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A NEW METHOD FOR CONVERSION WASTE BIOMASS INTO USEFUL PRODUCTS WITH WIDE APPLICATION POSSIBILITIES

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ABSTRACT

This study investigates a thermochemical method for the conversion of industrial biomass residues - specifically peach shells, walnut shells, and cherry stones - into high-value commodities. By employing flash pyrolysis across a range of temperature gradients, a waste-free processing cycle was established, yielding solid, liquid, and gaseous products.

The research focuses on identifying the optimal process parameters and characterizing the physicochemical properties of the resulting fractions. Results indicate that the yield and molecular composition of the products are highly dependent on the precursor's botanical origin and the specific pyrolysis temperature.

Keywords: biomass composition, flash pyrolysis, circular economy.

INTRODUCTION

The growing population of the earth requires an increase in agricultural production, which in turn is associated with an increase in agricultural waste. Biomass wastes are valuable feedstocks for producing products like the chemicals [1], bioactive compounds [2], biomaterials (composites, filters and films) [3], supercapacitors [4], bacterial and fungi immobilization [5], amendments [6], and biosorbents [7] to biofuels [8, 9]. Biomass contains compounds that can be extracted from the biomass by solvents and are not part of the structure itself [10].

Due to the increasing amount of this waste, it is necessary to treat and manage it properly to avoid the release of various emissions and greenhouse gases when it is burned. To avoid the environmental problems associated with the disposal of waste, various processes related to its processing are being developed [11].

Commonly used method for using agricultural waste is incineration (a thermochemical process that burns waste in a high-temperature and oxygen-rich environment). If the toxic pollutants released are controlled, incineration can be a potentially effective way to deal with agricultural waste. Unfortunately, more than 60 % of incinerators around the world do not apply such controls, resulting in toxic emissions into the environment (such as particulate matter (PM), acid gases, heavy metals, dioxins, furans and greenhouse gases).

The burning and landfilling of agricultural waste contribute to about 3 % of global greenhouse gas emissions. At the same time, they are potential raw materials to be converted into resources to produce

useful products such as gaseous fuel, liquid product (a mixture of organic compounds) and solid product with carbon adsorbent properties. The need to find resources and environmental sustainability leads to an increasing desire to move from a linear to a circular economy, creating a closed loop from agricultural production to agricultural waste management.

Thermochemical conversion is emerging as a potential way to create a reuse method (circular economy). This method can produce energy, chemical compounds and carbon adsorbent from agricultural waste. It is estimated that the pollutants released during this processing are at least 50 % less compared to landfilling and incineration.

In this sense, thermochemical conversion can be considered an important strategy for converting the organic mass of agricultural waste into useful products such as energy sources and carbon materials [12].

The direct burning is the most common method for receiving energy from waste biomass, with 90 % of the energy from biomass being received through combustion. One of the main requirements is the moisture content in waste from biomass to be under 50 %. The presence of nitrogen and sulfur oxides in the combustion flue gases is a major disadvantage of this method [13]. In addition to combustion, other processes have been developed for the utilization of waste biomass to obtain energy sources that pollute the environment to a significantly lesser extent and to obtain other useful products with important applications. The main developed processes are:

Gasification

Gasification is the thermo-chemical conversion of a carbonaceous fuel at high temperatures, involving partial oxidation of the fuel elements [14]. The aim of the processing is to achieve the conversion of the maximum possible part of the organic mass of the biomass into synthesis gas, containing mainly hydrogen and carbon monoxide, through incomplete oxidation at very high temperatures. The main disadvantage of gasification is the very high temperature of conduction and the production of tar, ash and polluting gases, which create a problem for environmental protection.

Liquefaction of waste biomass

The purpose of processing is to obtain biofuel, also called biocrude oil. It is a thermochemical or hydrothermal processing. The average yield of biocrude oil is over 70 %. The main parameters that influence the process are the temperature (between 250 and 360°C), material composition, pressure (between 10 and 25 MPa) and time (between 5 and 120 min). Compared to the liquid product obtained from biomass pyrolysis, biocrude oil has the advantages of lower moisture content and high calorific value, making it more suitable for industrial applications. It must be further processed to improve its quality (energy density, minimal by-products) in order to be a suitable substitute for petroleum and diesel. However, this further processing involves catalytic cracking, which requires additional energy input [15].

