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Determination of Minor Elements in Walnut Shells (*Juglans regia* L.)

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The content of individual macro- and micro-elements in solid biofuels has a significant impact on the processes involved in their combustion. An excessively high content of certain elements can lead to the formation of sludge or corrosion of furnaces. It is therefore important that a raw material used for energy purposes should meet the requirements contained in specialised standards for the energy industry. The main purpose of this study was to obtain information on the content of some minor elements in walnut shells, a potentially widely used raw material for energy purposes. It investigated the content of minor elements (Cr, Cd, As, Cu, Ni, Zn, Pb, Hg) in walnut (Juglans regia L.) shells from three locations in Poland. The trees where the nuts were harvested were not subjected to any chemical treatment that could affect the results of the study. In addition, the paper includes information on global walnut production. The study shows that it is possible to use walnut shells as a material for solid biofuels. The values obtained are generally lower than those for broadleaf and coniferous wood.

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1. Introduction

The Industrial Revolution formed the nucleus of dynamic industrial development, which has continued to the present day. Undoubtedly, the rapid growth of industrial activity has led to environmental degradation [1]. Fossil fuels have been a major source of heat and electricity worldwide for many years. The use of coal for energy purposes has many negative effects on the climate, among other things by increasing atmospheric carbon dioxide emissions, which are a significant factor in the increase in global warming [2]. An alternative to fossil fuels is solid biofuels, also known as biomass, which can be carbon-neutral. The material used to produce such fuels may be wood, plant material, the organic fraction of municipal solid waste, algal material, agricultural residues, or wood chips [3]. Biofuels are divided into four generations. The first is produced from edible raw materials such as sugar cane or vegetable oils. The second generation is related to the use of non-food raw materials such as waste or animal fat. The third and fourth generations are based on lipids and biomass found in algae [4], [5].

According to point (c) of Article 194.1 of the Treaty on the Functioning of the European Union, within the framework of the internal market, and taking into account the needs of caring for the environment, the European Union has committed, among other things, to the development of new and renewable energy sources [6]. This goal is fulfilled by Directive (EU) 2018/2001 of the European Parliament and of the Council of December 11, 2018, on the promotion of the use of energy from renewable sources, according to which by 2030 the share of renewable energy sources in the European Union's energy sector is to be 42.5% with a target of an additional 2.5%.

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In addition, the directive sets binding targets for the transport, heating and cooling, and construction sectors [7]. According to the Polish Central Statistical Office, between 2018 and 2022, the share of energy from RES in the acquisition of primary energy in Poland, i.e. the total energy contained in primary energy carriers, increased from 18.6% to 22.4%. Given the geographical conditions and the resources held by the Polish state in 2022, energy from RES was mainly derived from solid biofuels (64.5%), wind energy (12.7%), and liquid biofuels (8.0%) [8].

The shortage of wood for energy purposes in Poland is due to limited forestry resources and an increased demand for that material in industry. A possible alternative to wood is agricultural biomass, which has high potential for use for energy purposes. However, it poses certain challenges due to the technological problems associated with it [9].

Proper combustion of solid biofuels, especially in thermal power plants, requires optimization of the process to minimize the risk of emitting potentially harmful gases into the atmosphere and to avoid increased costs associated with plant maintenance.

During combustion, trace elements are bound in the ash, while less stable compounds are broken down and discharged as vapor or together with fly ash [10]. It is therefore important to use biomass of suitable quality, the properties of which are verified by laboratories.

The aim of this study was to determine the content of minor elements in walnut shells, as one of the parameters determining the suitability of such a raw material for solid biofuel production, and to compare the values obtained with typical values for virgin wood materials, with or without small amounts of bark, leaves and needles.

