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INVESTIGATION OF THE EFFECT OF WALNUT SHELL PROCESSING METHODS ON THE RELEASE OF ANTIOXIDANT SUBSTANCES

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The article presents the results of studies of the effect of processing methods on the output of antioxidant substances from walnut shells. Technological processes and modes of extract production are substantiated. At the same time, the size of the crushed shell, the extraction time and the concentration of the solvent were investigated.

Most of the nuts are shells and husks, low-value by-products rich in phenolic compounds. It is possible to organize production in which the shell will be concentrated and processed to isolate valuable biologically active substances, on the basis of which new domestic drugs of various therapeutic and preventive effects

can be developed.Phenolic compounds extracted from walnut shells are potentially good natural sources of antioxidants for the food industry and have numerous health benefits. Because of the special quality of lignin, it is very important to split and loosen the dense structure so that the solvent penetrates into the cell to extract phenolic compounds. Thus, in order to obtain a higher yield of phenolic compounds, the correct method of isolating antioxidant substances is crucial.

Key words: walnut shell, extraction, extract, extragent, size, concentration.

ГРЕК ЖАҢҒАҒЫ ҚАБЫҒЫН ӨҢДЕУ ӘДІСТЕРІНІҢ АНТИОКСИДАНТТЫ ЗАТТАРДЫҢ БӨЛІНУІНЕ ӘСЕРІН ЗЕРТТЕУ

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Мақалада грек жаңғағы қабығынан антиоксидантты заттардың шығуына өңдеу әдістерінің әсері туралы зерттеу нәтижелері келтірілген. Сығынды алудың технологиялық процестері мен режимдері негізделген. Ұсақталған қабықтың мөлшері, экстракция уақыты және еріткіштің концентрациясы зерттелді.

Грек жаңғағының көп бөлігі қабықтан тұрады – ол фенолдық қосылыстарға бай, құны бойынша аз бағаланатын жанама өнімдер. Әртүрлі емдік және профилактикалық әрекеттердің жаңа отандық препараттарын жасауға болатын құнды биологиялық белсенді заттарды бөліп алу үшін қабық шоғырланатын және өңделетін өндірісті ұйымдастыруға болады. Жаңғақ қабығынан алынған фенолды қосылыстар тамақ өнеркәсібі үшін антиоксиданттардың жақсы табиғи көзі болып табылады және денсаулыққа көптеген артықшылықтар береді. Лигниннің ерекше сапасына байланысты фенолдық қосылыстарды алу үшін еріткіш жасушаға енуі үшін тығыз құрылымды ыдырату және қопсыту өте маңызды. Осылайша, фенолдық қосылыстардың жоғары өнімділігін алу үшін антиоксидантты заттарды оқшаулаудың дұрыс әдісі өте маңызды.

Түйінді сөздер: жаңғақ қабығы, экстракция, сығынды, эстрагент, мөлшер, концентрация.

ИССЛЕДОВАНИЕ ВЛИЯНИЯ МЕТОДОВ ОБРАБОТКИ СКОРЛУПЫ ГРЕЦКОГО ОРЕХА НА ВЫДЕЛЕНИЕ АНТИОКСИДАНТНЫХ ВЕЩЕСТВ

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В статье представлены результаты исследований влияния способов переработки на выход антиоксидантных веществ из скорлупы грецкого ореха. Обоснованы технологические процессы и режимы получения экстракта. В то же время были исследованы размер измельченной скорлупы, время экстракции и концентрация растворителя.

Большая часть орехов состоит из скорлупы и шелухи – малоценных побочных продуктов, богатых фенольными соединениями. Возможно организовать производство, в котором скорлупа будет концентрироваться и перерабатываться для выделения ценных биологически активных веществ, на основе которых могут быть разработаны новые отечественные препараты различного лечебного и профилактического действия.Фенольные соединения, извлеченные из скорлупы грецкого ореха, потенциально являются хорошими природными источниками антиоксидантов для пищевой промышленности и обладают многочисленными преимуществами

для здоровья. Из-за особого качества лигнина очень важно расщеплять и разрыхлять плотную структуру, чтобы растворитель проникал в клетку для извлечения фенольных соединений. Таким образом, для получения более высокого выхода фенольных соединений решающее значение имеет правильный метод выделения антиоксидантных веществ.

