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Study of the carbonization process for the plant waste utilisation

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Abstract. In agriculture and the food industry, large amounts of vegetable waste are generated annually: straw, corn waste, nut shells, fruit kernels, etc. The problem of their disposal is extremely acute, and regularly people use the simplest methods that do not require further processing of this type of waste. An efficient way of recycling is to make sorbents out of it. In modern conditions of deficit and rising commodity prices, the search for new, cheapest methods is becoming more and more urgent. The origin of vegetable waste is an excellent annual renewable raw material base. Walnut shells and corn cobs were used as raw material samples for the production of sorbents. The carbonization was carried out at temperatures of 300, 400, 500, 600 and 700 °C. Researched properties of raw materials: moisture, ash content and yield of volatile components. The dependency of the sorbent yield on the final temperature was determined, the ash content and the sorption activity by iodine were determined. Based on the results of the study, the high quality of the sorbent obtained was noted. In this way, the use of plant waste as raw material for the production of sorbents allows not only to solve the problem of waste recycling, but also to obtain a high-quality product and reduce its cost.

Key words: plant waste origin, utilisation, carbonisation, sorbents, the sorption activity.

INTRODUCTION

One of the most pressing problems of the modern world is waste utilisation. Their accumulation in Europe every year is growing and causes a significant negative impact to the environment. Not all types of waste are appropriate to recycle, some waste can be reused in the production of new products or as raw materials. Today the 'green' economy is developing at a great pace, and the main task is not disposal, but recycling in order to obtain new materials (Kshitij et al., 2022). Such a solution will expand the raw material base, intelligently recycle some of the waste and reduce the cost of the finished product.

Every year a huge amount of waste of plant origin and by-products of the agricultural industry is formed: fruit tree bones, nutshells, husks of cereal crops, acorn nuts, straw, corn, sunflower, bamboo waste, etc. Disposal of such types of waste requires additional costs (Koul, Yakoob, and Shah 2022). The transportation and removal from the field and garden of plant origin by-products (POB) are a significant part of the expenditures. The main method of disposal of such waste is incineration. This not only pollutes the environment, but also does not allow obtaining new recycled products. Due to the difficult environmental situation, in many countries the incineration of these types of waste is forbidden or strictly regulated (Wen et al., 2020), (Sharma et al., 2022). This process affects global warming and climate change, pollutes the air and water contributes to the accumulation of heavy metals in plants, etc. Not all waste lends itself to incineration, and not all of these methods are feasible. For example, the low specific gravity of biomass combustion complicates transportation and reduces the feasibility of this process. Walnut shells and fruit bones in general problematic to burn, while, this type of waste is an excellent renewable raw material for the production of carbon sorbents (Sharma & Dubey 2020; Wilk et al., 2020; Durga et al., 2022; Espinoza Pérez et al., 2022.

The raw material base of carbon sorbent is quite diverse - from wood to brown and hard coal. Their use is increasing rapidly. Nowadays, carbon sorbent is used in many processes of water purification, the food industry, in processes of chemical technologies. In addition, the purification of waste gases and wastewater is based mainly on the adsorption of carbon sorbents(Czerwińska et al., 2022). With the development of nuclear technology, carbon sorbent is the main adsorbent for radioactive gases and wastewater of nuclear power plants. It is also used in complex medical processes, such as hemofiltration (purification of blood on a carbon sorbent). In general, low-melting coal, peat and birch are used as raw materials to produce sorbents. Reduction of raw material resources is an acute problem in the modern world: production of hard coal is reduced; areas of peat bogs and birch forests are reduced. Thus, the expansion of the raw material base through renewable sources is relevant. Waste of plant origin is a good alternative (Ayinla et al., 2019; Chairunnisa et al., 2020; Babu et al., 2022; Lima et al., 2022; Matveeva & Bronstein 2022; van Nguyen et al., 2022; Su et al., 2022).

