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ИЗВЕСТИЯ

РОО «НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК РЕСПУБЛИКИ КАЗАХСТАН»
ЧФ «Халық»

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В 2016 году для развития и улучшения качества жизни казахстанцев был создан частный Благотворительный фонд «Халык». За годы своей деятельности на реализацию благотворительных проектов в областях образования и науки, социальной защиты, культуры, здравоохранения и спорта, Фонд выделил более 45 миллиардов тенге.

Особое внимание Благотворительный фонд «Халык» уделяет образовательным программам, считая это направление одним из ключевых в своей деятельности. Оказывая поддержку отечественному образованию, Фонд вносит свой посильный вклад в развитие качественного образования в Казахстане. Тем самым способствуя росту числа людей, способных менять жизнь в стране к лучшему – профессионалов в различных сферах, потенциальных лидеров и «великих умов». Одной из значимых инициатив фонда «Халык» в образовательной сфере стал проект Ozgeris powered by Halyk Fund – первый в стране бизнес-инкубатор для учащихся 9-11 классов, который помогает развивать необходимые в современном мире предпринимательские навыки. Так, на содействие малому бизнесу школьников было выделено более 200 грантов. Для поддержки талантливых и мотивированных детей Фонд неоднократно выделял гранты на обучение в Международной школе «Мирас» и в Astana IT University, а также помог казахстанским школьникам принять участие в престижном конкурсе «USTEM Robotics» в США. Авторские работы в рамках проекта «Тәлімгер», которому Фонд оказал поддержку, легли в основу учебной программы, учебников и учебно-методических книг по предмету «Основы предпринимательства и бизнеса», преподаваемого в 10-11 классах казахстанских школ и колледжей.

Помимо помощи школьникам, учащимся колледжей и студентам Фонд считает важным внести свой вклад в повышение квалификации педагогов, совершенствование их знаний и навыков, поскольку именно они являются проводниками знаний будущих поколений казахстанцев. При поддержке Фонда «Халык» в южной столице был организован ежегодный городской конкурс педагогов «Almaty Digital Ustaz.

Важной инициативой стал реализуемый проект по обучению основам финансовой грамотности преподавателей из восьми областей Казахстана, что должно оказать существенное влияние на воспитание финансовой грамотности и предпринимательского мышления у нового поколения граждан страны.

Необходимую помощь Фонд «Халык» оказывает и тем, кто особенно остро в ней нуждается. В рамках социальной защиты населения активно проводится работа по поддержке детей, оставшихся без родителей, детей и взрослых из социально уязвимых слоев населения, людей с ограниченными возможностями, а также обеспечению нуждающихся социальным жильем, строительству социально важных объектов, таких как детские сады, детские площадки и физкультурно-оздоровительные комплексы.

В копилку добрых дел Фонда «Халык» можно добавить оказание помощи детскому спорту, куда относится поддержка в развитии детского футбола и карате в нашей стране. Жизненно важную помощь Благотворительный фонд «Халык» оказал нашим соотечественникам во время недавней пандемии COVID-19. Тогда, в разгар тяжелой борьбы с коронавирусной инфекцией Фонд выделил свыше 11 миллиардов тенге на приобретение необходимого медицинского оборудования и дорогостоящих медицинских препаратов, автомобилей скорой медицинской помощи и средств защиты, адресную материальную помощь социально уязвимым слоям населения и денежные выплаты медицинским работникам.

В 2023 году наряду с другими проектами, нацеленными на повышение благосостояния казахстанских граждан Фонд решил уделить особое внимание науке, поскольку она является частью общественной культуры, а уровень ее развития определяет уровень развития государства.

Поддержка Фондом выпуска журналов Национальной Академии наук Республики Казахстан, которые входят в международные фонды Scopus и Wos и в которых публикуются статьи отечественных ученых, докторантов и магистрантов, а также научных сотрудников высших учебных заведений и научно-исследовательских институтов нашей страны является не менее значимым вкладом Фонда в развитие казахстанского общества.

С уважением,
Благотворительный Фонд «Халык»!

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REMOVAL OF NICKEL IONS FROM INDUSTRIAL WASTEWATER USING ADSORBENTS OBTAINED FROM THE SHELLS OF PUMPKIN SEEDS

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Abstract. The presence of heavy metals in the environment, as a result of human activities, has resulted in water sources contaminated with these elements. This problem has attracted the attention of researchers to find solutions to treat contaminated water and wastewater, such as adsorption. Pumpkin is one of the most common foods in the world. Pumpkin is consumed without seeds which are discarded as unwanted product. In this work, pumpkin seed shells were used as a carbon precursor to produce pyrogal and then used as an adsorbent to treat wastewater containing nickel (Ni) ions. The first sample of pumpkin seed shells was obtained by washing pumpkin seed shells with distilled water. The second sample was obtained by carbonisation in a GSL-1400X tube furnace in a nitrogen atmosphere at 400 °C for 9 h. Derivatograms and thermogravimetric readings of the adsorbents were obtained on a derivatograph of MOM company from Budapest, as well as the results of structural analysis systems SEM and HKL-Basic were obtained.

According to the experimental results, the optimum uptake of nickel ions occurs at pH 6.8, after 360 min, at a concentration of 50mg/L, by the adsorbent AC (activated carbon) from PSS (pumpkin seeds shells), which is associated with an increase in the available surface area, which in turn increases the availability of interchangeable sites on carbon for Ni(II) adsorption. The results were obtained using Agilent 4200 AES.