Ключевые слова: скорлупа грецкого ореха, экстракция, экстракт, эстрагент, крупность, концентрация.

Introduction. In recent years, much attention has been paid to the assessment of polyphenol content and the antioxidant activity of various plant materials, especially of nuts, since their regular consumption is associated with a reduction in the risk of health issues as cancer and cardiovascular diseases [1, p. 380].

Walnut is a valuable crop due to its popularity and wide consumption among population. Green walnut, shell, peel, kernel, bark, root and leaves are widely used in the pharmaceutical and cosmetic industries [2, p. 349]. Walnut is also recognized as a rich source of various valuable chemicals, since the kernel, fresh green fruits, husk, shell, skin, bark, leaves and root have been comprehensively studied for use in the food, cosmetic and pharmaceutical industries. In this regard, all parts of the walnut tree can be used as an excellent source of various compounds with antioxidant and antimicrobial potential, as well as antidepressants with antidiabetic, immunomodulatory, hepatoprotective, stimulating effects to the central nervous system. Also, it`s worth noting the inflammatory, wound healing, and many other properties of walnut tree that positively affect human health [3, p. 751].

Walnuts are not only used in human nutrition, but also young green walnuts are highly valued in traditional folk medicine in some countries, as well as for making jams and a healthy alcoholic drink called nut liqueur, which contains a large amount of phenolic compounds and vitamins, where the fruits are left to soak in food ethanol. This liqueur is made from fresh walnuts with green husks just before the endocarp hardens [4, p. 627].

Agriculture produces a significant number of by-products, rich in phenolic compounds, which are facing increasing interest due to their excellent antioxidant properties [5, p. 349]. At the same time, walnut is recognized as one of the agricultural crops that produce more waste. It is estimated that about 70% of the weight of the fruit is the shell and peel, low-value waste rich in various chemicals, mainly phenolic compounds [6, p. 59].

The walnut shell surrounding the nut is a product of agricultural waste, which is widely used in folk medicine for the treatment of skin diseases. However, in recent years, it has received more and more attention in modern pharmacology, mainly due to its antioxidant properties [7, p. 193]. It has been valued as a source of natural compounds with antioxidant and antimicrobial properties [8, p. 126]. The effective technology of the shell utilization is a critical issue because its use as an abundant source of phytochemicals will emphasize the importance of walnut production, and suggest the use of a by-product that is produced in large quantities [9, p. 232].

Therefore, the scientific novelty of this article is the study of the influence of walnut shell processing methods, methods affecting the extraction of antioxidant substances from walnut shells.

Materials and methods

Materials and methods

The material for the study is a walnut shell. Extraction was carried out on a semi-automatic device using the "ASV-6" Soxlet.

Grinding and at the laboratory mill "MSHL-1P". The mill "MSHL-1P" is a periodic device. The removable drum of the mill is filled with pre-crushed dried walnut shell and grinding steel balls on the "Novital Magnum 4V" crusher. When the drum rotates, the material is crushed as a result of the abrasion and impact action of the balls. The grinding time depends on the fineness of the grinding and varies from 1 to 3 hours.

Extraction on a semi-automatic extraction apparatus according to the "ASV-6" Soxlet. To begin the analysis, samples are prepared for extraction. Filter paper sleeves are made, and crushed walnut shells in an amount of 5 g are placed in it. 45 ml of solvent (water, ethanol) is poured into the extraction flask and installed on a water bath, lifting the corresponding glass refrigerator and the sample installed in it. After reaching the set temperature, the sample is moved to the solvent, where the sample is processed for 30 minutes. After that, the sample is transferred to the position for washing with a clean solvent. The process of washing with a clean solvent is the main stage of extraction, this stage takes place within 60-180 minutes. After the extraction is completed, the solvent passes into the upper part of the refrigerator for 30 minutes, and the extracted substance remains in the extraction flask. The total phenol content was evaluated by the Folin–Chocalteu colorimetric method, and the results were expressed in milligrams of gallic acid equivalents (mg GAE/extract).