Various organic-origin raw materials are widely studied in the world (Table 2), but not limited to. In each country, in each region is appear organic - origin by products it could be as a raw material for carbon-based absorbents. For example: in China and Pakistan widely studied rise husk (Shen et al., 2019; Tauseef et al., 2022), in Nigeria and India coconut shell (Sruthi et al., 2019; Alabi & Sambo 2023), in Algeria cactus fruit and pomegranate peels (Sruthi et al., 2019; Akkari et al., 2022), at coastal-zones - reed (Patuzzi et al., 2013; Park et al., 2014; Yue et al., 2020) as organic-origin raw materials for pyrolysis with aim to obtain various applications carbon-based products.

A number of research dedicated to pollutant removal from the aqueous solution. Using of untreated biomass (Vincevica-Gaile et al., 2019; Irtiseva et al., 2021, 2022) for removing oils from water surface, natural and organic-origin by products for dyes adsorption from the aqueous solution: the adsorption of Orange Bezaktiv (SRL-150), onto diatomite thermally treated at 100 °C and at 300 °C was investigated in aqueous solution (Aguedal et al., 2017, 2019) was determined as 20.2 and 23.8 mg g^{-1}. Activated carbon prepared from industrial wastewater treatment plant dry sludge was proven to be efficient for the removal of refractory dye red scarlet nylosan (F3GL). The 434.78 mg g^{-1} for treated material and 169.49 mg·g^{-1} sorption ability for the unmodified material was determined (Li et al., 2016; Bensalah et al., 2018).

Production of activated coals from plant waste consists of two stages: carbonisation and activation. Pyrolysis is performed in an oxygen-free environment at temperatures of 400–850 °C. Even without the activation process carbonisate has sufficient sorption properties for its further use as a sorbent. The activation stage is carried out at higher temperatures of 600–900 °C which allows for a significant increase of the surface area of the sorbent due to the development of a porous structure. At the same time activation increases the cost of the finished product many times over.

The carbonisation process largely determines the yield of carbonisate and affects the formation of the porous structure (Wang et al., 2022). Temperature conditions and heating rate are the important criteria influencing the sorbent quality, which are individual for each type of raw material and depend on the initial characteristics (Tian et al., 2016; Ye et al., 2017; Celiktas and Alptekin 2019; Niu et al., 2021; Sirichan et al., 2022). For waste materials of plant origin carbonisation temperature is lower than the carbonisation temperature for hard coal and peat.

Well new Table 1 added with references to works dedicated to organic-origin raw materials pyrolysis and compared with this research. For example, the sorption capacity of the product obtained from corn cobs at 400 °C and at 600 °C does not differ significantly. It is presumably unprofitable to carry out the carbonization process at a higher temperature because it does not significantly affect the structure, and economically very costly.

Table 1. Characteristics of biochars produced from different feedstocks

Feedstock	Pyrolysis temp. (°C)	Yield (%)	Matter (%)	Ash (%)	Absorption capacity ($mg \cdot g^{-1}$)	Pore volume ($cm^3 \cdot g^{-1}$)	References
Corn cobs	200	71.53	-	-	39.22		(Nguyen et al., 2022)
Corn cobs	400	34.40	-	-	34.84	-	
Corn cobs	600	13.04	-	-	33.90	-	
Walnut shell	400	-	-	-	-	0.078	(Xu et al., 2022)
Walnut shall	500	-	-	-	-	0.150	
Walnut shall	600	-	-	-	-	0.296	
Walnut shall	700	-	-	-	-	0.695	
Walnut shall	550	-	-	3.72	-	-	(Xu et al., 2022)
Rapesed husk	200	47.14	-	-	-	-	(Elaigwu & Greenway 2016)
Rice Husk Char	-	-	9.04	53.47	-	0.16	(Konneh et al., 2021)
Coconut Husk Char	-	-	6.53	3.33	-	0.83	
Coffee Husk Char	-	-	4.42	4.54	-	0.296	
Bamboo	500–900	-	-	-	-	-	(Tong et al., 2020)
Oil palm fibers	529	-	-	-	-	0.222	(Ighalo, et al., 2022)