Keywords: Activated carbons, thermal analysis, heavy metals, adsorption, nickel, AES, SEM

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АСҚАБАҚ ТҰҚЫМЫНЫҢ ҚАБЫҒЫНАН АЛЫНҒАН АДСОРБЕНТТЕРДІ ҚОЛДАНА ОТЫРЫП, ӨНДІРІСТІК АҒЫНДЫ СУЛАРДАН НИКЕЛЬ ИОНДАРЫН ЖОЮ

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Аннотация. Адам әрекеті нәтижесінде қоршаған ортада ауыр металдардың болуы су көздерінің осы элементтермен ластануына әкелді. Бұл мәселе зерттеушілердің назарын ластанған су мен адсорбция сияқты Ағынды суларды тазарту шешімдерін іздеуге аударды. Асқабақ-әлемдегі ең көп таралған тағамдардың бірі. Асқабақ қажетсіз өнім ретінде тасталатын тұқымсыз жейді. Бұл жұмыста асқабақ тұқымының қабығы пирогты алу үшін көміртегі прекурсоры ретінде пайдаланылды, содан кейін құрамында никель иондары (Ni) бар ағынды суларды тазарту үшін адсорбент ретінде қолданылды. Асқабақ қабығының алғашқы үлгісі асқабақ қабығын тазартылған сумен шаю арқылы алынды. Екінші үлгі GSL-1400X құбырлы пеште 400 °C температурада 9 сағат

ішінде азот атмосферасында көміртектену арқылы алынды. Адсорбенттердің дериватограммалары мен термогравиметриялық көрсеткіштері Будапешттегі MOM компаниясының дериватографынан алынды, сонымен қатар SEM және HKL-Basic жүйелерін құрылымдық талдау нәтижелері алынды. Эксперимент нәтижелеріне сәйкес, никель иондарының оңтайлы сіңуі PH 6,8-де, 360 минуттан кейін, 50 мг/л концентрацияда, асқабақ тұқымының қабығынан алынған белсендірілген көмір адсорбентімен жүреді, бұл қол жетімді бетінің ұлғаюымен байланысты, бұл өз кезегінде Ni(II) адсорбциясы үшін көмірде ауыстырылатын учаскелердің болуын арттырады. Нәтижелер Agilent 4200 АЭС көмегімен алынды.

Түйінді сөздер: белсендірілген көмір, термиялық талдау, ауыр металдар, адсорбция, никель

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УДАЛЕНИЕ ИОНОВ НИКЕЛЯ ИЗ ПРОМЫШЛЕННЫХ СТОЧНЫХ ВОД С ИСПОЛЬЗОВАНИЕМ АДСОРБЕНТОВ, ПОЛУЧЕННЫХ ИЗ СКОРЛУПЫ СЕМЯН ТЫКВЫ

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Аннотация. Присутствие тяжелых металлов в окружающей среде в результате деятельности человека привело к тому, что источники воды загрязнены этими элементами. Эта проблема привлекла внимание исследователей к поиску решений для очистки загрязненной воды и сточных вод, таких как адсорбция. Тыква – один из самых распространенных продуктов питания в мире. Тыква употребляется в пищу без семян, которые выбрасываются как отходы. В данной работе скорлупа тыквенных семечек использовалась в качестве прекурсора углерода для получения пирогала, а затем применялась в качестве адсорбента для очистки сточных вод, содержащих ионы никеля (Ni). Первый образец тыквенной скорлупы был получен путем промывания тыквенной скорлупы дистиллированной водой. Второй образец был получен путем карбонизации в трубчатой печи GSL-1400X в атмосфере азота при температуре 400 °C в течение 9 ч. Дериватограммы и термогравиметрические показания адсорбентов были получены на дериватографе компании МОМ из Будапешта, а также были получены результаты структурного анализа систем SEM и HKL-Basic. Согласно результатам эксперимента, оптимальное поглощение ионов никеля происходит при pH 6,8, через 360 мин, при концентрации 50 мг/л, адсорбентом активированного угля из оболочки семян тыквы, что связано с увеличением доступной площади поверхности, которая в свою очередь увеличивает наличие взаимозаменяемых участков на угле для адсорбции Ni(II). Результаты были получены с использованием Agilent 4200 АЭС.

Ключевые слова: активированные угли, термический анализ, тяжелые металлы, адсорбция, никель

Introduction

Heavy metals are a group of chemical elements with metal properties and a density equal to or greater than the density of iron (8 g/cm^3). Typical heavy metals include chromium, iron, vanadium, cobalt, copper, zinc, nickel, cadmium, tin, and molybdenum, among others. The primary sources of metal-containing wastewater are industries with chemical and electrochemical treatment of metals (including electroplating), mechanical engineering, non-ferrous and ferrous metallurgy, instrumentation, automotive, metalworking, machine tools, aviation, electronic, leather, and mining industries, among others. On the one hand, small doses of metals are vital to the body because they participate in various forms of metabolism, such as the transfer and synthesis of substances. On the other hand, excessive amounts of metals have a harmful effect on the human body. They can accumulate in tissues and organs, causing several diseases and mutations, including inherited ones. Insufficiently treated wastewater entering reservoirs accumulates heavy metals in sediments and water becoming a secondary pollution source and negatively affecting the health of people, animals, and plants. This has become one of the most severe environmental problems at present. Removing heavy metals from the environment is of particular concern because of their persistence. Currently, several methods are known for removing heavy metals from wastewater, such as chemical and electrochemical deposition, membrane separation, ion exchange, and adsorption (Jose L. Diaz De Tuesta et.al., 2022).