The achievement of the set goals and objectives will be based on the use of the following Technical specifications and GOST standards: GOST 32874-2014 "Walnuts. Technical specifications"; GOST 17299-78 "Ethyl alcohol. Technical conditions". GOST R 57990-2017 "Method of determination of quercetin". GOST ISO 14502-2-2015 "Method for determining the content of catechins".

Results and discussion

As the demand for natural antioxidants in the food industry is growing dynamically, agricultural and food waste is becoming an ideal material for the extraction of phenolic compounds as natural antioxidants.

To isolate antioxidant substances, the extraction method is most often used. The antioxidant activity and the amount of extraction yield are related to the solvent used. Most often, methanol, ethyl alcohol, chloroform, water, N-butanol and ethyl acetate are used to extract antioxidant compounds, whilst for food purposes, the organic compounds are used.

In this study, several types of solvents of different polarities (water, ethanol) were used to extract walnut shells. Ethanol and water were chosen as extraction solvents not only due to their higher extraction yields, but also because they are safer and less toxic compared to methanol and other organic solvents.

The extraction was carried out on a semi-automatic Soxlet device "ASV-6". In the future, according to the results of the research, the optimization of the extraction modes of walnut waste was implemented on the basis of a full-factor experiment, and the regularities regarding walnut shell extraction were established.

To conduct a full-factor experiment of the extraction process, certain variables were considered, namely:

-the grinding degree of raw materials. The crushing of the shell was carried out in two stages. Preliminary crushing with the "Novital Magnum 4V" crusher to fractions of 1-2.5 mm and final – to fractions of 300 μm on the laboratory ball mill "MSHL-1P". The duration of crushing varies from 20 minutes to 3 hours depending on the size of the grinding;

-the type of extractant used. Extraction was carried out using the following solvents: water, water-ethanol in various ratios, and ethanol;

-time of extraction.

To obtain a mathematical model of the technological process, which is a regression equation, a rotatable second-order plan (Box plan) was used (Table 1).

Table 1 – Coding of intervals and levels of variation of input factors

Factors		Levels of variation					Variation intervals
Natural	Encoded	-1,68	-1	0	+1	+1,68	
Solvent concentration	x_1	50	60	70	80	90	10
Fineness of grinding	x_2	300	400	500	600	700	100
Duration of extraction	x_3	60	90	120	150	180	30

The analysis of variance for a quadratic model of the response surface is shown below (Table 2).

Table 2. Analysis of variance for a quadratic model of the response surface.

Encoded values			Natural values			Optimization criteria
x_1	x_2	x_3	C, %	K , μm	t, min	Y
-1	-1	-1	60	400	90	3,05
-1	-1	1	60	400	150	3,25
-1	1	-1	60	600	90	2,96
-1	1	1	60	600	150	3,11
1	-1	-1	80	400	90	3,85
1	-1	1	80	400	150	4,15
1	1	-1	80	600	90	3,02
1	1	1	80	600	150	3,15
-1,68	0	0	60	500	120	3,08
1,68	0	0	90	500	120	3,45
0	-1,68	0	70	300	120	4,67
0	1,68	0	70	700	120	3,09
0	0	-1,68	70	500	60	3,05
0	0	1,68	70	500	180	4,07
0	0	0	70	500	120	2,63
0	0	0	80	300	120	4,57
0	0	0	90	500	90	3,17
0	0	0	60	500	90	2,59
0	0	0	90	300	120	5,02
0	0	0	70	500	150	3,33

The resulting regression equation for the extracting process, for the encoded values of the input variables, has the following form:

$$y = 3,56 + 0,18x_1 - 0,35x_2 + 0,18x_3 - 0,2x_1x_2 + 0,01x_1x_3 - 0,03x_2x_3 - 0,15x_1^2 + 0,06x_2^2 - 0,5x_3^2. \quad (1)$$

