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COMPARISON OF THE EFFECTIVENESS OF DIFFERENT TYPES OF EXTRACTANTS FOR RECOVERY OF IRON FROM MINERAL WOOL WASTE

PORÓWNANIE SKUTECZNOŚCI WYKORZYSTANIA RÓŻNEGO TYPU EKSTRAHENTÓW DO ODZYSKU ŻELAZA Z ODPADOWEJ WEŁNY MINERALNEJ

Abstract: The paper presents the results of iron extraction from wool waste. In studies as extractant was used: water, EDTA, acetic acid, formic acid, DTPA, ammonium lactate and calcium lactate. For analysis has been taken wool after one year cultivation of tomato and cucumber and wool after two years of mixed cultivation. The main aim of the analysis was to determine which extractant allows the greatest recovery of the iron from waste mineral wool. Current trends in the agriculture development and the fertilizer industry are aimed at maximizing the recovery of nutrients from waste for re-use. Demonstration of the effectiveness of the recycling of valuable trace elements contained in the mineral wool can make develop a method of wool utilization profitable. In addition, the high cost of fertilizer components makes a new type of medium containing nutrients obtained by extraction from mineral wool waste, can be an interesting option in the future. Implementation of this type of process is consistent with the objectives of Sustainable Development, as well as the environmental policy of the European Union. The proposed way to get iron from mineral waste wool consists of following steps: drying at 30 °C for 24 hours, grinding to particles with a sieve size of 0.40 mm, extraction and phase separation. The studies included the effect of time, temperature and type of extractants on the efficiency of the process. The iron content in the samples was determined by spectrophotometric method based on the ferric ion complex of 2,2-pyridyl in a solution at pH of 3.1.

Keywords: horticultural mineral wool, iron, fertilizers, re-use

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Introduction

Disposal of post-mineral wool is still unsolved problem. Until now, the only way to deal with this waste is storage in landfills.

Mineral wool is obtained from rock. The production process consists of two stages. The first stage involves the grinding of raw materials in ball mills and melted in a furnace. Melting point of such kind of rocks is about 1550 °C. In the second stage, the molten material is fiberized [1–4]. Process for the preparation of mineral wool is continuously refined to reduce its energy consumption and waste production. Mineral wool is important product for the infrastructure and economy [5].

The main market of mineral wool, besides the construction industry, is agriculture. From the economic and trade point of view the most profitable method of cultivation is hydroponics [4, 6]. Hydroponics has several advantages. It allows to overcoming the impact of pathogens and gives a high degree of control over the factors that stimulate growth [7]. In the soilless cultivations inert substrates such as described wool are used. Except wool inert substrates also include such materials as pumice and perlite. Farmers also often used organic substrate as peat, coconut wool or straw [8, 9]. The problems associated with disposal of mineral wool cause the higher consumption of organic substrates. Especially coconut fiber is highly appreciated. This phenomenon is also observed in the construction sector. Today a lot of research is carried out to increase the hydrophobicity requirements of natural materials (flax, hemp) and which are met by mineral wool [10].

Researchers of the disposal problem of mineral wool made a number of proposals for its reuse [11–16]. The main ideas relate to the use of wool waste as a raw material for reclamation and rehabilitation of degraded land [11, 12]. Some research which was carried out with a solution of DTPA demonstrated that it is possible to obtaining some of the microelements from the structure of the mineral wool [13]. In addition, mineral wool after the completion of the crop cycle contains a residue of fertilizer. This gave the opportunity to analyze the possibility of utilization of mineral wool with obtaining nutritional ingredients.

Generally wastes are used in the production fertilizers in small scale. The main raw materials are local waste. The purpose of this type of production is utilization of the wastes which are produced in neighborhood. Wastes which are generally used in the production of fertilizers derive from agri-food production as sugar plants, starch plants, distilleries but also industrial waste as leather waste from tanneries [17].

The idea of using waste in the production of fertilizers is consistent with the principles of Sustainable Development. Modern fertilizers should be produced according with the new trends. The current requirements are not only the economic but also environmental and social [17]. The environmental aspect during launching of new products on the market is very important. Before placing a product on the market, it is important to determine its impact on the environment. For this purpose a Life Cycle Assessment (LCA) is used [18]. According to the LCA analysis liquid fertilizers are safer for the environment. They also have other advantages such as ease of application, efficacy, long duration of action and many others.