In this study we focused on the locally available organic-origin raw materials walnut shells and corn cobs. The main focus was on the sorption ability dependence on the carbonization temperature, as far as lower temperature lead to material self-cost decreas, but form the other hand it affecting (decreasing) sorption ability. The carbonization was carried out at temperatures of 300, 400, 500, 600 and 700 °C. Is necessary also determine moisture, ash content and yield of volatile components, the dependency of the sorbent yield on the final temperature.

MATERIALS AND METHODS

The choice of materials for the study and their characteristics

As raw materials for sorbent production, two types of POB were chosen: walnut shells (WS) and maize cob (MC). Samples were collected in the eastern part of Ukraine as waste after processing. Walnut and corn are common crops in Ukraine. Every year a large amount of walnut shells are formed as a result of its processing confectionery factories. Disposal of this type of waste is difficult and expensive, while the shells are an excellent raw material for the production of carbon sorbent. Corn is the most popular type of animal feed in Ukraine, part of the crop is used in the food industry. The stalk and leaves after harvesting are used as fodder for livestock, and the empty cobs without grains are not processed and burned.

Derivatographic study

The first stage of the study for WS was the dynamic thermal analysis of the sample. The study was carried out in a nitrogen medium on derivatograph Labsys evo SETARAM TG DTA DSC+1600 (SETARAM Instrumentation, France) in the temperature range from 20 to 600 °C with a heating rate of 5 $°C \cdot min^{-1}$, the sample weight 100 mg.

Sample preparation

Before carrying out the carbonisation process, the starting material was examined. Plant waste material was pre-dried in a desiccator at 60 °C for 4 hours. The dried CRM material was ground in the mill Knifetec 1095 (Tecator, Sweden) and dispersed into fractions. For further studies, the fraction of WS is from 2.00 to 2.5 mm was used, and the size of pieces of MC did not exceed 0.5×0.5 mm.

Determination of the moisture content

To determine the moisture content of the sample weight of 5 g in an aluminum sample box, pre-dried in a desiccator to a constant weight, was placed in a UF 110 (Memmert, Germany) for two hours at 105–110 °C. Then the boxes with the samples were placed in a desiccator and after 30 minutes weighed on an analytical scale KERN ABJ 220-4M (Kern, Germany) the moisture content was calculated by the formula:

$$\text{Moisture (\%)} = \frac{(m_1 - m_2)}{m_0} \tag{1}$$

where m_1 – initial mass, g; m_2 – moisture mass, g; m_0 – initial mass, g

Determination of the ash content

Ash content was determined in the muffle furnace LE 05111 (LAC, Czechia). Samples of 2 g in porcelain crucibles were placed in a muffle furnace at a temperature of 800 °C for 2 hours, after cooling in the desiccator samples were weighed on an analytical scale, ash content was calculated by the formula.

$$\text{Ash (\%)} = \frac{m_1}{m_2} \text{ x } 100 \tag{2}$$

where m_1 – weight, g; m_2 – oven dry weight, g.

Determination of the volatile matter

The determination of the volatile matter in a muffle furnace porcelain crucibles with lids. A sample of about 10 mL was weighed on an analytical scale in a crucible with a known mass. Then the crucible, covered with a lid, was placed in a muffle furnace heated to 800 °C for 4 − 6 min. The crucibles cooled in the desiccator were weighed and the index was calculated by the formula:

$$\text{Volatile matter (\%)} = \frac{m_1}{m_2} \quad (3)$$

where m_1 – weight of volatile, g; g = weight before heating – weight after heating and m_2 – oven dry weight, g.

The results of the analyses were taken as the arithmetic mean of the results of six parallel determinations, the absolute discrepancy between them does not exceed the allowable discrepancy of 0.5%.