Pyrolysis

Pyrolysis is used to convert biomass waste into various products. Pyrolysis is a high-temperature treatment in which the organic mass of biomass waste is destroyed under anaerobic conditions. Depending on the rate of temperature rise and the final temperature of the process (between 400°C and 1000°C), pyrolysis can be divided into slow, fast and flash. During the process, decomposition and synthesis processes occur simultaneously, resulting in gaseous, liquid and solid products. The liquid product contains various organic compounds, with oxygen-containing ones with hydroxyl, carboxyl and carbonyl groups prevailing. The gaseous product includes H_2, CH_4, CO and CO_2. The gaseous products obtained by pyrolysis of biomass can be refined and processed into ecological fuels such as hydrogen and syngas as a raw material to produce hydrocarbons. The solid product is the only one that does not require reprocessing after pyrolysis of biomass. The composition and ratio of the products depend on the starting material, the final processing temperature and the rate of temperature rise [15, 16]. Depending on the results of product analyses, a suitable area for their application can be sought [17].

Torrefication of biomass waste

A type of waste biomass processing like pyrolysis (light pyrolysis) but carried out at a much lower temperature (between 200 and 300°C). The main goal of this processing is to improve the quality of the processed biomass by increasing the calorific value and hydrophobicity, reducing the oxygen and hydrogen content. Due to the use of a lower processing

temperature, torrefaction is less energy-intensive and has a smaller environmental footprint. However, the resulting tar is toxic to humans and ecosystems [18]. Biochar produced by torrefaction of agricultural waste can be used as a solid biofuel and incorporated into other materials, promoting a circular economy, reducing carbon emissions, and contributing to environmental sustainability [19].

As can be seen from the above, it is necessary to work on improving the developed methods for processing on biomass and the effective application of the resulting products.

The aim of this work is to implement a circular economy through:

All thermal processing described in the article are known and have been applied repeatedly to various raw materials. However, research continues with the aim of expanding the range of raw materials used. It is necessary to determine the influence of the chemical composition and texture of the raw material on the properties of the final products and determine the optimal processing conditions depending on the purpose of the final products. These studies are necessary for each selected raw material. In this work, three wastes from the processing industry were selected to establish the applicability of flash pyrolysis specifically for them for their processing into useful products. Much attention has been paid to the properties and applicability of the various gaseous, liquid and solid products, which is important information for the efficiency and applicability of processing specifically for them. More serious attention has been paid to the solid product, unlike many other studies in this field which are aimed at obtaining liquid and gaseous products.

EXPERIMENTAL

Flash pyrolysis of waste biomass

50 g cherry stones, peach and walnut shells with a grain size of 1 - 5 mm are placed in a reactor, heated to the desired temperature and left at this temperature for 30 min. The volatiles and solid products are cooled. The liquid and gas products are collected.

Methods for characterization of raw materials and resulting products

Thermal characterization was performed using a NETZSCH STA 449 F3 Jupiter analyser (Selb, Germany). The system allows for simultaneous TG/DSC analysis with a weighing sensitivity of 0.1 μg and adjustable heating rates from 0.001 to 50 K min^{-1} within a 25°C - 1400°C range. Experimental parameters included the use of corundum, aluminium, or platinum crucibles, with a specialized clamp for liquid samples. All tests were conducted under an argon atmosphere using sample masses between 10 and 25 mg.

Elemental composition analysis (C, H, N, S) was conducted using a Vario Macro Cube analyzer (Elementar Analysensysteme GmbH, Langenselbold, Germany), with oxygen content determined by calculation. The system operates by combusting 1 - 50 mg samples, encapsulated in tin foil, within an oxidation environment heated to 1150°C. The resulting ash is retained in a quartz tube, while the gaseous combustion products - CO_2, H_2O, SO_2, and various nitrogen oxides - are directed through a copper-catalyst reduction column, which facilitates the conversion of nitrogen oxides into N_2.

The gaseous mixture is subsequently passed through a series of specialized adsorption columns that selectively trap CO_2, H_2O, and SO_2. A thermal conductivity detector (CVD) then sequentially quantifies these species by measuring gas flow conductivity. Nitrogen is analyzed last, and the instrument utilizes these high-precision conductivity data (with an absolute standard deviation < 0.1% for C, H, N, and S) to calculate the final weight percentage (wt. %) of each element in the sample.

Textural properties of the synthesized carbon samples were evaluated via nitrogen adsorption isotherms measured at -196°C, using a Quantachrome Autosorb iQ-C-XR/MP automatic volumetric system (Boynton Beach, FL, USA). To ensure the reliability and reproducibility of the BET surface area and pore volume calculations, the instrument features a pressure measurement accuracy of 0.1 % of the full scale. Prior to analysis, all samples were subjected to overnight vacuum degassing at 350°C. The acquired isotherms served as the basis for determining the specific surface area (SBET), total pore volume (Vtotal), and micropore volume, with the latter estimated through the T-plot method [20 - 23].