The demonstration of the possibility of recovery of essential nutrients contained in the mineral wool waste may allow for obtain cost-effective liquid fertilizer. The main aspect of the profitability of the process is the selection of the extracting agent [19]. This type of fertilizer may be a breakthrough product on the market.

Materials and methods

The aim of the study was to determine the possibility of obtaining liquid fertilizer with extract from mineral wool waste. In the study the following extractants were used: water, EDTA, acetic acid, formic acid, DTPA, ammonium lactate and calcium lactate. For analysis has been taken wool after one year of tomato cultivation, one year of cucumber cultivation and wool after two years of mixed cultivation.

Before appropriately extraction process wool waste has been subjected to drying process. The dried material has a sufficient brittleness to be able to comminute it on a sieve of mesh size 0.40 mm. The milled wool with a mass of 5 g was subjected to extraction process in flasks with a capacity of 250 cm³. The mass ratio of solid to liquid phase was 1 : 10. This step was carried out at different temperature (25, 30, 50, 70 °C) time – 1, 3, 6, 15 hours. Process of extraction was carried out on a shaker ELPAN company equipped with a thermostat type 357 Water Bath Shaker. The phase separation was carried out on a laboratory centrifuge MPW-360.

Samples of raw mineral wool waste, and a liquid phase after the extraction process, were mineralized in a mixture of nitric acid (20 cm³) and sulfuric acid (30 cm³). Samples were heated for 30 min after the start of boiling. After this time the sample were taken from the heater, cooled to room temperature and poured into the 100 cm³ of distilled water.

Analysis of the iron content was carried out according to standard PN-85/C-84092 [20]. The iron content was analyzed by spectrophotometric method based on formation of a colored ferric ion complex of 2,2-pyridylin solution at pH = 3.1. The absorbance was measured in a quartz cuvette with an absorption layer thickness of 1 cm at a wavelength of 520 nm using an spectrophotometer Jasco V-630.

Results and discussion

The Table 1 shows the results of iron content in the liquid phase obtained after its extraction from wool after one year of tomato cultivation and using water as extractant depending on temperature.

The Table 2 shows the results of iron content in the liquid phase obtained after its extraction from wool after one year of cucumber cultivation and using water as extractant depending on temperature.

Table 1

The iron content in the liquid phase obtained from mineral wool after one year cultivation of tomato and using water as extractant depending on process temperature

The temperature of the extraction process	Time [h]	Content [% mas. Fe]
25 °C	1	0.0028
	3	0.0019
	6	0.0029
	15	0.0395
30 °C	1	0.0002
	3	0.0039
	6	0.0031
	15	0.0406
50 °C	1	0.0080
	3	0.0033
	6	0.0013
	15	0.1206
70 °C	1	0.0035
	3	0.0060
	6	0.0047
	15	0.0558

Table 2

The iron content in the liquid phase obtained from mineral wool after one year cultivation of cucumber and using water as extractant depending on process temperature

The temperature of the extraction process	Time [h]	Content [% mas. Fe]
25 °C	1	0.0018
	3	0.0006
	6	0.0008
	15	0.0379
30 °C	1	0.0003
	3	0.0008
	6	0.0005
	15	0.0525
50 °C	1	0.0021
	3	0.0051
	6	0.0027
	15	0.0377
70 °C	1	0.0076
	3	0.0022
	6	0.0010
	15	0.0358

On the basis of the results it can be seen that for both types of wool, the iron content in the extract is comparable. Figures 1 and 2 showed that in both cases of extraction process the range of favorable efficiency can be obtained even at temperature of 25 °C. Increasing the temperature to 70 °C allowed to achieve similar results as in the process carried out at a temperature of 25 °C or led to a slight increase in efficiency of the process. Process at 70 °C requires a considerable amount of energy, so more preferred will be conduct process at lower temperatures. It seems to be advantageous to lowering the temperature of the process even up to 20 °C and use another, more effective extractant to improve iron extraction efficiency like for example chelating agents.