Carbonisation

Carbonisation of vegetable waste was carried out in a horisontal tube furnace SUOL-0.25.1/12-I1(TermoPro, Ukraine) with a regulator final temperature and heating rate OMRON E5CB (TermoPro, Ukraine) in a nitrogen atmosphere (nitrogen consumption 60 $mL \cdot min^{-1}$). The sample with the plant material in a quartz capsule under the filling of calcined sand was placed in a through ceramic tube of a tubular electric furnace filled with nitrogen. The samples were carbonised at different temperature regimes and heating rates. The experiment yielded 5 types of carbonisates for each raw material. Carbonisation was carried out at atmospheric pressure and heating rates of 5 $°C \cdot min^{-1}$ (specimens designated as WS5; MC5) and 10 $°C\ min^{-1}$ (specimens designated as WS10; MC10) to the final heating temperatures of 300, 400, 500, 600 and 700 °C, followed by holding at the final temperature for 1 hour. The yield of the finished product was determined by the formula:

$$\text{Yield (\%)} = \frac{m_1}{m_2} \text{ x } 100 \quad (4)$$

where m_1 – mass of raw material, g; m_2 – mass of sorbent, g.

Characteristics of the sorbent: Iodine sorption activity

The sorption activity on the iodine of the obtained carbon materials was determined. A sample 1g was placed in a 250 cm^3 flask, 100 cm^3 of iodine solution in potassium iodide was added, corked and shaken on a laboratory shaker OS-3000 (Jeio Tech Co, Korea) for 30 minutes. After settling the solution, a 10 cm^3 aliquot was titrated with 0.1 H sodium thiosulfate solution, and a starch solution was used as an indicator.

Characteristics of the sorbent: Sorption activity by methylene blue

For the carbonysates obtained from WS, we carried out additional studies of sorption properties by methylene blue. The optical density was measured on a Specord 50 plus (Analytik Jena, Germany) spectrophotometer at 390−410 nm. The instrument was calibrated with methylene blue solution of various concentrations to build a calibration graph. A sample of carbon with 25 cm^3 of methylene blue solution at a mass concentration of 1,500 $mg\ g^{-1}$ was shaken in the OS-3000 apparatus for 20 min. Afterwards the mixture is separated in an LMC-4200R centrifuge. An aliquot of 1 cm^3 was diluted in a 100 cm^3 flask with distilled water to the mark and the optical density of the solution was measured. The adsorption activity of WS5 and WS10 sorbents was calculated by the formula:

$$X(\text{mg·g}^{-1}) = \frac{(C_1 - C_2 \times K)0.025}{weight\ of\ the\ sorbent}, \quad (5)$$

where C_1 – concentration of methylene blue solution, mg·dm^{-3}; C_{21} – concentration of methylene blue solution after contact with the sorbent, mg·dm^{-3}; K – solution dilution coefficient.

RESULTS AND DISCUSSION

With derivatograph the properties of WS and MC were studied. As can be seen from Fig. 1 and Fig. 2 in the temperature range of 100–210 °C endothermic effect is observed, which is associated with a release of some amount of water from the material.