2. Characteristics of raw material

Walnut (Juglans regia L.), also known as Persian or English walnut, is an oil plant rich in fatty acids, belonging to the family Juglandaceae [11], [12], [13], [14], [15], [16]. It occurs in temperate and subtropical regions of the northern hemisphere [17]. It is recognized as one of the most economically important nut tree species in the world, having been cultivated since ancient times and being sold throughout the world [18], [19], [20]. In global nut production, walnuts take second place, just behind almonds [21]. Juglans regia L. has been in cultivation in Poland for centuries. It may grow as high as 35 meters. It has a spreading crown and a round trunk. The fruit of Juglans regia L. is a drupe. Its pericarp is made up of an exocarp that falls off easily, and a hard endocarp which surrounds the edible seed [22] (Fig. 1). Mature walnut trees are located by means of deep palm root systems and fine-grained trunks, which are responsible for the production of wood used in furniture making. The leaves of Juglans regia L. are pinnate and elongated and consist of 5 to 23 leaflets [23]. The lignocellulosic material contained in walnut shells has a high carbon content, as well as a high number of reactive functional groups. Its rheological properties indicate its potential use as a reinforcing material in polymer composites. Juglans regia L. shells consist of 50.3% lignin, 23.9% cellulose, and 22.4% hemicelluloses, as well as extractives such as tannins, fats or waxes [24], [25].

The last decade has shown a marked increase in interest in the use of plant processing by-products. Due to their high availability, walnut shells used as a biosorbent, or binding agent, are receiving increasing attention in the production of biofuels in the form of briquettes [26], [27], [28].

Fig. 1. Walnut shell and core

3. Global production

The global volume of walnut production from 1990 to 2022 shows a clear increase in interest in the harvest of the studied raw material. In 1990 it was 890,515 tons, while in 2022 it will be 3,874,024.7 tons, which is more than four times the value at the start of the period under study (Fig. 2). The increase in interest in walnuts allows us to hypothesize a need to manage waste from its production. The increase in consumption of this raw material causes the production of more waste, including shells, which are a potential material for use in the production of solid biofuels.

Between 2020 and 2022, the total volume of walnut production in the world amounted to 10,856,098 tons, of which Africa produced 513,252 tons (4.73%), the Americas 3,077,832 tons (28.35%), Asia 6,092,077 tons (56.12%), Europe 1,154,859 tons (10.64)%, and Oceania only 18,078 tons (0.17%).

This means that the most productive region in terms of walnut production in the world is Asia, while Oceania has the smallest output (Fig. 3).

Walnut production in Poland accounts for a low percentage of global production. Between 2020 and 2022, Poland produced 24,500 tons of the material (Figure 4), which is only 0.23% of global output. Nevertheless, the value of walnut production in Poland is higher than in Oceania as a whole, which accounts for 0.17% of worldwide production.

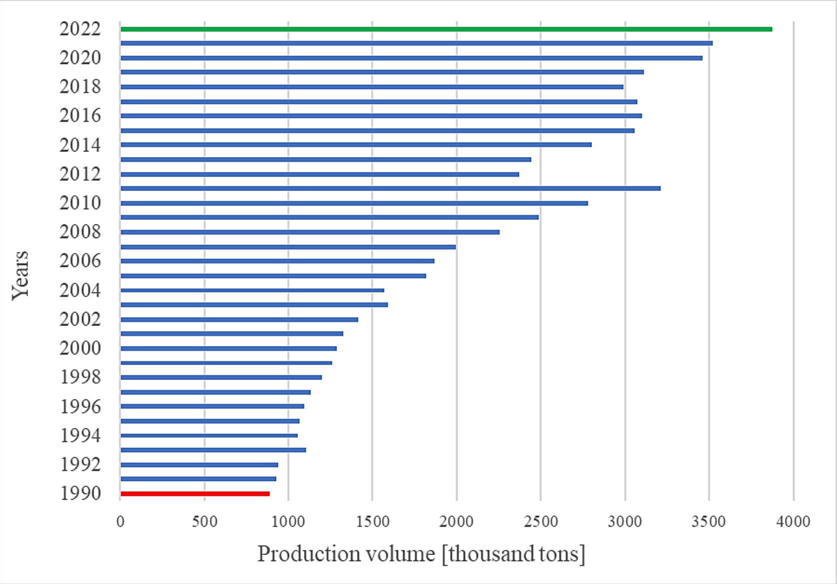


Fig. 2. World walnut production between 1990 and 2022 [29]