The fracture surfaces of the composite materials were examined via scanning electron microscopy (SEM) using a JEOL JSM-6390 (Japan) instrument. Specimens measuring 3 mm by 5 mm were prepared to facilitate detailed observation of the fracture topography. To

enhance imaging quality and conductivity, the samples were gold-coated (10 Å layer) utilizing a sputtering system under an argon atmosphere. High-resolution imaging, with a spatial resolution of 3.0 nm, was employed to obtain accurate representations of the pore morphology and structural features. Throughout the characterization process, samples were maintained under vacuum conditions to ensure detector efficiency and to capture high-quality, representative micrographs of the fracture zones.

Raman spectroscopic analysis was performed on a Bruker Senterra II spectrometer (Bruker GmbH, Denkendorf, Germany) using a 532 nm laser at 6.5 mW. Approximately 10 mg of each sample was analysed on a glass substrate in a 180° backscattering geometry with a 20× objective, 1 cm^{-1} resolution, and 100 s exposure time. This non-destructive technique provided high-resolution insights into the microstructure of the carbon materials, including defect density, layer orientation, and doping levels.

The iodine number indicates the porosity and surface area of the activated carbon, and it is defined as the amount (mg) of iodine adsorbed by 1 g of carbon. The iodine adsorption was determined using a sodium thiosulfate volumetric method.

Adsorption towards methylene blue was determined spectrophotometrically [24].

The pH of the carbon samples was determined using the Standard NORIT procedure. Briefly, 4.0 g of the non-dried, pulverized carbon material was added to 100 mL of distilled water in a 250 mL beaker. The suspension was boiled for 5 min, after which the supernatant was decanted at a temperature of 60°C. The collected liquid was subsequently cooled to 25°C before measuring the pH value with a precision of ± 0.1.

RESULTS AND DISCUSSION

DTG-DSC analysis of the selected raw materials (shells from peaches and walnuts and stones from cherries). The data from this analysis are very informative about the dynamics of thermal destruction of the samples and the nature of the chemical reactions occurring during thermal treatment. The dynamics of thermal destruction also provides information about the chemical composition (content of hemicellulose, cellulose and lignin) in the sample [25]. The results obtained from the thermal analysis of the selected raw materials are very useful information when choosing the optimal thermal treatment regime for the carbonization process. The results of the analyses are presented in Fig. 1, 2 and 3.

The comparison of Fig. 1, 2 and 3 shows differences in the kinetics of thermal destruction of the samples and the chemical reactions occurring during thermal treatment. Lignocellulosic materials show a noticeable decline on the curve between 250°C and 300°C, which shows decomposition on hemicellulose. Cellulose and lignin begin the process of decomposition respectively at 350°C to 400°C and 250 to 600°C [26, 27]. Observes loss on table starting from 100°C. This is due to evaporation of external and constitutional moisture of the raw materials. In peach shells, a decrease in the curve is observed at 210°C, which is due to the degradation of hemicellulose.

In peach stones, there is a drop at 300°C, which is due to the beginning of cellulose decomposition. In cherry stones and walnut shells, this drop is barely noticeable. The drop at 360°C in all raw materials indicates the decomposition of cellulose and lignin. The presence of a drop above 420°C in cherry stalks, which is not observed in the other two raw materials, is an indication of the higher content of lignin, which is a highly cross-linked polymer, which is the cause of its decomposition at high temperatures. The noted differences in the chemical composition and behaviour of the selected raw materials during thermal treatment led to the production of products with different properties under the same other conditions. The behaviour of the raw materials during heating provides important information when choosing an appropriate one.

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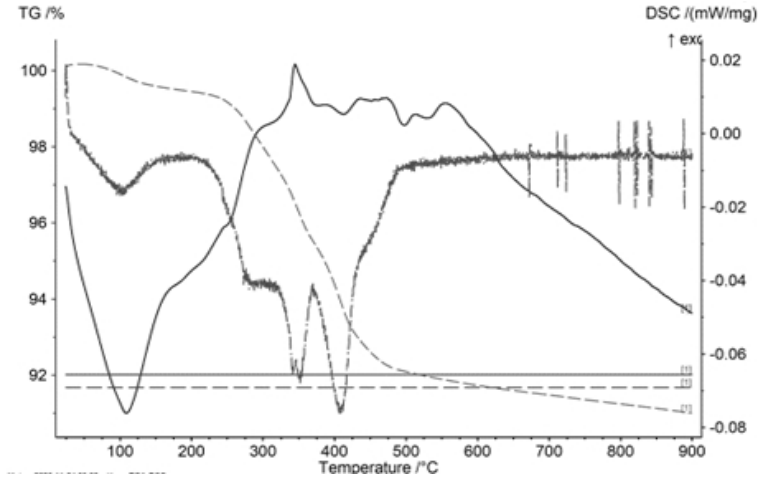


Fig. 1. Thermal analysis (TG-DSC) of cherry stones (dash line-TG, solid line-DSC).