The basis of wastewater treatment technology for heavy metals removal is usually the conversion of metals from soluble to insoluble forms, followed by water separation from metal-containing suspensions. Most often, the transfer of metals from soluble to insoluble forms is carried out by chemical methods, less often by electrochemical deposition. The suspensions are separated from water by settling using coagulant and flocculant to accelerate the separation process. Filtration through sorbents, ion exchange resins, or reverse osmotic membranes is also used. These methods make it possible to reduce the residual concentrations of heavy metals to very low values but to supply water to the resin or membrane; it must first be cleaned of those types of contaminants to which these methods are very sensitive. The choice of wastewater treatment technology is not an easy process because the composition of wastewater discharge and water quality requirements at each enterprise is different, and these methods are often financially and technically expensive. Adsorption using carbon materials, i.e., adsorption using activated carbons (AC), seems to be an alternative for the removal of heavy metals from wastewater, being characterized as an effective and economically feasible method. Carbon is classified as the fourth most abundant element in the universe (Olivares-Marin et.al., 2012). Due to its excellent characteristics, carbon is used in various fields, such as mining and chemical industries, for water and gas purification (Okoye et.al., 2010).

Adsorption is a spontaneous process of increasing the concentration of a dissolved substance at the interface of two phases (solid—liquid). The adsorption method is usually used for deep purification of wastewater from dissolved organic substances if the concentration of these substances in the water is low, does not biodegrade, or is highly toxic. The adsorbents method is also used for wastewater treatment from heavy metals, where activated carbon is usually used as adsorbents (Sobgayda, Makarova, 2011). The advantage of the method is its high efficiency and the possibility of wastewater treatment containing several substances.

The most effective adsorbents are activated carbons of various brands. The significant disadvantages of activated carbon are its high cost and the need for periodic replacement of the load in the filter since activated carbon regeneration is carried out at high temperatures in special furnaces. The porosity of the coals is 60–75 %, and the specific surface area is 400–900 m^2/g.

There is an increasing need for cheaper sorbents with improved physical, chemical, and operational characteristics. Sorbents used in industry are developed based on activated carbons. Sorbents made from recycled materials are also of interest. Such materials can solve, in addition to the problem of water purification, another task, namely, waste disposal (Nikiforova, 2010).

Creating sufficiently effective and environmentally safe sorbents and technologies is feasible due to forming new functional groups on polysaccharide polymers that firmly bind heavy metal ions. Similarly, so-called polymer sorbents are obtained. It is worth noting that an important direction in creating biopolymer sorbents is the modification of the structure of natural cellulose-containing raw materials, which causes the immobilization of new sorption-active centers on the cellulose matrix, which are fragments of complexions. Increasing their selectivity and sorption capacity and reducing sorption time (Nikiforova, 2010).

A complete set of various materials of plant origin represents the raw materials used for the sorption of heavy metals. To produce carbon, organic waste such as rice husks and rice straws (Cazetta, et.al., 2011), orange peels (Rosas et.al., 2010), pumpkin seed shells (Ilknur Demiral et.al., 2016), date stones (Ahmed et.al., 2012), walnut (Hayashi et.al., 2002), apricot kernel shells (entorun-Shalaby et.al., 2006), palm shell (Lim et.al., 2010), coconut shell (Cazetta et.al., 2011), tea leaves (Gurten et.al., 2012) can be used as promising raw materials. The use of waste solves fundamental environmental problems since it turns waste into valuable products (Timur et.al., 2010). At the same time, some cellulose-containing sorbents have a relatively low capacity concerning TM, while others in their properties may be comparable or even superior to ion exchange resins used in industry.

Among heavy metals, Nickel (Ni) is one of the most commonly used in industrial processes, such as the production of alloys, stainless steel, and car batteries, as well as in electroplating, which leads to high concentrations in the wastewater of these industries (Jiamin et.al., 2019).

The novelty of our work lies in the utilization of pumpkin seed husks as a natural adsorbent for purifying nickel ions from wastewater. By developing activated carbon from these husks through a unique carbonation process, we were able to achieve higher adsorption efficiency compared to traditional methods. Our study also revealed interesting findings regarding the effects of pH, contact time on the overall adsorption process. This innovative approach showcases the potential of utilizing agricultural waste materials for sustainable and effective water purification methods. Our work aimed to obtain husks from the shells of pumpkin seeds and an adsorbent from them and use them to purify nickel ions from wastewater. Activated carbons were prepared by carbonation in nitrogen at 400 °C for 9 hours.

It has been established that the adsorption efficiency of the natural shell of pumpkin seeds and carbon obtained by carbonation in nitrogen during the purification of water containing nickel ions is higher than the husk of pumpkin seeds. It was also found that cleaning nickel ions at a concentration of 50 mg/l with activated carbon showed a high removal percentage but decreased as the concentration of nickel ions increased. Periodic adsorption experiments were carried out to study the effect of pH, contact time on the adsorption of nickel ions from an aqueous solution by two samples of adsorbent - the husk of pumpkin shells and activated carbon from it.