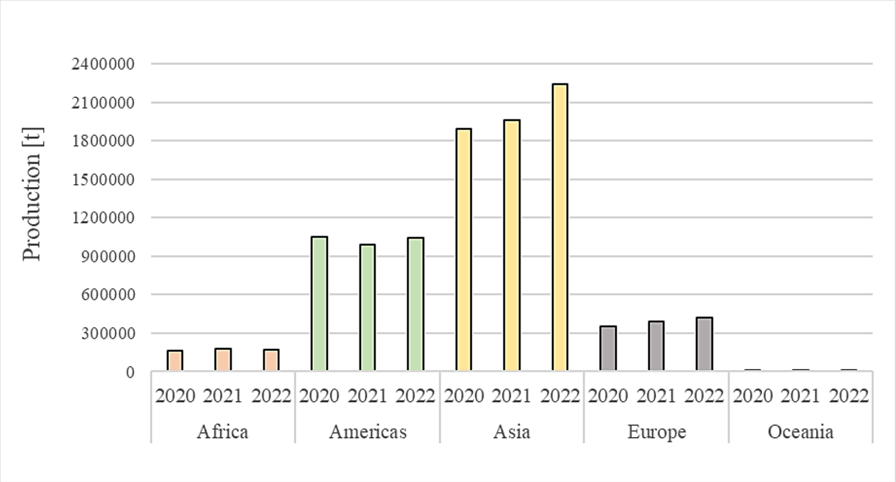


Fig. 3. Walnut production by world regions in 2020–2022 [29]

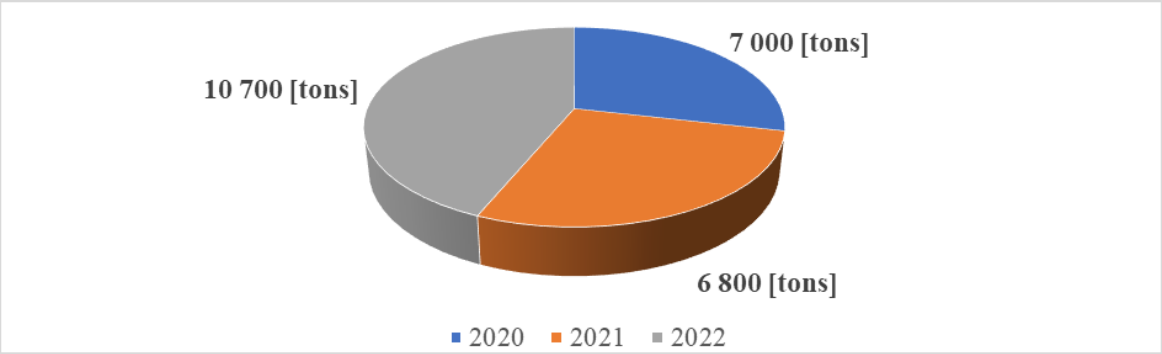


Fig. 4. Walnut production in Poland between 2020 and 2022 [29]

4. Atomic absorption spectrometry

The development of analytical methods reflects the demands placed on analytical chemistry by other fields of technology or science. The determination of elements whose concentration levels are very low requires the selection of an appropriate method that minimizes the risk of factors affecting its correctness [30]. The most commonly used techniques in such studies are flame atomic absorption spectrometry (FAAS), graphite furnace atomic absorption spectrometry (GFAAS), and inductively coupled plasma optical emission spectrometry (ICP-OES) [31]. The measurement principle is based on a phenomenon related to the absorption of radiation of specific wavelength by free atoms of an element. According to the premise of the method, the source of the absorption lines are free atoms that can absorb radiation of such wavelengths that they themselves can emit, while absorption results in an increase in energy [32].