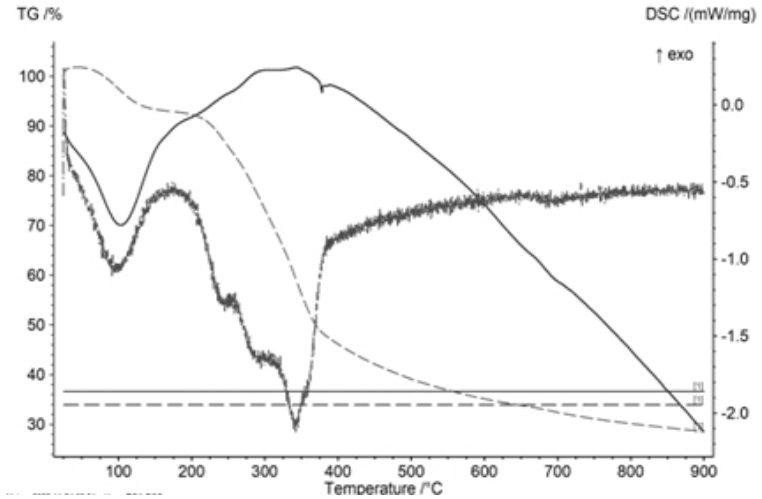


Fig. 2. Thermal analysis (TG-DSC) of walnut shells (dash line-TG, solid line-DSC).

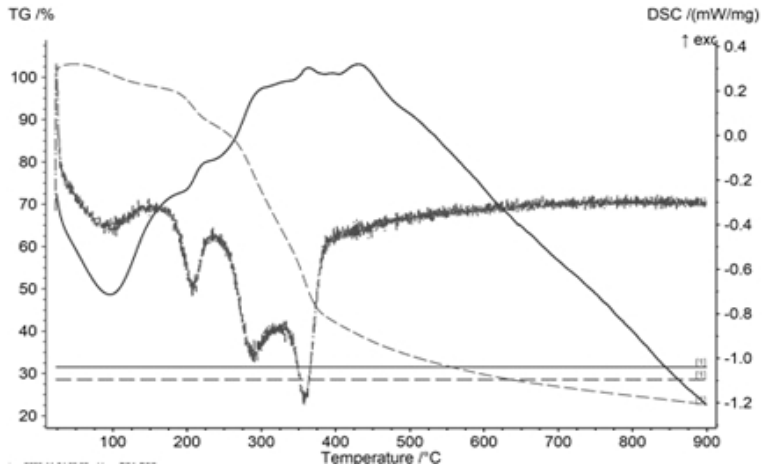


Fig. 3. Thermal analysis (TG - DSC) of peach shells (dash line-TG, solid line-DSC).

heating provides important information when choosing an appropriate processing mode. However, it should be borne in mind that the kinetics and depth of decomposition of the organic mass of the raw materials may undergo serious changes when subjecting the raw materials to flash pyrolysis, especially at higher temperatures. Subjecting the raw material to thermal shock will lead to changes in the dynamics of the thermal destruction of the organic mass of the material. It is necessary to study the influence of the temperature of the processing of the raw materials on the composition and properties of the resulting solid, liquid and gaseous products.

In this regard, a study was conducted on the influence of flash pyrolysis temperature on the quantity, composition and properties of the obtained solid, liquid and gaseous products. The material balance of the flash pyrolysis treatments of the selected raw materials at different temperatures was determined. The results are shown in Table 1.

The data in Table 1 show that at lower flash pyrolysis temperatures, the highest yield of solid product is obtained from cherry stones. This is due to the higher amount of lignin in them. At higher temperatures, the yield of solid product for different raw materials is similar. The amount of liquid product obtained from flash pyrolysis at different temperatures is slightly higher for peach stones. This may be due to the content of less heat-resistant structures. It can be noted that the higher amount of gas products from cherry stones at the highest processing temperature. This is probably due to the continued destruction of more heat-resistant aromatic structures formed by the decomposition of lignin. More interesting from a practical point of view is the change in the ratio of solid, liquid and gas products with increasing flash pyrolysis temperature. For all raw materials, with increasing temperature, the yield of solid product decreases and the amount of liquid and gas products increases. The reason for this is the higher speed and depth of thermal destruction at high temperature and the faster removal of the resulting compounds from the reaction space, which does not allow the occurrence of synthesis reactions between the resulting radicals. The results obtained are useful for choosing the optimal flash pyrolysis temperature depending on the interest in the different products.