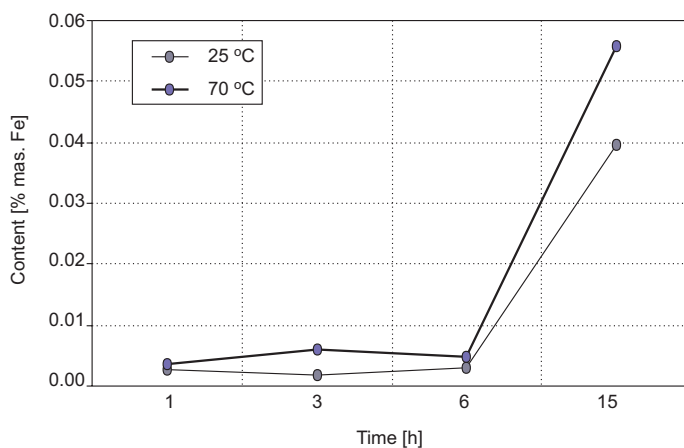


Fig. 1. The dependence of the iron content according to the temperature in the liquid phase obtained from wool after the cultivation of tomato and using water as extractant

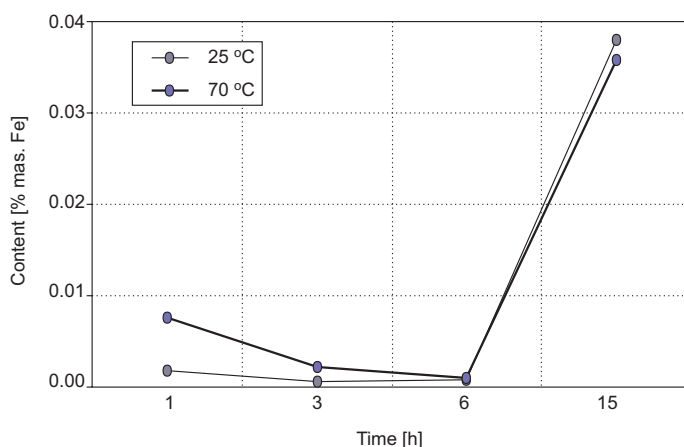


Fig. 2. The dependence of the iron content according to the temperature in the liquid phase obtained from wool after the cultivation of cucumber and using water as extractant

The Table 3 shows the results of iron content in the liquid phase obtained after its extraction from wool after one year of tomato cultivation and using 0.1 M EDTA as extractant depending on process temperature.

Table 3

The iron content in the liquid phase obtained from mineral wool after one year cultivation of tomato and using 0.1 M EDTA as extractant depending on process temperature

The temperature of the extraction process	Time [h]	Content [% mas. Fe]
25 °C	1	0.1634
	3	0.1238
	6	0.1415
	15	0.1292
30 °C	1	0.1418
	3	0.1529
	6	0.1394
	15	0.0870
50 °C	1	0.1591
	3	0.1436
	6	0.1274
	15	0.1118
70 °C	1	0.1341
	3	0.1416
	6	0.1333
	15	0.1071

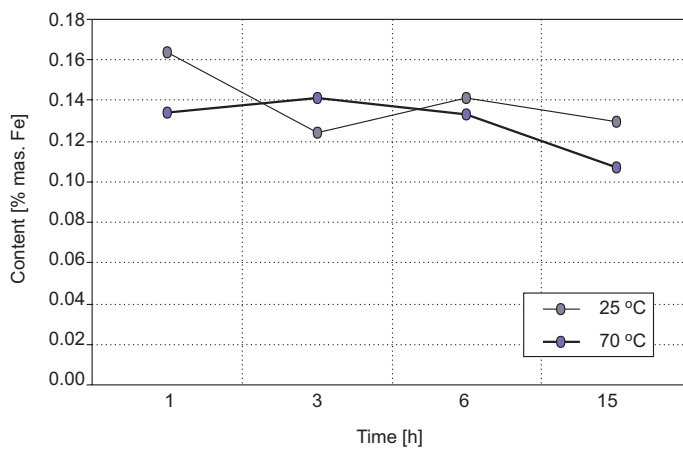


Fig. 3. The dependence of the iron content according to the temperature in the liquid phase obtained from wool after the cultivation of tomato and using 0.1 M EDTA as extractant

The Table 4 shows the results of iron content in the liquid phase obtained after its extraction from wool after one year of cucumber cultivation and using 0.1 M EDTA as extractant depending on process temperature.