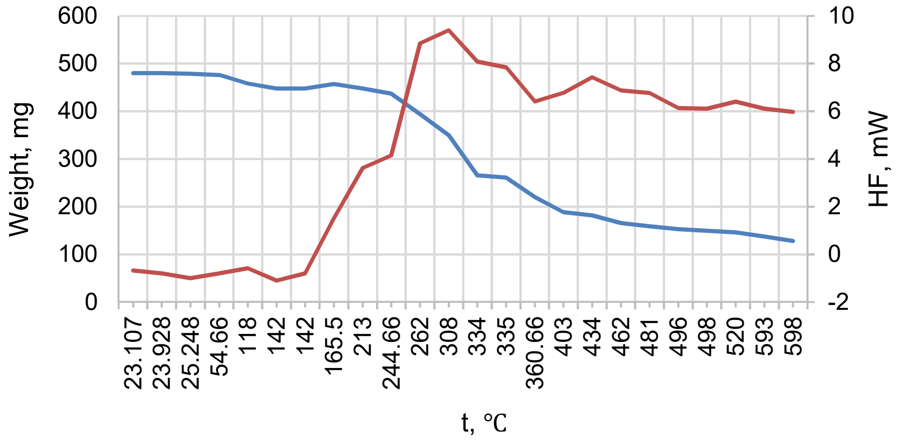

Figure 1. Thermogram of walnut shell decomposition.

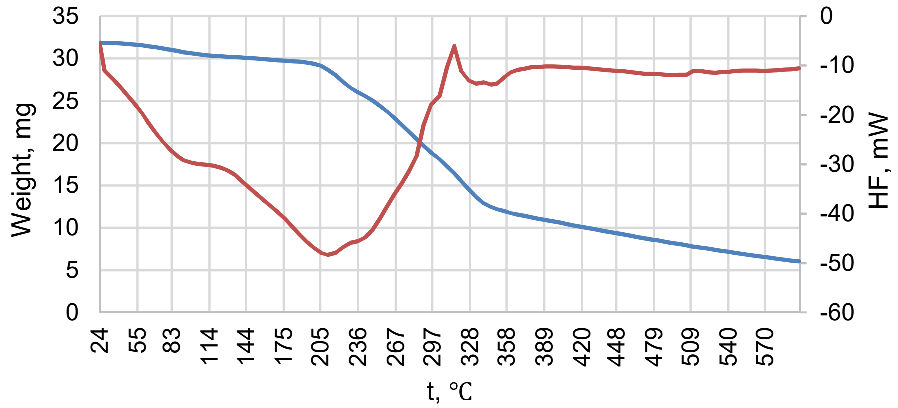

Figure 2. Thermogram of corn cobs decomposition.

On the TG curve for WS, mass loss averages from 8 to 9 wt.%, which is very close to the values obtained in determining the sample moisture content. Significant decrease in mass of the samples is observed in the temperature range of 250–410 °C and is more than 65%. The mass decrease of the samples corresponds to two pronounced exothermic maxima on the DTA curve at 250–300 and 350–400 °C temperature interval. On the TG curve for MC significant decrease in mass of the samples is observed in the temperature range of 220–450 °C. The mass decrease of the samples corresponds to two pronounced exothermic maxima on the DTA curve at 230–250 and 330–340 °C temperature interval. At temperatures above 450 °C, some insignificant decrease in mass is observed, which may indicate the absence of obvious chemical changes in the structure of the WS and MC samples.

Analysis of the raw materials

As follows from the data Table 2, the non-volatile carbon content of MC exceeds the non-volatile carbon content of WS by 10 wt.%. Humidity of the feedstock differs insignificantly, and ash content of MC exceeds ash content of WS by 4 times. It is obvious that WS has low ash content which increases its value as a raw material for production of carbon sorbents.

Table 2. Characteristics of raw materials

Raw materials	Moisture, wt.%.	Volatile matter, wt.%.	Ash content, wt.%.
Walnut shells	7.80 ± 0.02	78.00 ± 0.09	0.60 ± 0.02
Maize cob	4.90 ± 0.03	88.00 ± 0.07	2.50 ± 0.09

Carbonisation

The yield of the material does not vary significantly between each other, regardless of the final carbonisation temperature and heating rate Fig. 3. The effect of heating rate on the yield of the product is not significant, but still, there is. It should be noted that at a heating rate of 5 grad·min^{-1} the sorbent, yield is higher on average by 1–3 wt.% for both WS and MC.