Table 2 shows the results of the technical and elemental analysis of the starting raw materials and the carbon adsorbent obtained on their basis. The lowest ash content is shown by peach shells and the adsorbent obtained from them. The greatest decrease in oxygen content and, accordingly, an increase in carbon content is also observed in peach shells. This indicates a greater destruction of oxygen-containing structures with the formation of a larger amount of reactive free radicals that participate in the synthesis of condensed aromatic hydrocarbons. This is also confirmed by the lowest hydrogen content in the carbon adsorbent obtained from peach shells.

Table 3 presents the yield, pH and adsorption properties of the solid products obtained from flash pyrolysis of the raw materials. The results show very close values for the surface area of the solid products obtained from walnut and peach shells. The same applies to the iodine number, which is often used as a preliminary guide to the surface area of a given sample.

Table 1. Material balance of flash pyrolysis of raw materials at different temperatures on the number of products obtained.

Sample	Temperature, °C	Amount of solid product, %	Amount of liquid product, %	Amount of gas products, %
Cherry stones	600	27.7	22.1	50.2
	700	24.0	23.5	52.5
	800	21.3	24.1	54.6
Walnut shells	600	26.5	22.0	51.5
	700	24.1	23.3	52.6
	800	22.1	24.2	53.7
Peach shells	600	26.1	23.2	50.7
	700	24.5	24.3	51.2
	800	21.6	24.6	53.8

Table 2. Chemical composition of the raw materials and the solid products obtained at 800°C flash pyrolysis.

Raw materials solid products	Proximate analysis, wt. %			Elemental analysis, wt. %				
	W	Ash	Vol.	C, wt. %	H, wt. %	N, wt. %	S, wt. %	O, by diff.
Walnut shells	6.85	0.61	79.23	51.25	5.98	0.22	0.13	42.42
ACwalnutshells	3.11	2.23	3.22	81.11	1.29	0.18	0.34	17.08
Peach shells	6.31	0.33	79.88	51.52	6.13	0.23	0.18	41.94
ACpeachshells	1.71	1.23	3.41	87.01	0.99	0.29	0.33	11.38
Cherry stones	4.81	1.53	78.28	54.19	7.13	0.44	0.28	37.96
ACcherrystones	1.72	2.98	4.31	85.11	1.57	0.89	0.38	12.05

Table 3. Yield and adsorption properties of solid products obtained from flash pyrolysis of selected raw materials.

Raw material	Surface area, $m^2\ g^{-1}$	pH	Adsorption, mg g^{-1}	
			Iodine	Methylene blue
Peach shells	610	7.2	650	216
Cherry stones	475	7.5	512	155
Walnut shells	593	7.1	598	172

The data on the adsorption of iodine and methylene blue show that the solid products obtained from flash pyrolysis of the selected raw materials have good adsorption properties, which makes them suitable for use in purification methods.

Fig. 4 shows N_2 (77K) adsorption isotherms of carbon adsorbents obtained by flash pyrolysis from agricultural waste products. The N_2 isotherms character (a step increase with tendency for saturation) is indicative of the microporosity of the received carbons. The course of the isotherms shows that the three adsorbents have a predominantly microporous texture and the presence of a smaller amount of mesopores. The adsorbents obtained from peach and walnut shells have a similar surface area, while the adsorbent from cherry stones is characterized by a lower one. The increase after P/P_0 0.95 suggests the presence of macropores in the three adsorbents.

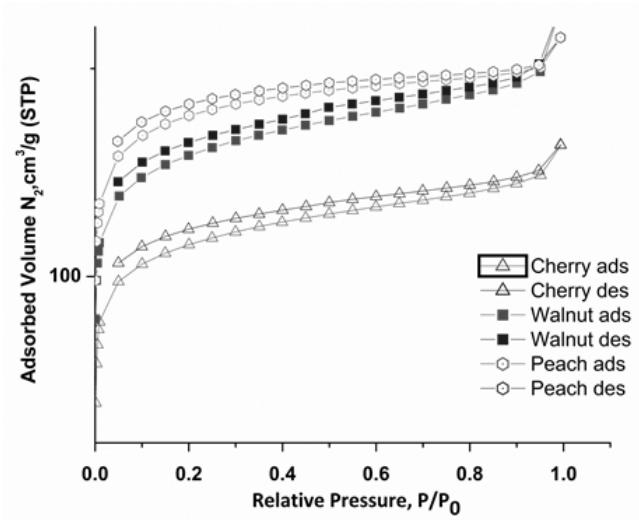


Fig. 4. N_2 (77K) adsorption isotherms of carbon adsorbents obtained by flash pyrolysis from agricultural waste products.

The Fig. 5, 6 and 7 show that the three adsorbents have a pronounced microporous texture, with the largest amount of micropores with very small sizes in the adsorbent obtained from cherry stones. In the adsorbents from peach and walnut shells, micropores with larger sizes predominate. The amount of mesopores is largest in the adsorbent from walnut shells.