5. Raw material preparation

Walnuts from three locations – Płock, Wolsztyn, and Poznań – were selected for analysis to determine the content of minor elements in their shells. Firstly, each batch of raw material was washed under tap water to remove soil residues (Fig. 5). Each group of nuts was next placed in a laboratory dryer at 60 °C for 60 minutes to dry.

The preparation of samples for analysis consisted first of extracting the cores from the nuts and then pre-crushing the shells with a hammer (Fig. 6). In the next step, the shells were ground with a Fritsh grinder to a fraction of less than 0.75 mm (Fig. 7), which is required for minor elements analysis by atomic absorption spectrometry methods.

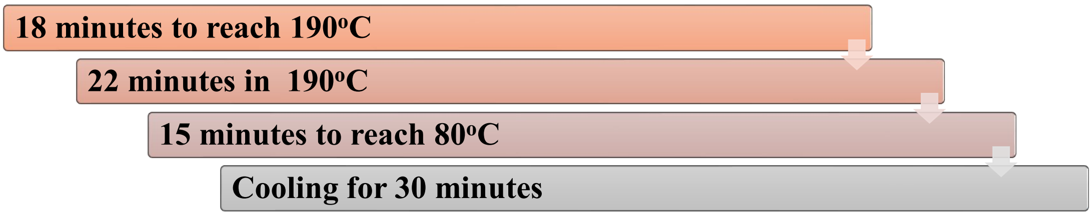
Fig. 5. Walnuts after washing under tap water

Fig. 6. Nut shells after initial crushing

Fig. 7. Walnut shells after grinding

5. Methods

In the first step, dissolution of the raw materials was carried out with a MARS microwave digestion system, using nitric acid. The process parameters are shown in Scheme 1. A DMA-80 mercury analyzer using graphite cuvettes, with a detection limit of 0.0015 ng of mercury, was used to determine mercury in the studied walnut shells. The analytic parameters are shown in Scheme 2.



Scheme 1. Parameters of the mineralization process using a microwave digestion system

Scheme 2. Parameters for mercury (Hg) determination using the DMA-80 mercury analyzer.

5. Analysis of results

The content of minor elements was calculated using the formula:

$$w_i = \frac{(ci - ci{,}0)x\,V}{m} \text{ x } \frac{100}{(100 - Mad)} \quad [\text{mg/kg}] \ (1)$$

where,
w_i is the concentration of the element in the sample, on a dry basis [mg/kg],
c_i is the concentration of the element in the diluted sample digest [mg/l],
$c_{i,0}$ is the concentration of the element in the solution of the blank experiment [mg/l],
V is the volume of the diluted sample digest solution [ml],
m is the mass of the test portion used [g],
M_{ad} is the moisture content in the analysis test sample [% m/m].

Table 1 presents the results of the laboratory tests. Walnuts originating from Wolsztyn had the highest contents of arsenic (As), zinc (Zn), and cadmium (Cd). Nuts of *Juglans regia* L. from the city of Płock had the highest levels of chromium (Cr), copper (Cu), and nickel (Ni). The raw material from Poznań had the lowest content of all minor elements apart from copper (Cu). Cadmium was not detected in any of the walnut shell samples tested. Figure 8 presents calibration curves for all tested minor elements.

Zinc was the element found in the highest amounts in all of the raw materials studied: from Płock (average value 8.038 mg/kg), Wolsztyn (8.155 mg/kg), and Poznań (2.403 mg/kg). Copper was the second most abundant, with average contents of 3.056 mg/kg in Płock, 1.398 mg/kg in Wolsztyn, and 2.3045 mg/kg in Poznań. Cadmium was not detected in the studied material. Mercury also reached very low values, with average results ranging between 0.00061 mg/kg in Poznań and 0.001 mg/kg in Wolsztyn and Płock (Fig. 9).

