparison with the coals studied by Hodgson et al. [25] where a removal of up to 51% of zinc, was found, AC displayed the greatest zinc adsorption performance and removed 89.4–90.9% in the same concentrations of zinc in water.

The maximum experimental sorption capacities for the three HMs are shown in Table 5, the values being estimated graphically using a linear anamorphosis of the Langmuir adsorption isotherm.

Table 5
Adsorption capacity of heavy metal ions by activated carbons from *Miscanthus*

Sample	Adsorption capacity of sorbents, mg/g		
	Copper ions	Zinc ions	Lead ions
AC	90±2.97	100±1.84	500±3.42

Significant at $p < 0.05$

Whereas the affinity for copper and zinc ions is virtually identical the affinity of AC from *Miscanthus* straw to lead ions apparently is significant higher by a factor 5. These results are in agreement with a previous study on the sorption of Cu, Zn and Pb ions from aqueous solutions on glauconite [39], where the maximum sorption capacities from Langmuir isotherms were estimated to be 2.96, 3.07 and 9.12 mg/g for Cu, Zn and Pb, respectively, i.e., virtually identical sorption of Cu and Zn and a significantly higher sorption for Pb. A similar relation between Zn and Pb was found by Jain and Ram [40].

The removal of heavy metals is mainly due to sorption, simple metal deposition, or more complex surface interactions. Thus, the overall process should probably be associated with a combination of several simultaneous processes and obviously depending on the nature of the metal [41].

A series of experiments was conducted to establish the nature of the sorption processes of metal ions by AC from *Miscanthus* straw. Comparative analyses were carried out comparing the X-ray spectra of AC before and after the sorption of HM, and further following the regeneration of AC with dilute nitric acid (cf. sect. 2.4).

The X-ray spectra of activated carbons, obtained after sorption show intense peaks of adsorbed metal ions. This indicates the presence of metal ions on the surface and in the volume of AC, i.e. successful sorption process.

After treatment with a solution of nitric acid the X-ray spectra of AC demonstrate that the regeneration of the treated activated carbons completely returns the carbon to its origin state and thus applicable for further use. It should be noted that the present study does not include elaborate studies of the sorption-regeneration sequence, i.e., finding out the amount of reuse and the average loss of sorption capacity during sorption-desorption cycles.

4. Conclusions

It has been shown that highly effective carbon sorbents, based on cheap non-traditional and sustainable raw materials of plant origin, *Miscanthus* can be obtained in a two-stage treatment: carbonization in a nitrogen atmosphere and subsequent activation with superheated water vapor. The most optimal carbonization regime for *Miscanthus* straw was found to be a calcination temperature at 400–500 °C for 30 min., leading to coal samples with high carbon content up to 85–90% by weight.

Activation of the obtained carbonizate for 60 min at a temperature of 800–850 °C with superheated water vapor allows obtaining activated carbon with a specific surface area of 541.9 m²/g, and with a specific pore volume of 0.232 cm³/g.

Activation of the samples allows increases the sorption capacity for iodine to 59.5%, and for methylene blue to 62.5%. A comparison of the physico-chemical characteristics of traditional BAU and activated carbon derived from *Miscanthus* straw showed comparable characteristics, which indicates the prospect of using the investigated coal in water treatment technologies. The obtained AC showed good adsorption properties to the ions of the studied TM. The adsorption capacity for copper, zinc and lead ions is 90, 100, 500 mg/l, respectively. The studies demonstrated that activated carbon materials produced based on *Miscanthus* straw as an attractive alternative to traditional coal sorbents.

Studies have shown high sorption capacities of metal ions (Cu, Zn, Pb) for the obtained AC from *Miscanthus* straw.

The inclusion of coal treatment in the complex of technological schemes of water purification will lead to a decrease in the content of heavy metal ions. Further the material may find a variety of industrial applications and hereby reducing costs compared to expensive sorption materials.

It should be noted that the present study compare the basic characteristics for the AC material with the requirements of GOST to BAC using a material close the *Miscanthus* applied in the present study. However, a direct evaluation of the present study to other methods for preparing AC is outside the scope of this study and left to a forthcoming study evaluation the preparation of AC from different source materials, like wood, coconut shells, apricot kernels and biochar, applying various activation techniques including both chemically and physically based techniques in comparison to the here reported AC preparation from *Miscanthus*.

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