The data shows that other things being equal the differences in the porous texture of the obtained carbon adsorbents are due to the difference in the chemical composition and texture of the raw material from which they were obtained. The data show that the best characteristics are shown by the carbon adsorbent obtained from peach shells. Therefore, the remaining studies were conducted on it.

The Raman spectrum of AC from peach shells is shown in Fig. 8. The bands at around 1360 cm^{-1} and 1580 cm^{-1} are the D-peak and G-peak of carbon

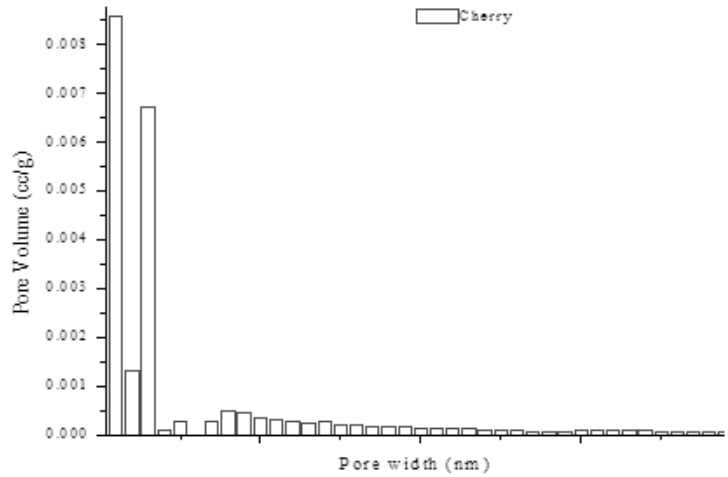


Fig. 5. Pore size distribution of the AC cherry stones.

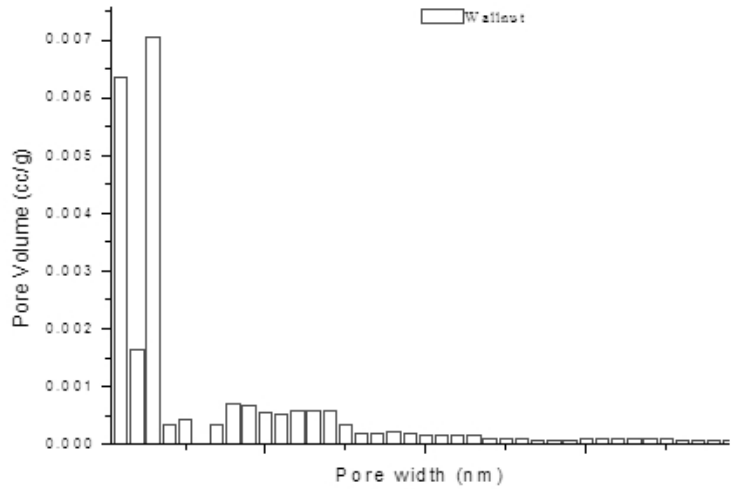


Fig. 6. Pore size distribution of the AC walnut shells.

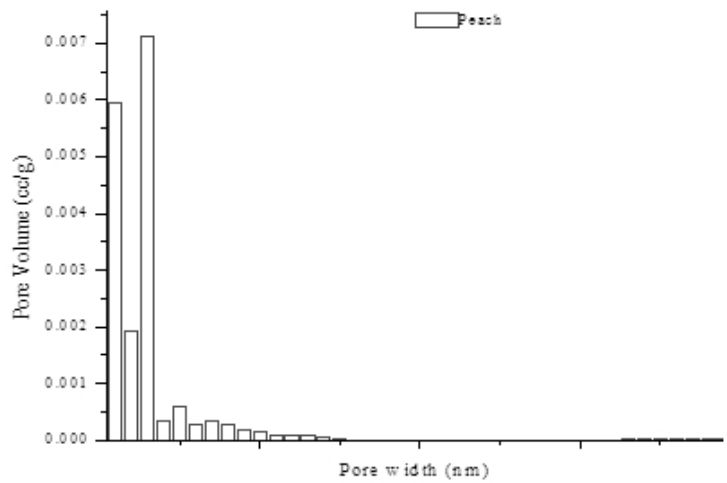


Fig. 7. Pore size distribution of the AC peach shells.

material, and the D-peak represents the defect of the C atom lattice which represented disordered vibration carbon, and the G-peak represents the in-plane tensile vibration of the sp^2 hybridization of the C atom which represented the ideal graphite lattices. The intensity ratio of D and G peaks (ID/IG) was calculated to quantify the graphitized degree of AC. The ID/IG ratio of the sample was 0.788, indicating a higher degree of graphitization in the sample.