Materials and methods

Preparation and preparation of adsorbents

Shell of the pumpkin seeds (PSS) used for adsorption was grown in the Otyrar district of the Turkestan region of Kazakhstan. The shells of pumpkin seeds are easily converted into carbon since they contain a small number of inorganic components. As a raw material for two samples, the shell of pumpkin seeds was taken to prepare adsorbing materials. The collected shell of pumpkin seeds was washed several times, first with boiled water, then with deionized water to remove the stuck dirt. Drying was carried out in a drying cabinet at 60 °C for 24 hours to a constant weight. The dried sample was crushed, sieved through a sieve to a particle size of 0.3–1 mm, and stored in a plastic bottle for further use. The sample was obtained by processing the shell of pumpkin seeds with distilled water. Also, the shells of pumpkin seeds were obtained by carbonation in a GSL-1400 X tubular furnace in a nitrogen atmosphere at 400 °C for 9 hours.

The mass of 5 g of crushed dried pumpkin seed shell was weighed on analytical scales and placed in a horizontal tubular furnace with quartz coating for pyrolysis. Carbonation occurred at a continuous flow rate of N_2 100 nm^3/min. The furnace was programmed to heat at a rate of 10 °C min-1 and maintain isothermal phases for 60 minutes at 120 °C, for 90 minutes at 240 °C and 240 minutes at 400 °C, then an isothermal phase lasting 140 minutes at 400 °C was carried out, as a result of carbonization, a activated carbon (from pumpkin seed shell) was obtained.

Characteristics of materials

Scanning electron microscope (SEM) JSM-6490LV with INCA Energy microanalysis and HKL-Basic structural analysis systems. FT-IR analysis was used to determine the physico-chemical characteristics of pumpkin seed shells. IR spectra of pumpkin seed shells were obtained on an IR Fourier device (Infraspec, model FSM 2202, Russia, St. Petersburg) with a resolution of 1 cm-1 and a scanning range of 5000–500 cm-1 using a sample. The multi-purpose scanning electron microscope (useful magnification of 300,000) combines the possibilities of working in both standard and low-vacuum modes. It allows you to examine samples without spraying with a conductive layer.

Additionally, it is equipped with an INCA Energy 350 energy dispersive microanalysis system and a prefix for studying the texture and structure of polycrystalline HKL Basic samples. Thermogravimetric analysis of derivatives was carried out on a derivatograph of the company "MOM" - Budapest (Hungary). The method used is based on the recording by the device of changes in the thermochemical and physical parameters of a substance that can be caused when it is heated. The thermochemical state of the sample is described by the curves: T (temperature), DTA (differential thermoanalytical), TG (thermogravimetric), and DTG (differential thermogravimetric), the latter curve is a derivative of the function TG. DTA- DTG- TG-. The concentration of Ni (II) was determined by the NPP method. All measurements were carried out using Agilent 4200 MP-AES equipped with an Agilent 4107 nitrogen generator.

Reagents and materials

The sample entry system consisted of a double-pass cyclone spray chamber, a OneNeb sprayer, a Solvaflex pump tube (orange/green), and an Easy-fit burner for sample introduction. Multi-element calibration standards containing Ni(II) with a concentration of 50 mg/l were used. The standard was prepared in an environment of 5 % HNO_3/0.2% HF (vol./vol.) (US production). The pH was determined using a pH meter of the brand pH-009(1)A.

Preparation of the model solution, the effect of contact time and pH values

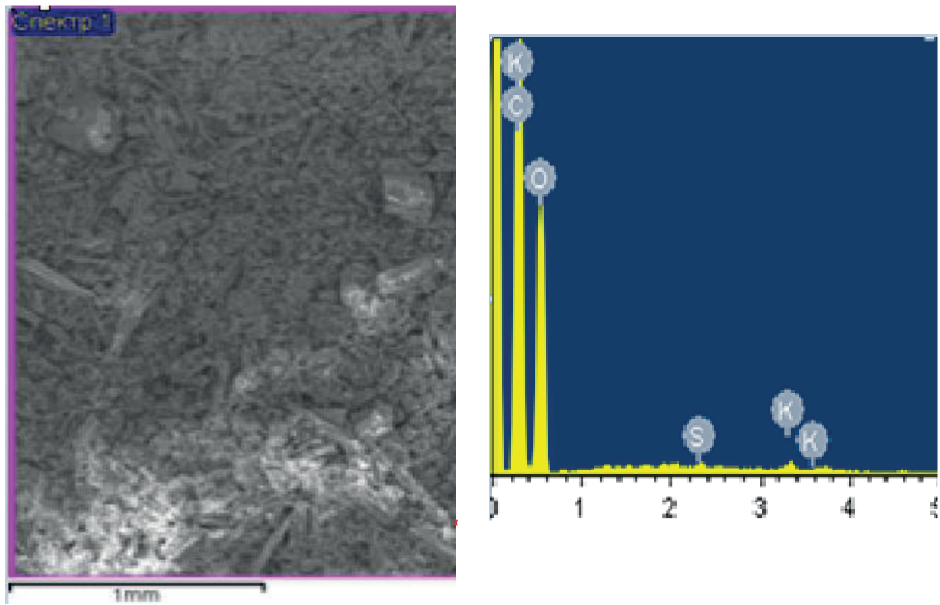
By dissolving a sample of nickel sulfate crystallohydrate (NiSO4•6H2O) 0.2390g, 1 liter of the solution was prepared, with a concentration of 50 mg/L. The pH of the solutions was adjusted using 0.01 M HCl solutions and 0.01 M NaOH solutions.