Table 1. Contents of minor elements in tested walnut shells

Minor element	**Blank sample**	**Płock**						**Wolsztyn**						**Poznań**					
	Minor element content on a dry basis [mg/kg]	Minor element content on a dry basis [mg/kg]			average value [mg/kg]	SD	% RSD	Minor element content on a dry basis [mg/kg]			average value [mg/kg]	SD	% RSD	Minor element content on a dry basis [mg/kg]			average value [mg/kg]	SD	% RSD
Arsenic	0.00000	0.000[a]	0.005	0.009	0.007	0.003	40.41	0.009	0.016[a]	0.010	0.010	0.001	7.44	0.000	0.006	0,000	-		
Chromium	0.00000	1.954	1.735	1.762	1.817	0.119	6.57	0.973	0.994	1.033	1.000	0.030	3.04	0.970	0.958	0.854	0.927	0.064	6.879
Copper	0.00533	3.854[a]	3.186	3.056	3.121	0.092	2.95	1.423	1.388	1.383	1.398	0.022	1.56	2.386	3.107[a]	2.223	2.3045	0.115	5.001
Mercury[b]	-	0.00107	0.00107	0.00096	0.001	0.000	6.02	0.00130	0.00120	0.000978	0.001	0.000	14.32	0.00064	0.00064	0.000534	0.00061	0.000	10.205
Nickel	0.00200	0.382	0.345	0.393	0.373	0.025	6.74	0.257[a]	0.140	0.164	0.152	0.017	11.16	0.171	0.307[a]	0.096	0.134	0.05303	39.725
Lead	0.00000	0.271	0.311[a]	0.244	0.258	0.019	7.41	0.147[a]	0.352	0.481	0.417	0.091	21.90	0.110	0.102	0.167[a]	0.106	0.006	5.337
Zinc	0.06000	10.717[a]	7.504	8.572	8.038	0.755	9.40	11.424[a]	8.157	8.153	8.155	0.003	0.03	2.138	10.686[a]	2.667	2.403	0.374	15.570
Cadmium	0.00024	**Below the limit of detection**			-			**Below the limit of detection**			-			**Below the limit of detection**			-		
Average moisture content of the test sample [%]		**6.67**						**7.98**						**6.34**					