After the transition from the encoded to the natural values of the independent variables, the regression equation acquired the following form:

$$B = 3,56 + 0,18C - 0,35K + 0,18t - 0,2CK + 0,01Ct - 0,03Kt - 0,15C^2 + 0,06K^2 - 0,5t^2. \quad (2)$$

The values of the intervals for estimating the significance of the coefficients of the regression equation have the form: $\Delta b_0 = \pm 0.83$, $\Delta b_i = \pm 0.55$, $\Delta b_{ii} = \pm 0.54$, $\Delta b_{ij} = \pm 0.72$.

After walnut shell extraction, 3 different ratios were obtained as the best result.

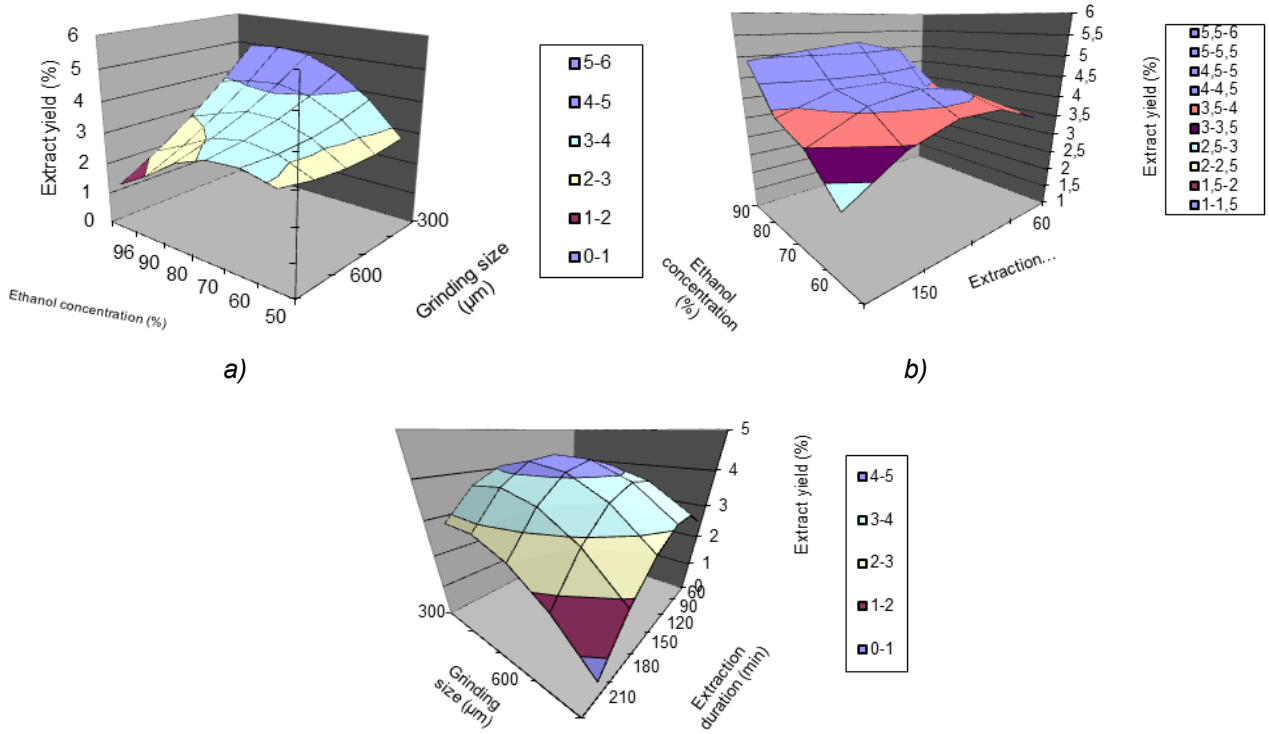
These include:

1) the ratio of ethanol to water (70% + 30%), the grain size of the shell is 300 μm, the extraction time is 150 minutes.