In six erlenmeyer flasks with a volume of 250 ml, 100 ml of a solution containing 50 mg / l of Ni (II) solution was taken, 0.25 g of pumpkin husks and activated carbon were added to each, and the pH was adjusted to 3, 6.8, 9 using 0.01 M solutions of HCI and NaOH, the mixture was shaken on a laboratory shaker at various intervals of 15, 30, 60, 120, 240 and 360 minutes. The NPP method determined the residual concentration of Ni (II) in each sample's filtrate.

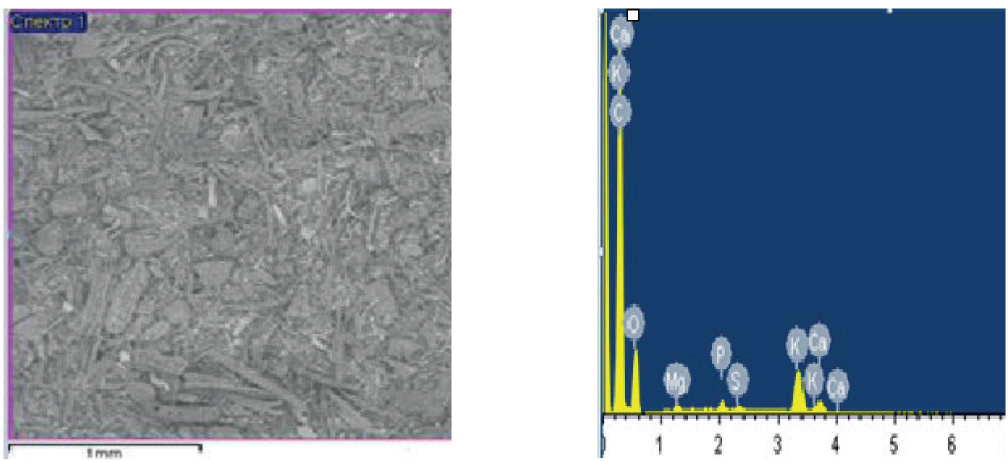
Results and Discussion

Elemental Composition

The results of the elemental composition of the pumpkin seed shell and the activated carbon of the AC rom PSS at 400 °C were obtained using a scanning electron microscope JSM-6490LV with INCA Energy Energy microanalysis and HKL-Basic structural analysis systems are presented in Figure 1.



a)



b)

Figure 1 Results of SEM and HKL-Basic structural analysis systems
(a)pumpkin seed shell (b) activated carbon from PSS

Table 1 shows the content of elements in the shell of pumpkin seeds and the carbon shell of pumpkin seeds calcined at 400 ^{0}C. According to the results of elemental analysis, the shell of pumpkin seeds contains carbon (60.97 %), oxygen (38.61 %), there are elements such as potassium (0.25 %) and sulfu r (0.17 %), and in carbon, the carbon content has increased (72.52 %), oxygen (22.76 %) and potassium (2.98 %) decreased. However, elements such as magnesium (0.31 %), phosphorus (0.42 %), and calcium (0.79 %) appeared.

Table 1

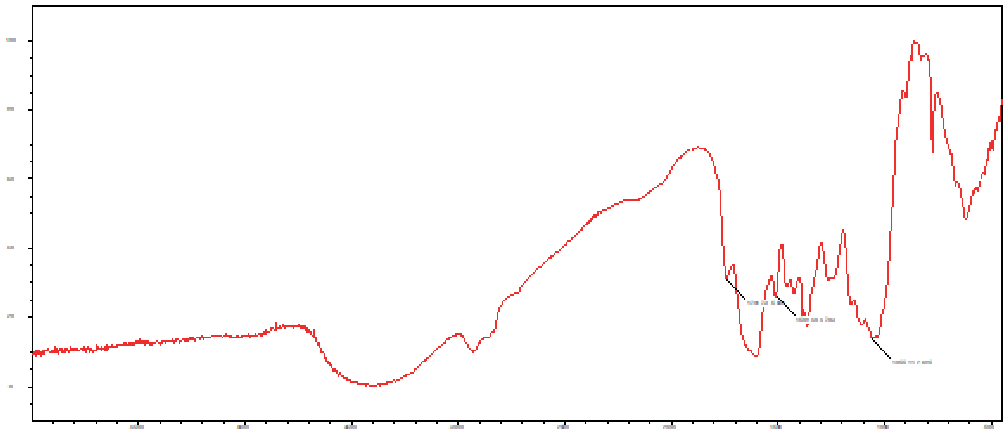
Elemental composition

Materials	Weight of the element (%)						
	C	O	S	K	Mg	Ca	P
Pumpkin Seed Shell	60,97	38,61,	0,17	0,25	-	-	-
Carbon shell of pumpkin seeds calcined at 400 ^{0}C	72,52	22,76	0,22	2,98	0,31	0,79	0,42