Table 4

The iron content in the liquid phase obtained from mineral wool after one year cultivation of cucumber and using 0.1 M EDTA as extractant depending on process temperature

The temperature of the extraction process	Time [h]	Content [% mas. Fe]
25 °C	1	0.0609
	3	0.0614
	6	0.0767
	15	0.0888
30 °C	1	0.0882
	3	0.0793
	6	0.0988
	15	0.1080
50 °C	1	0.0651
	3	0.0946
	6	0.0992
	15	0.0950
70 °C	1	0.0842
	3	0.1075
	6	0.1121
	15	0.1086

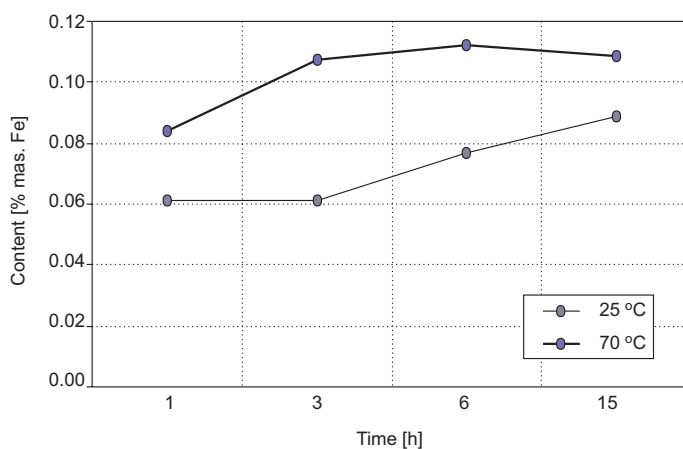


Fig. 4. The dependence of the iron content according to the temperature in the liquid phase obtained from wool after the cultivation of cucumber and using 0.1 M EDTA as extractant

In the presented preliminary studies as a nutrient extractant water was used. The content of iron obtained from the mineral wool waste, under the conditions of the experiments, are relatively low. In the liquid phase obtained by using a solution of EDTA can be notice that iron content is increased. This observation has forced the need to verify a similar increase in the efficiency of using another extractant and taking into account the optimal technological conditions. Figures 5 and 6 show the results of iron content in the liquid phase obtained after its extraction from wool respectively after one year of tomato cultivation and after two years of mixed cultivation and using different extractants which have similar properties as EDTA. These processes were conducted at 25 °C and for 1, 3 and 6 hours

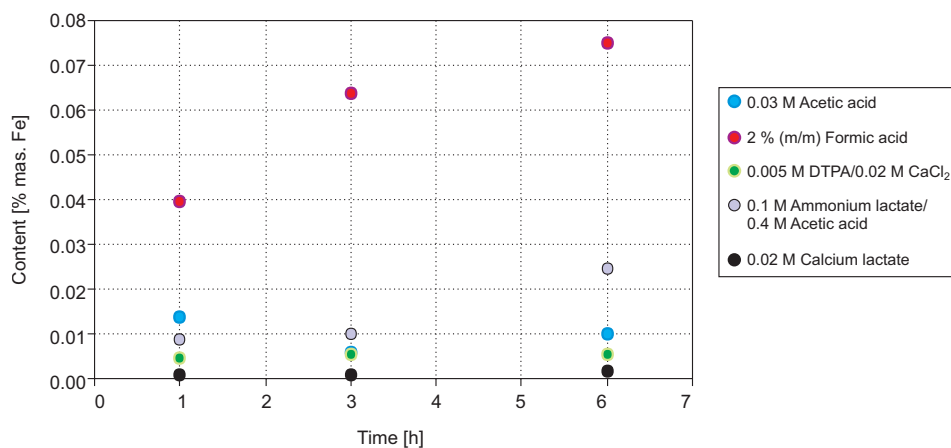


Fig. 5. Comparison of the iron content in the liquid phase obtained from mineral wool after one year cultivation of tomato depending on extractant type