2) the ratio of ethanol to water (80% + 20%), the grain size of the shell is 300 μm, the extraction time is 120 minutes.

3) the ratio of ethanol (90%) the ratio of ethanol, the grain size of the shell is 300 μm, the extraction time is 150 minutes.

Figure 1 shows the response surfaces describing the values of the yield of the extract from the walnut shell from the percentage of ethanol, the duration of extraction and the fineness of grinding for the extraction process.



a – dependence (%) on the concentration of ethanol (%) and the fineness of grinding (μm) for the extraction duration of 120 min; b – dependence (%) on the concentration of ethanol (%) and the duration of extraction (min) for the fineness of grinding 300 μm; c – dependence (%) on the duration of extraction (min) and grinding fineness (μm) at an ethanol concentration of 80%

Figure 1 – Dependence of the extraction yield of antioxidant substances from walnut shells on the percentage of ethanol, the duration of extraction and the fineness of grinding

As can be seen from Figure 1, in the selected area of input variables, there are optimal values that ensure maximum extract output.

Thus, Figure 1a shows the results obtained with an extraction duration of 120 min. It follows from the graph that with a concentration value of more than 70% ethanol and a grinding fineness of less than 400 μm, an increase in extract yield is observed, with a maximum value of 5.0%.

Figure 1b shows the results obtained with a grinding fineness of K-300 μm. It follows from the graph that when the extraction duration is over 70 min and the ethanol concentration is above 60%, an increase in the extract yield is observed, with a maximum value of 3.75%.

Figure 1c shows the results obtained at an ethanol concentration of 80%. It follows from the graph that when grinding is less than 400 μm and the extraction duration is over 90 min, an increase in the extract yield is observed, with a maximum value of 4.75%.

It has been established that ethanol and water as extraction solvents lead to higher extraction yields, they are also safer and less toxic compared to methanol and other organic solvents. With a grinding fineness of 300 μm, there was established an dependence of an increase in the extraction yield from 2.71 to 4.93% with an increase in the ethanol concentration from 50 to 90%, as well as an increase in the extraction yield from 3.80 to 4.81% with an extraction duration from 60 to 180 min, however the maximum extraction value was reached after 150 min.

The analysis of the effect of the grinding fineness on the extraction duration (Figure 1) shows that the extraction yield decreased to 1.4 mg when the grinding fineness was above 600 μm. At the same time, the longer the extraction was carried out, the greater the yield of the extract.

Based on the analysis of the data obtained, it was revealed that solvents, grinding and duration are the basis of extraction of antioxidant substances from the walnut shell.

The results obtained are limited by the selected limits of variation of technological parameters: ethanol concentration 60-90%; grinding fineness 300-700 μm; extraction duration 60-180 min.

Next, the flavonoid composition of the walnut shell was studied in the obtained extract samples (in the amount of 100 mg of each sample), the level of quercetin and catechin was studied. The flavonoid composition of walnut shell is characterized by the content of quartzetin, catechin – antioxidants very useful for the heart, contributing to the protection of brain functions, supporting connective tissue and improving blood circulation, having antibacterial effect.

The level of quercetin and catechin is shown in Figure 2.