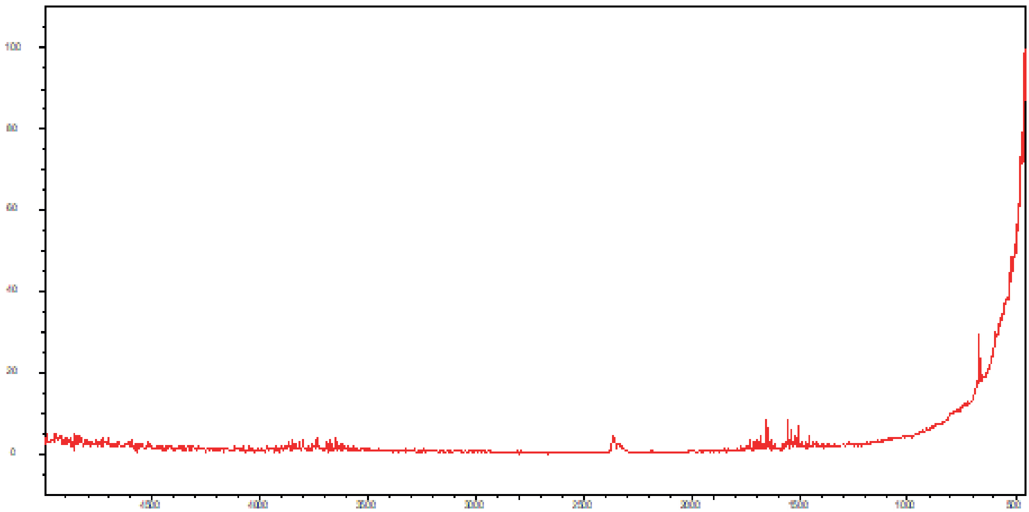
According to the results of elemental analysis, the shell of pumpkin seeds contains, in addition to carbon (60.97 %), oxygen (38.61 %), there are elements such as potassium (0.25 %) and sulfur (0.17 %), and in carbon, the carbon content has increased (72.52 %), oxygen (22.76 %) and potassium (2.98 %) decreased. However, elements such as mag nesium (0.31 %), phosphorus (0.42 %) and calcium (0.79 %) appeared. This is due to the release of volatile substances during carbonation, which led to the removal of non-carbon parts and carbon enrichment. It can be said that chemical activation accelerated the removal of part O, which, as expected, led to an increased content of C.

FT- IR spectroscopic analysis

Fourier transform infrared (FT-IR) analysis was applied to pumpkin seed shells to determine surface functional groups using an FTIR spectroscope, where spectra were recorded from 4000 to 400 cm^{-1}.



a)



b)

Figure 2 FT-IR spectroscopy (a) pumpkin seed shell, (b) activated carbon from PSS

The FTIR spectrum of the pumpkin seed shell (Figure a) shows distinct peaks at 1510,3–1462,1 cm^{-1} (stretching C=O); The strong bands located around 1361,8-1261,5 cm^{-1} can be attributed to the stretching vibration of hydrogen-bonded P=O groups from phosphates or polyphosphates, the O–C stretching vibration in the P–O–C (aromatic) linkage, and P=OOH (Puziy et.al., 2005). Stretch marks in cyclic ethers attached to double bonds or asymmetric, the area of 1261,5-1159,3cm^{-1} can be attributed to the mode of oscillations of O—O ester, ether, phenolic or carboxyl groups, but in this area, it is not easy to attribute peaks due to the overlap of absorption bands from many oxygen-containing substances. Functional groups are fundamental characteristics of activated carbon, as they determine coal's surface properties and quality. The results of the IR-Fourier analysis of the shell of pumpkin seeds and activated carbon (IR = 3:1, 400 °C) are shown in the figure. 2(b). the absorption bands in the region 464.86–455.22cm^{-1} indicate the presence of alkynes and alkyl halides (Mahapatra et.al., 2012).

***Thermal analysis (DTA and TGA*)**

The analyses were performed on a derivatograph of the company "MOM" - Budapest (Hungary). The method used is based on the recording by the device of changes in the thermochemical and physical parameters of a substance that can be caused when it is heated. The thermochemical state of the sample is described by the curves: T (temperature), DTA (differential thermoanalytical), TG (thermogravimetric), and DTG (differential thermogravimetric); the latter curve is a derivative of the TG function. DTA- DTG- TG analysis was carried out in an air environment, in the temperature range from 20 to 1000 °C.

The heating mode of the furnace is linear (dT/dt = 10), and the reference substance is calcined Al2O3. For clarity, the shooting conditions of the sample were strictly 50 mg, with the sensitivity of the scales – 50 mg. The analysis was taken within the following limits of the measuring systems of the device: DTA = 250 μV, DTG = 500 μV, TG= 500 μV, T = 500 μV.

The sample and the reference substance for analysis were placed in ceramic crucibles. As a result of the dynamic heating of these samples, the curves DTA, DTG, and TP noted the manifestations caused by the occurrence of various types of reactions in the system. Among them are processes associated with the release into the atmosphere of H2O, hydroxyls during the decomposition of carbo, and reactions n materials, with CO_2 emissions due to the combustion of organic matter (biomass). When the shell of pumpkin seeds is dynamically heated, the DTA, TG, and DTG curves show the manifestation caused by dehydration of the biological mass of the sample and then its combustion. These processes took place in the range of 20–530 °C. The exit from the molecular water system ensured the formation of an endothermic peak on the DTA curve at 90 °C.

The thermogravimetric (TG) line in the range of 40–125 °C formed the stage of water removal from the system, corresponding to a weight loss of 9.5 %. The differential thermogravimetric (DTG) curve in this temperature range described a peak, the trajectory of which, at each moment, indicated a change in the emission rates from the H2O sample. Further heating of the sample when the temperature rises from 125 to 310 °C leads to the complete combustion of the test sample. This process leaves a powerful exothermic protrusion on the DTA curve with two vertices at 365 and 440 °C.