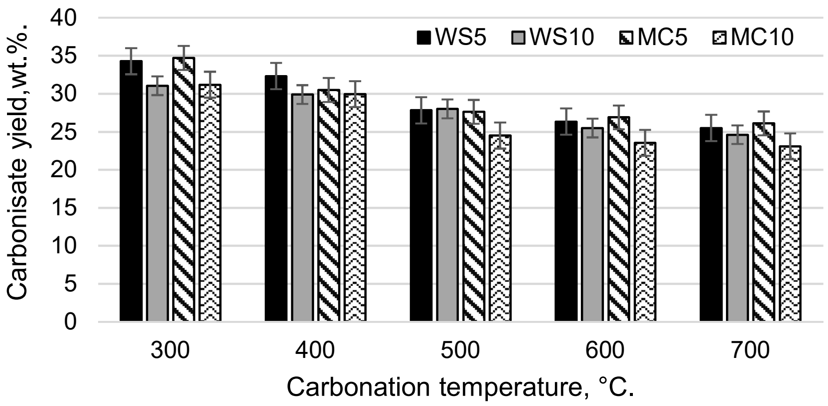

Figure 3. Dependence of the material yield on the final carbonisation temperature.

Ash content of the sorbents.

The ash content of the sorbents obtained is shown in Fig. 4. The results showed that the ash content of the sorbent obtained from WS increased by 0.71 wt.% with an increase in the coking temperature at a heating rate of 5 grad·min^{-1} and at a heating rate of 0.71 wt.% by 10 grad·min^{-1} by 0.69 wt.%.

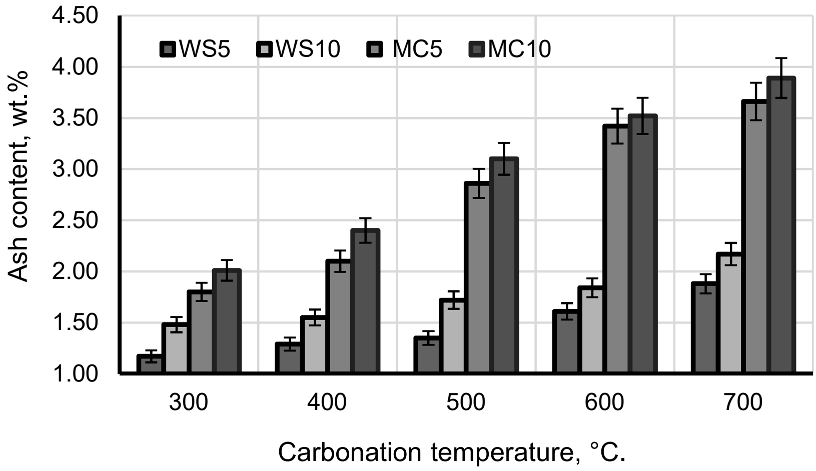

Figure 4. Dependence of ash content of carbonisate on final temperature of raw material heating.

Consequently, an insignificant increase in the ash content of the WS sorbent with an increase in the coking temperature is evident. Increasing the heating rate increases the ash content of the sorbent, but insignificantly. For MC-derived materials, the change in ash content is more significant. MC sorbent obtained at a heating rate of 5 °C·min^{-1} contains more ash than a sorbent obtained at a heating rate of 10 °C·min^{-1}. This difference is more significant than WS sorbent obtained at the same heating rate. The low ash content of the carbon material compared to the data in (Adekanye et al., 2022) is due to carbonization in a nitrogen environment. The low ash content of the sorbent increases its value.

Iodine sorption activity

Fig. 5 shows the sorption activity of the sorbents for iodine. Sample WS10 showed the worst sorption properties compared to other samples. Reducing the heating rate to 5 °C min^{-1} allows better sorbent surface area development for iodine solution sorption. WS5 obtained at 500, 600 and 700 °C relative to WS10 obtained at the same temperatures has 8% higher sorption activity: 2.3% and 8.3%, respectively. MC sorbent absorbs iodine better than WS sorbent; at 500 °C and 600 °C the sorption activity is twice as high. With an increase in the final temperature of carbonization, the sorption activity of sorbents increases, which indicates a more developed surface area of the carbon material obtained.