The SEM micrograph of the obtained carbon adsorbent (Fig. 9) clearly shows that it has a dense microporous texture, with cracks in the samples, characteristic of well-developed activated carbon cells. An important task of the current study is to determine the areas of possible application of the resulting solid, liquid and gaseous products obtained because of flash pyrolysis of various raw materials.

The data obtained so far from the analyses of solid products from flash pyrolysis of waste biomass show that they have the properties of a carbon adsorbent with a relatively well-developed porous texture and adsorption capacity. This means that they have the applicability characteristic of carbon adsorbents in many branches of method.

The investigations on the composition of the tar from flash pyrolysis of the three raw materials showed that the main part of them is oxygen containing compound - derivatives of phenol, guaiacol, veratrol, syringol, free fatty acids and esters of fatty acids. The experiments showed that during prolonged residence of liquid products, because of the ongoing polycondensation reactions their rheology changes significantly. These structures with appropriate processing can be involved in polycondensation and polymerization processes and can be used for the fabrication of monolithic carbons with different properties. Research has shown that with appropriate processing, carbon foam can be obtained from the obtained liquid product (Fig. 10).

The data on the chemical composition of carbon adsorbents obtained by flash pyrolysis of peach shells shows that they contain mainly carbon and oxygen. The remaining elements are of mineral origin and in much smaller quantities.

Table 5 presents the composition of the gas products obtained because of the flash pyrolysis of peach shells.

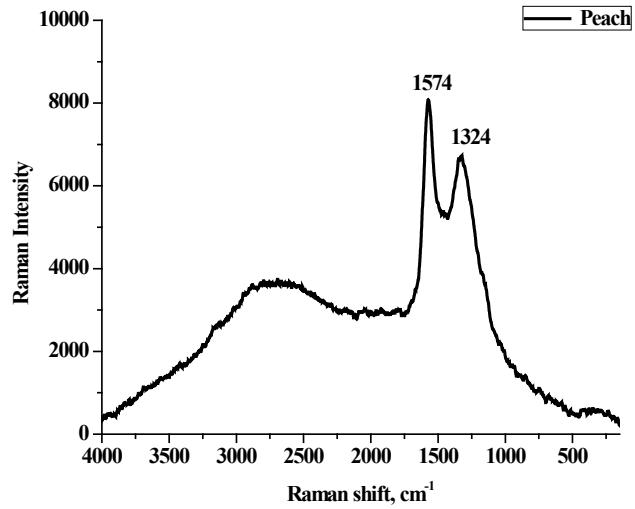


Fig. 8. Raman spectrum on carbon adsorbent, obtained from peach shells.

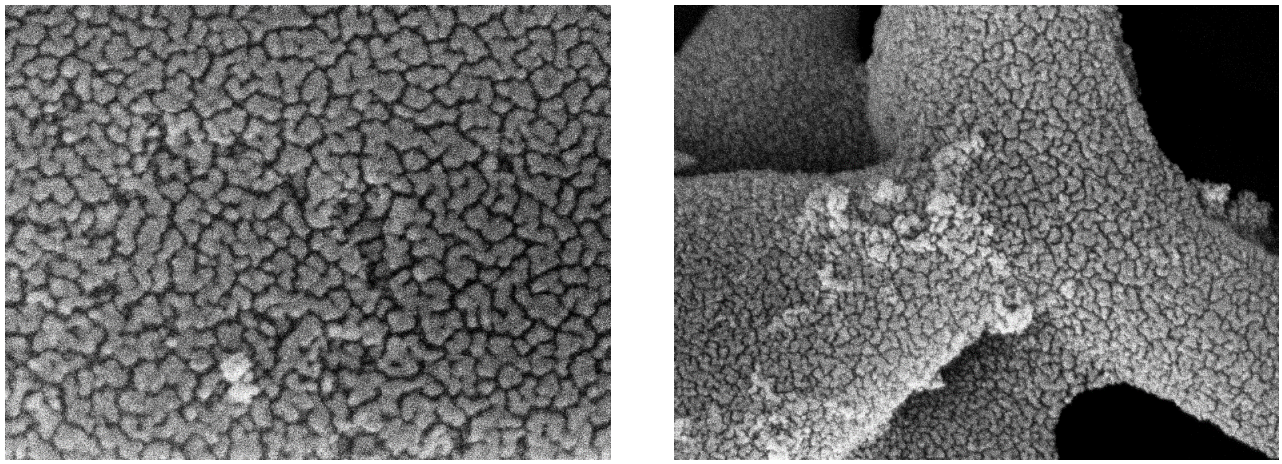
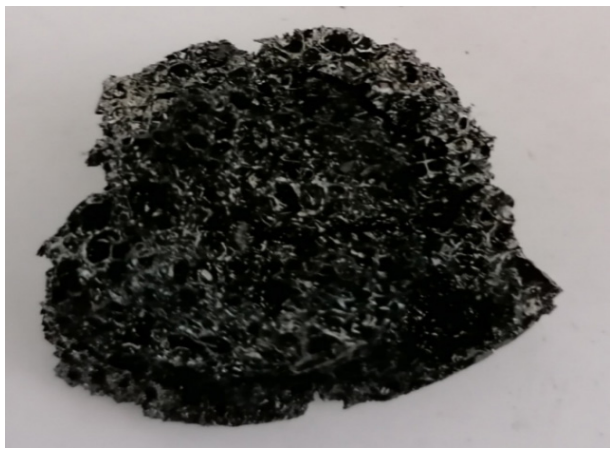