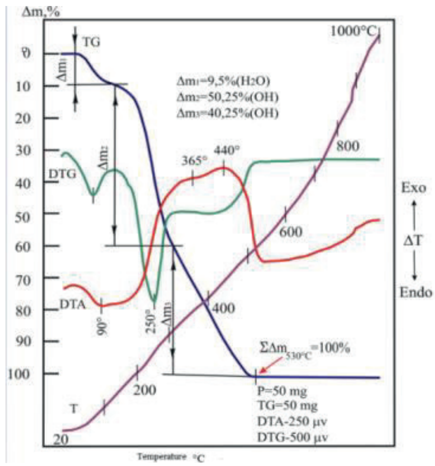


Figure 3 Derivatogram of the shell of pumpkin seeds

Table 2

Thermogravimetric readings of pumpkin seed shells in the range of 20–1000 °C

Weight Loss Sequence	Weight Loss,%	Volatile components of the heated sample	Stages of decomposition, °C
Δm_1	9,5	H_2O	20–125
Δm_2	50,25	H_2O+OH	125–310
Δm_3	40,25	CO_2	310–530
$\sum\Delta m_{1000°C}$	100	H_2O, OH, CO_2	20–1000

The formation of two smoothed peaks on the curve in the marked temperature regions indicates two stages of oxidation of organic matter(s). This is evidenced by the formation of two peaks on the DTG curve at 125–310 and 310–530 °C, respectively. The formation of such peaks on this line results from emissions into the atmosphere within the specified temperature limits of two portions of carbon monoxide equal to weight loss – Δm_2 and Δm_3, Figure 4, Table 3. The carbon of pumpkin shells calcined at 400 °C behaves similarly to the shells of pumpkin seeds when fired. He also showed the effects associated with the powder sample's dehydration and its oxidation in the air. The intensities of these processes occurring in the compared samples are approximately the same.

Indeed, against the background of one general failure of a section of the line of the specified curve in the interval 180–660 °C, the development of any adjacent (in temperature) reactions is not explicitly traced. The process of preparation causes such kinetics of thermal destruction of S carbonates.

As for the presence of adsorbed water (Δm_1) in the test sample in an amount of 11.75 %, this is atmospheric water, which was introduced after carbonation of the initial sample (i.e., after preliminary heat treatment of the initial substance, carbon rehydration occurred).

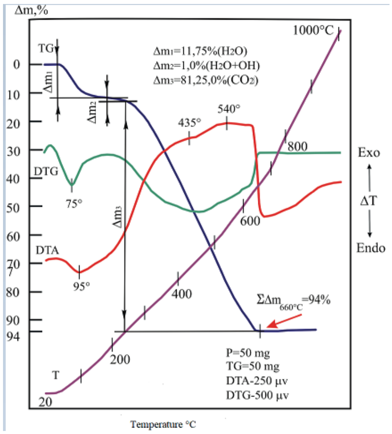


Figure 4 Derivatogram of the carbon shell of pumpkin seeds calcined at 400 °C

Table 3

Thermogravimetric indications calcined pumpkin shell carbon at 400 °C in the range of 20-1000 °C

Weight Loss Sequence	Weight Loss,%	Volatile components of the heated sample	Stages of decomposition, °C
Δm_1	11,75	H_2O	20-180
Δm_2	1,0	H_2O+OH	180-240
Δm_3	81,25	CO_2	240-660
$\sum\Delta m_{1000°C}$	94	H_2O, OH, CO_2	20-1000

Adsorption

The effect of different pH values of the model solution prepared for adsorption process on the adsorbent is shown in Figure 5. According to the results obtained, the pH value was tested within the range of 3.0; 6; 9.0 and respectively, the adsorption reached from 38.19 % to 54.77 %.At pH - 3.0 the adsorption of Ni (II) metal ions was high. And at high pH value, the adsorption of Ni (II) is not high. In the purification of Ni (II) heavy metal ions through adsorption process for PSS adsorbents pH=3.0, contact time 360 minutes, Ni (II) concentration = 50 mg/l showed the maximum result.

Table 4 below summarises the results of the analysis obtained from the AES instrument of adsorbents. Based on the results obtained, the PSS adsorbent showed a yield of 54.77 % in 1 g/L mass.

Table 4

Effect of contact time and pH on the adsorption of Ni(II) on the adsorbent (PSS)

Results of the analysis of the effect of the reaction time on the adsorption process AAES Experience	Time of reaction	Volume *ml*	[cat./ ads.] *g/L*	pH	[Ni (II)] *mg/L*	[Ni (II)] *mg/L·25*	%
PSS (pumkin seed shell)	0	25	1	3	1,99	49,75	100
	15 min				1,72	43	13,56
	30 min				1,53	38,25	23,12
	60 min				1,36	34	31,66
	120 min				1,24	31	37,69
	240 min				1,11	27,75	44,22
	360 min				0,9	22,5	54,77
PSS (pumkin seed shell)	0	25	1	6,8	1,99	49,75	100
	15 min				1,14	28,5	42.71
	30 min				1,13	28,25	43.21
	60 min				1,11	27,75	44.22
	120 min				1,09	27,25	45.22
	240 min				1,01	25,25	49.24
	360 min				0,97	24,25	51,26
PSS (pumkin seed shell)	0	25	1	9	1,99	49,75	100
	15 min				1,81	45,25	9,04
	30 min				1,73	43,25	13,06
	60 min				1,69	42,25	15,07
	120 min				1,6	40	19,6
	240 min				1,4	35	29,65
	360 min				1,23	30,75	38.19

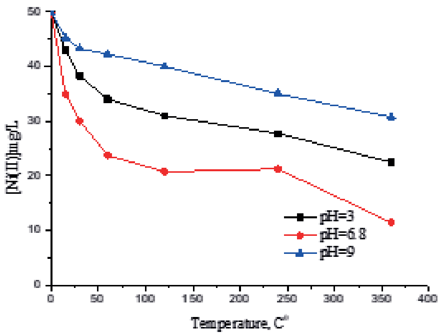


Figure 5 The effect of contact time and pH on removing Ni (II) by adsorption of pumpkin seed shells. Conditions: Ni(II) concentration = 50 mg/l, 2.5 g/l adsorbent, pH = 3; pH = 6.8; pH = 9.