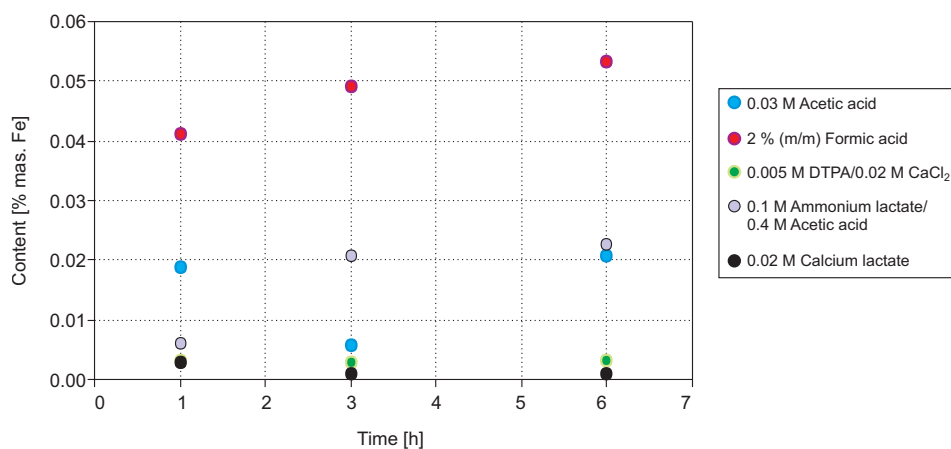


Fig. 6. Comparison of the iron content in the liquid phase obtained from mineral wool after two years of mixed cultivation depending on extractant type

Comparing the obtained results it can be concluded that the least efficient extractant of all included in the study in respect of iron extraction is 0.02 M solution of calcium lactate. Formic acid solution was characterized by the relatively high degree of extraction. However, the content of iron in this case was lower than with using 0.1 M EDTA solution.

Moreover studies have shown that iron extraction from wool after two cycles of cultivation (several processes of fertilization) not always give better results of iron content in extract than in this obtained from wool after one year of cultivation.

Conclusions

The extraction process was a key element of the proposed process of the re-use of mineral wool. Selecting the appropriate parameters of the process is cost-determining factor for the possible implementation of the process. This operation should allow to achieve a relatively high performance extraction of nutrients at a relatively low cost.

The highest recovery of iron from mineral wool waste is possible thanks to use solution of EDTA as extractant. The most important from a technological point of view is that it is possible for low-temperature and short time of extraction. Studies have shown that wool after two cycles of cultivation (several processes of fertilization) does not give better results of iron content in extract than in this obtained from wool after one year of cultivation. Favorably results of iron content were also obtained for process with using acetic acid solution.

The studies make it possible to analyze the practical use of mineral wool waste in the production of fertilizers.

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References

- [1] ROCKWOOL INTERNATIONAL A/S, Hedehusene, DK: Method of making mineral wool, WO2008/138537.
- [2] ROCKWOOL INTERNATIONAL A/S, Hedehusene, DK: Process and apparatus for making mineral fibers, US2011/023233A1.
- [3] ROCKWOOL INTERNATIONAL A/S, Hedehusene, DK: Produkt do stosowania zwłaszcza jako podłoże wzrostowe dla roślin, zastosowanie tego produktu i sposób wytwarzania podłoża, PL208067B1.
- [4] Saint-Gobain Isover Courbevoie: Włna mineralna, jej zastosowanie i sposób jej wytwarzania, PL194126B1.
- [5] Qi GC, Shan FJ, Zhang QK. Mater Sci Forum. 2013;743-744:301-305. DOI: 10.4028/www.scientific.net/MSF.743-744.301.
- [6] Raviv M, Lieth JH. Soilless Culture: Theory and Practice. Elsevier: Amsterdam; 2008.
- [7] Evangelou MWH, Ebel M, Koerner A, Schaeffer A. Chemosphere. 2008;72:525-531. DOI: 10.1016/j.chemosphere.2008.03.063.
- [8] Argo WR, Biernbaum JA. Hort Sci. 1995;30(3):535-538. <http://hortsci.ashspublications.org/content/30/3/535.full.pdf>