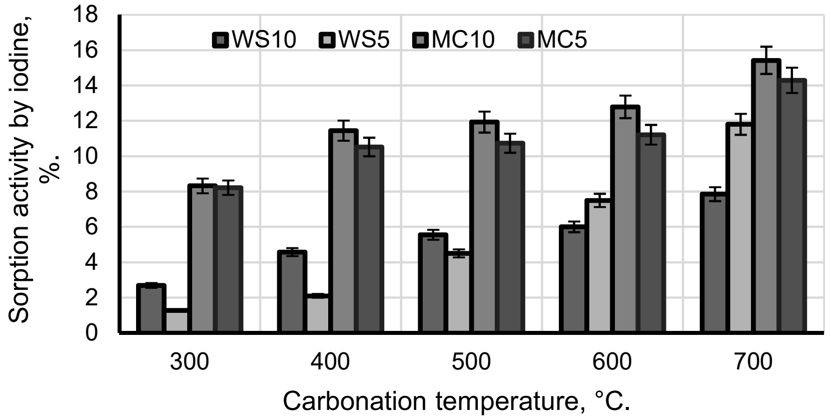

Figure 5. Dependence of iodine sorption activity on carbonisation temperature.

Methylene blue sorption activity

It was decided to conduct additional studies to evaluate the quality sorption properties by methylene blue. The results of the study are shown in Fig. 6. A study of samples from MC showed that the samples obtained have greater sorption activity than samples obtained from WS under the same conditions. The coconut shell sorbent after activation with $ZnCl_2/CO_2$ (Baysal et al., 2018) absorbs methylene blue worse than the inactive carbon sorbent obtained from the walnut shell. As the carbonization temperature is increased, the sorption properties of methylene blue increase. A low heating rate makes it possible to obtain a more developed sorbent surface and increase its sorption properties.

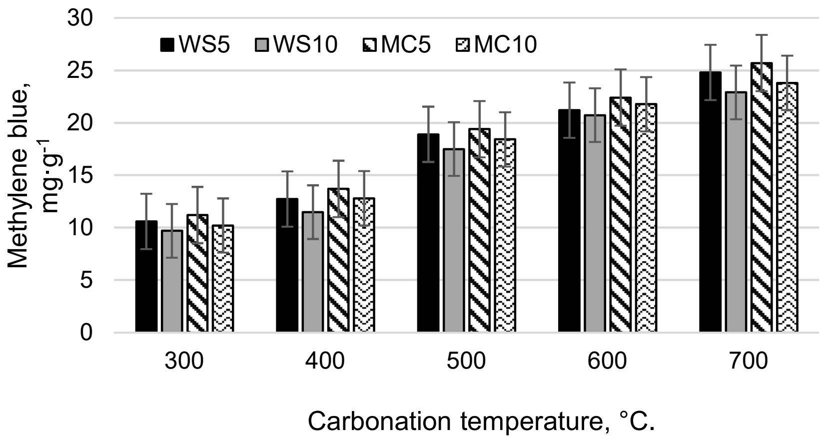

Figure 6. Dependence of methylene blue sorption activity on the carbonisation temperature.

CONCLUSIONS

Wastes of plant origin: Walnut shells and corn cobs were pyrolyzed in a tubular electric furnace in nitrogen medium at different final heating temperatures (300–700 °C) and heating rates of 5 and 10 $°C \cdot min^{-1}$. The physicochemical and sorption properties of the obtained sorbents were studied. The results show that the carbonization temperature and the heating rate had a significant impact on the surface development of the carbon material and its sorption properties. These plant wastes are good raw materials for carbon sorbents. Non-activated carbonates can also be used as inexpensive sorption materials. This way of recycling will expand the raw material base of sorbents and produce renewable and high-quality raw materials every year, as well as reduce the sorbent deficit in the market, which is increasing every year.

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