The effect of different pH values of the model solution prepared for adsorption process on the activated angle obtained from RYY is shown in Figure 6. Based on the results obtained, the pH value was tested at 3.0; 6.8; 9.0 and respectively adsorption was achieved from 43.21 % to 76.88 %. At pH=6.8, the adsorption of Ni (II) metal ions was high. And at high and low pH value, the adsorption of Ni (II) is not high. In the purification of heavy metal ions Ni (II) through adsorption process for adsorbents AC fom PSS pH=3.0, contact time 360 minutes, Ni (II) concentration = 50 mg/L, adsorbent mass = 1 g showed the maximum result.

Table 5 below shows the results of the analyses obtained from the AES instrument of the adsorbents. Based on the results obtained, AC from PSS adsorbent showed a yield of 76,88% in 1 g/L mass, with a value of pH=6.8.

Table 5

Effect of contact time and pH on the adsorption of Ni(II) on the adsorbent (AC from PSS)

Results of the analysis of the effect of the reaction time on the adsorption process AAES Experience	Time of reaction	Volume *ml*	[cat./ ads.] *g/L*	pH	[Ni (II)] *mg/L*	[Ni (II)] *mg/L·25*	%
Activated carbon from PSS	0	25	1	3	1,99	49,75	100
	15 min				1,83	45,75	8,04
	30 min				1,79	44,75	10,05
	60 min				1,76	44	11,56
	120 min				1,46	36,5	26,63
	240 min				1,24	31	37,69
	360 min				1,10	27,5	44,82
Activated carbon from PSS	0	25	1	6,8	1,99	49,75	100
	15 min				1,4	35	29,64
	30 min				1,2	30	40
	60 min				0,95	23,75	52,26
	120 min				0,85	21,25	57,28
	240 min				0,83	20,75	58,29
	360 min				0,46	11,5	76,88
Activated carbon from PSS	0	25	1	9	1,99	49,75	100
	15 min				1,92	48	3,51
	30 min				1,81	45,25	9.04
	60 min				1,64	41	17.59
	120 min				1,38	34,5	30.65
	240 min				1,29	32,25	35,18
	360 min				1,13	28,25	43,21

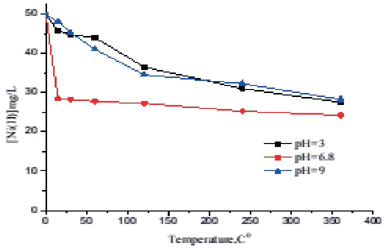


Figure 6 The effect of contact time and pH on removing Ni(II) by adsorption of AC from PSS. Conditions: Ni (II) concentration = 50 mg/l, 2.5 g/l adsorbent, pH = 3; pH = 6.8; pH = 9.

Adsorption was carried out for 6 hours with an interval of 15 to 120 minutes. The results obtained are shown in Figure 5,6. It was found that the optimal absorption of nickel ions takes place at pH 6.8 for 1 hour, which is associated with an increase in the available surface area and, in turn, increases the availability of interchangeable sites on carbon for Ni(II) adsorption. The optimal uptake of nickel ions at pH 6.8 within 1 hour can be explained by several factors. First, pH 6.8 is close to the optimum pH for nickel ion uptake by carbon materials. At this pH, the surface charge of the carbon material is likely neutral or slightly positive, which favors the adsorption of nickel ions. Second, at pH 6.8, the surface of the carbon material is likely to have interchangeable regions on the surface of the carbon material, which maximizes adsorption capacity. Third, at pH 6.8, it is possible to remove impurities or contaminants that may interfere with the adsorption process. It is observed that the adsorption of Ni(II), at pH 3, increases with the shell of pumpkin seeds from 13.56 to 54.77 %, and the AC from PSS from 10.05 to 44.82 %. (Figure 6). At a pH of 6.8 with a pumpkin seed shell from 8.04 to 51.26 %, and AC from PSS from 29.64 to 76.88 %. However, with an increase in pH to 9, the degree of nickel (II) adsorption decreases for pumpkin seed shells from 9.04 to 38.19 %, and for AC from PSS from 3.51 to 43.21 %.

Conclusion

Using natural waste materials as adsorbents to absorb heavy metals from aqueous solutions shows encouraging results.

The chemical and physical properties of the obtained adsorbent are determined. It was found that several parameters, such as pH and contact time, influence the adsorption process. Regression dependences of Ni extraction from aqueous solutions have been found, which make it possible to establish optimal sorption parameters: pH = 6.8, T = 25 °C, and the time for establishing sorption equilibrium is 360 minutes.

The carbon content in the activated carbon samples obtained for various activation conditions was increased compared to the shell of pumpkin seeds due to an increase in the larger surface of the adsorbent and at a dose of 0.250 g/L of the adsorbent obtained from the shell of pumpkin seeds and the initial concentration of nickel ions in a solution of 50 mg/L the results of the study convincingly proved the effectiveness of the AC from PSS as adsorbents for the removal of Ni(II) from wastewater.

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