a – rejected values

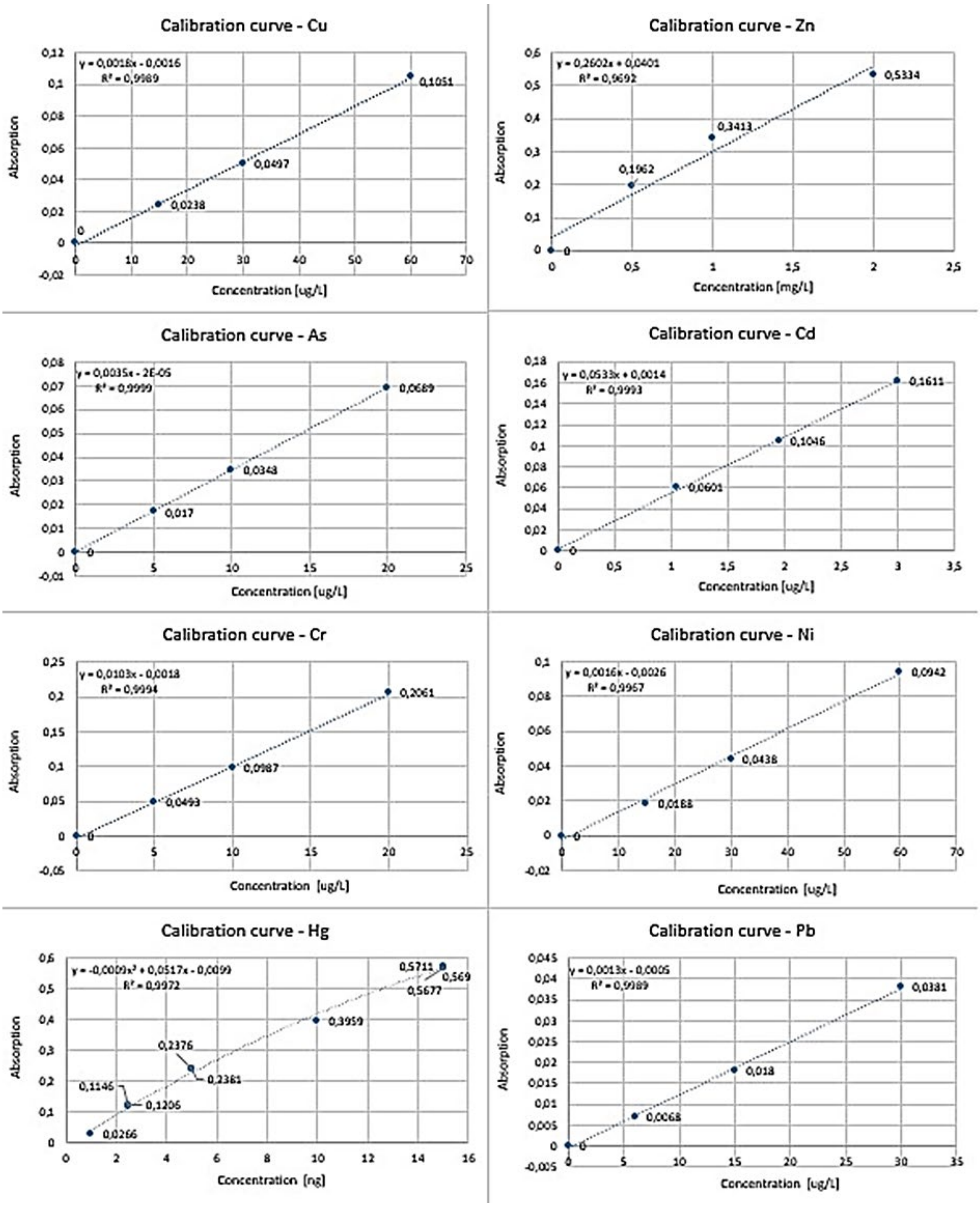


Fig. 8. Calibration curves for minor elements

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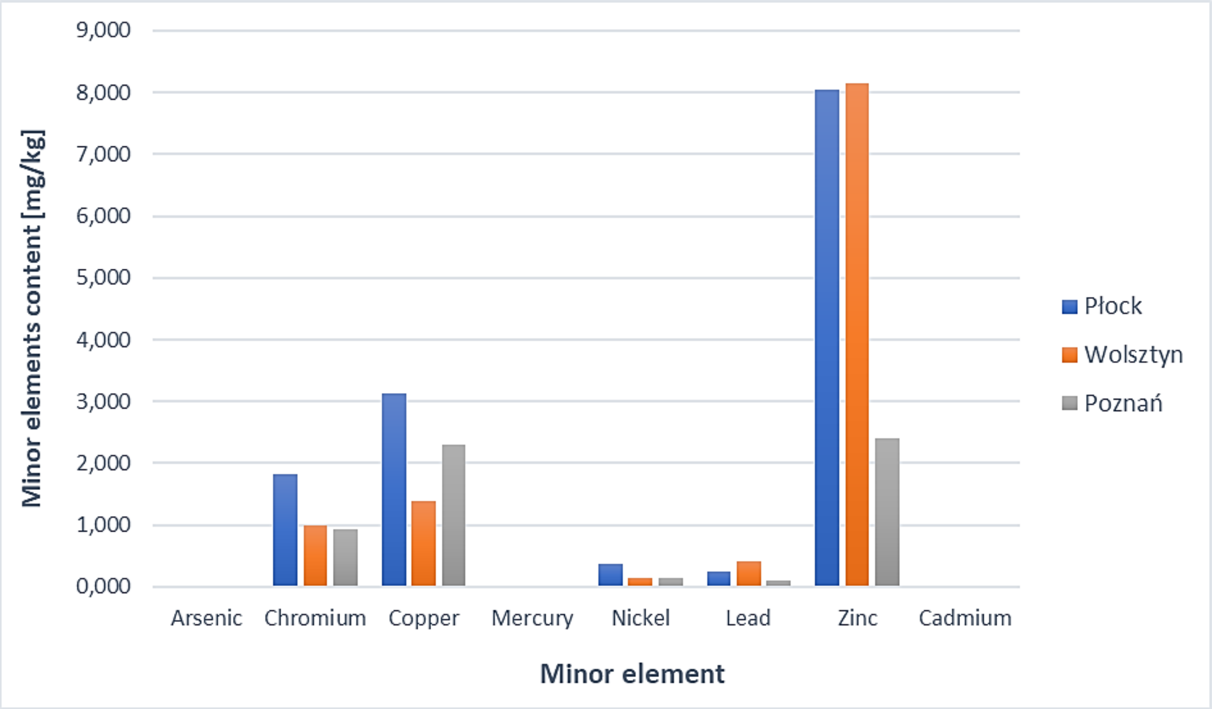