Fig. 9. SEM micrograph of a carbon adsorbent derived from peach stones.



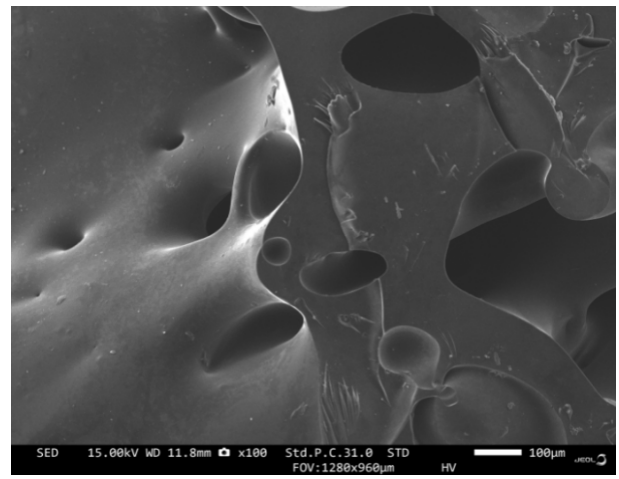


Fig. 10. Photograph and SEM of carbon foam obtained from liquid products of flash pyrolysis of peach shells.

Table 4. Chemical composition of carbon adsorbent obtained by flash pyrolysis of peach shells EDS analysis.

Wt., %	C	About	Na	Mg	Cl	K	Ca	Fe	Total
AC	79.14	16.17	1.24	0.22	0.78	1.12	1.07	0.26	100

Based on the data obtained, several possibilities for the application of the gas products can be noted. The high percentage of carbon and methane in the gas products indicates the possibility of using them as a source of hydrogen. Another possibility for their application, based on the relatively large amount of hydrogen and carbon monoxide, is to use them as a source of synthesis gas. Through appropriate catalytic processing, hydrocarbons can be obtained from the synthesis gas through the Fischer-Tropsch (FT) synthesis [28]. Considering the high calorific value of the gas, it can also be noted the possibility of using it as a source of energy.

Table 5. Composition of gas obtained in flash pyrolysis of peach stones at 800°C.

Component	vol. %
Hydrogen (H_2)	34.3
Methane (CH_4)	12.1
Ethane (C_2H_6)	0.1
Propane (C_3H_8)	0. 3
Carbon monoxide (CO)	37.7
Carbon dioxide (CO_2)	15. 5

CONCLUSIONS

A waste-free method for conversion by flash pyrolysis, allowing a fast-processing process with high productivity, of peach shells, walnut shells and cherry stone to useful solid, liquid and gaseous products has been developed. It has been determined that the solid products have properties of carbon adsorbents, the liquids are a suitable raw material for the synthesis of carbon materials, the gaseous products can be used as a source for obtaining hydrogen, synthesis gas and a source of energy. It has been established that the properties of the final products are influenced by the chemical composition of the raw material and the temperature of its processing. The results show that by using appropriate raw material and the temperature regime, final products with different properties can be obtained. The analysis of obtained products allowed the definition of practical applications for each of them.

The results obtained indicate that flash pyrolysis is a suitable method for the conversion of waste biomass to useful products.

The results of the research conducted are an indication of the potential for implementing the developed method in the principles of the circular economy.

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Authors' contributions

I.S., B.P., P.D., N.P., B.Ts.: Conceptualization; I.S., B.P., N.P., B.Ts., B.R., A.K., P.D.: Methodology; I.S., B.P., N.P., B.Ts., A.K., B.R.: Investigation; I.S., B.P., N.P., B.R.: Writing - original draft preparation; I.S., B.P., N.P., B.Ts., A.K., P.D.: Writing - review and editing; I.S., N.P., B. Ts., P.D.: Supervision; I.S., B.Ts., P.D.: Project administration; I.S., B.Ts., N.P., P.D.: Funding acquisition. All authors have read and agreed to the published version of the manuscript.

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