- [9] Bussel WT, McKennie S. *New Zeal J Crop Hort.* 2004;32:29-37.
DOI: 10.1080/01140671.2004.9514277.
- [10] Zach J, Hroudová J, Žižková N. *Adv Mater Res.* 2014;897:153-156.
DOI: 10.4028/www.scientific.net/AMR.897.153.
- [11] Baran S, Oleszczuk P, *Roczn Glebozn.* 2006;LVII(1/2):13-20.
http://ssa.ptg.sggw.pl/files/artykuly/2006_57/2006_tom_57_nr_1-2/tom_57_nr_1-2_13-20.pdf.
- [12] Piróg J. *Zesz. Probl. Post. Nauk Roln.* 1998;461:357-364.
- [13] Rupp LA, Dudley LM, *Hort Sci.* 1989;24:258-260.
- [14] Novitskii AG, Efremov MV. *Refract Ind Ceramic.* 2006;47(2):121-124.
DOI: 10.1007/s11148-006-0069-y.
- [15] Jeong BR, Hwang SJ. *Acta Hort.* 2001;554:89-94. http://www.actahort.org/books/554/554_8.htm.
- [16] Diara C, Incrocci L, Pardossi A, Minuto A. *Acta Hort.* 2012;927:793-800.
http://www.actahort.org/books/927/927_98.htm
- [17] Hoffmann K, Hoffmann J. *Am J Agric Biol Sci.* 2007;2(4):254-259.
DOI: 10.3844/ajabssp.2007.254.259.
- [18] Hoffmann J, Skut J, Skiba T, Hoffmann K, Huculak-Mączka M. *Proc ECOpole.* 2011;5(2):537-542.
http://tchie.uni.opole.pl/PECO11_2/PECO_2011_2p2.pdf.
- [19] Huculak-Mączka M, Hoffmann K, Klem E, Hoffmann J. *Przem Chem.* 2014;93(6):1029-1032.
DOI: 10.12916/przemchem.2014.1029.
- [20] PN-85/C-84092 Surowce fosforowe. Metody badań. Oznaczanie składników podstawowych.

PORÓWNANIE SKUTECZNOŚCI WYKORZYSTANIA RÓŻNEGO TYPU EKSTRAHENTÓW DO ODZYSKU ŻELAZA Z ODPADOWEJ WEŁNY MINERALNEJ

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Abstrakt: W pracy zostały przedstawione wyniki ekstrakcji żelaza z odpadowej wełny mineralnej. W badaniach jako ekstrahenty wykorzystano: wodę, roztwór EDTA, kwas octowy, kwas mrówkowy, DTPA, mleczan amonu i mleczan wapnia. Analizie została poddana wełna jednoroczna po uprawie pomidora i ogórka, a także dwuletnia po uprawie mieszanej. Głównym celem analizy było określenie, który ekstrahent umożliwi największy odzysk żelaza z poprodukcyjnej wełny ogrodniczej. Obecne tendencje rozwoju rolnictwa, jak i przemysłu nawozowego są skierowane na maksymalizowanie odzysku składników odżywczych z odpadów, w celu ich powtórnego wykorzystania. Wykazanie skuteczności recyklingu cennych mikroelementów zawartych w wełnie mineralnej może pozwolić na opracowanie opłacalnej metody utylizacji tego odpadu. Ponadto wysoki koszt komponentów nawozowych sprawia, że nowy typ pożywki zawierający składniki pokarmowe pozyskane na drodze ekstrakcji z odpadowej wełny mineralnej może być w przyszłości interesującym rozwiązaniem. Wdrożenie tego typu procesów jest zgodne z założeniami Zrównoważonego Rozwoju, a także polityką ochrony środowiska Unii Europejskiej. Mikroelementowe żelazo pozyskiwane było z odpadowej wełny na drodze: suszenia w temperaturze 30 °C przez 24 h, rozdrabniania na sicie na cząsteczki o wymiarach 0,40 nm, ekstrakcji i rozdziału faz. W prowadzonych badaniach analizie poddano wpływ czasu ekstrakcji, temperatury, a także rodzaju stosowanego ekstrahenta. Zawartość żelaza w próbkach oznaczano metodą fotokolorymetryczną z wytworzeniem kompleksu jonów żelazowych z 2,2-pirydylem w roztworze o pH wynoszącym 3,1.

Słowa kluczowe: ogrodnicza wełna mineralna, żelazo, nawozy, wtórne wykorzystanie