Fig. 9. Contents of minor elements in tested walnut shells

Table 2. Average contents of minor elements in coniferous and broadleaf wood according to PN-EN ISO 17225-1:2021 [36] and values obtained from tests on walnut shells

Minor element	Wood		Walnut shell		
	coniferous	**broadleaf**	**Płock**	**Wolsztyn**	**Poznań**
	Typical elemental concentration value on a dry basis [mg/kg].		Average minor element content on a dry basis [mg/kg]		
Arsenic	<0.1	<0.1	0.007	0.01	-
Chromium	1.0	1.0	1.817	1	0.927
Copper	2.0	2.0	3.121	1.398	2.3045
Mercury	0.02	0.02	0.001	0.001	0.00061
Nickel	0.5	0.5	0.373	0.152	0.134
Lead	2.0	2.0	0.258	0.417	0.106
Zinc	10	10	8.038	8.155	2.403
Cadmium	0.1	0.1	-	-	-

Table 2 shows the contents of individual trace elements in coniferous and deciduous wood as presented in PN-EN ISO 17225-1:2021 [36], together with the values obtained in the present study. As can be seen from the table, almost all trace elements are present in lower quantities in walnut shells than in wood used for solid biofuel production. The exceptions are copper, which has a higher content in the shells from Płock and Poznań than in the wood, and chromium in the shells from Płock, for which the content is almost twice as high as the typical value for the wood of both species. The higher trace element values for a given location may be due to the environment where the raw material was harvested. The nuts from Płock were collected in close proximity to an industrial district. The raw material from Poznań was obtained from a district of the city containing single-family houses. The nuts from

Wolsztyn were collected near a national road with high traffic volume.

6. Summary

The main purpose of this paper was to obtain information on the content of some minor elements in walnut shells, a potentially widely used raw material for energy purposes. The results obtained concern one of several fuel characteristics included in the standards for energy materials. The results presented here indicate that walnut shells are competitive with coniferous wood and hardwood in terms of trace element content. Walnut is undoubtedly a raw material with high potential for use in many economic sectors. While the fruit is used in the food and cosmetics industries, there is also potential for the use of other parts of the plant, such as the shells. Among other uses, walnut shells serve as an abrasive material, an ingredient in cosmetics for exfoliation, and filler in the construction industry. In the present study, contents of minor elements were analyzed for walnut shells from three locations in Poland. The most abundant minor elements in all groups were zinc, copper, and chromium. No presence of cadmium was detected.

The variation in minor element content within the same species may be due to a number of factors. The most likely factor is the location of the trees (an urban fringe, an area in close proximity to a production plant, and a village through which a high-traffic road passes). The study was conducted on a laboratory scale. In order to obtain a complete picture of the energetic uses of walnut shells, further studies should be carried out focusing on ash content, chlorine content, sulfur content and calorific value, among other parameters.

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