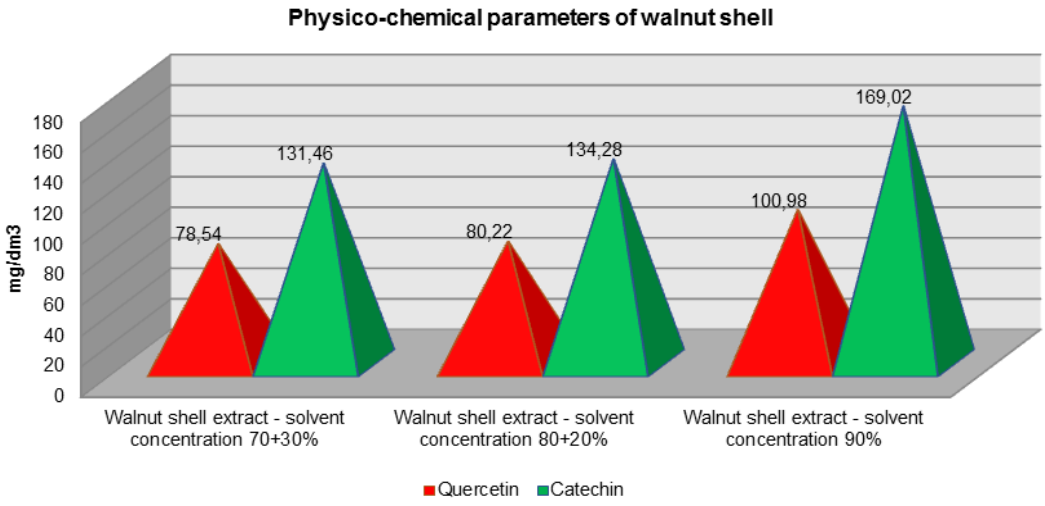


Figure 2 – Catechin and quercetin content in walnut shell extract, mg/dm^3

As can be seen from Figure 2, as the solvent concentration increases, the content of quercetin and catechin increases. The maximum value was in the extract obtained with a 90% solvent. The maximum value of catechin is 169.92 mg/dm3, and quercetin is 100.98 mg/dm3. Considering that all antioxidants protect the body from damage by harmful free radicals – toxins that enter from the environment and damage healthy cells, leading to inflammatory processes, it is necessary to note the significant role of the flavonoid composition of walnut shells as one of the components in determining the further direction of research.

Conclusions

Based on the results of the conducted research, the following conclusions can be drawn:

- extraction of several types of solvents (water, ethanol) was carried out;
- optimization was carried out. By the method of mathematical modeling, the most optimal extraction modes were identified, in which all the antioxidant properties of the walnut shell are most fully extracted;

By quantitative ratio:

- according to the optimization results, it was revealed that during extraction with 90% ethanol, the extraction time was 150 minutes, while the extract yield was the highest;
- when extracted with the "ethanol + water" extractant in a ratio of 70/30, the extraction time was 150 minutes, while the maximum extract yield was the highest;

- when extracted with the "ethanol + water" extractant in a ratio of 80/20, the extraction time was 120 minutes, while the maximum extract yield was the highest.

Based on the analysis of the data obtained, it was revealed that solvents, grinding and duration are the basis for the regularity of the release of antioxidant substances from the walnut shell. Knowledge of the laws of extraction of antioxidant substances from walnut shells will allow you to regulate the extraction process and obtain products with specified characteristics and parameters.

According to the results of the study, the most optimal extraction mode was determined, in which the antioxidant properties were extracted as much as possible.

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УРОВЕНЬ УРОЖАЙНОСТИ НОВЫХ ОТЕЧЕСТВЕННЫХ СОРТОВ СОИ НА ОРОШАЕМЫХ ПОЧВАХ ЮГО-ВОСТОКА КАЗАХСТАНА В ЗАВИСИМОСТИ ОТ НОРМЫ ВЫСЕВА

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В статье приведены исследования влияния нормы высева на урожайность отечественных сортов сои. Сделаны выводы по рентабельности производства сортов сои. Приведен расчет экономической эффективности возделывания сортов сои с разными нормами высева. Показана высокая прибыль изучаемых сортов сои в зависимости от норм высева, а также средняя урожайность сортов сои по годам исследования в зависимости от норм высева. В исследованиях принимали участие пять среднепоздних и позднеспелых сортов сои Ласточка, Жансая, Ай Сауле, Акку, Айзере. Сорта допущены к производству на юге и юго-востоке Казахстана. Результаты всех трех лет исследований показывают самые высокие урожайности у всех изучаемых сортов при норме высева 600 тыс./га. Наиболее продуктивный был сорт сои Ай Сауле при норме высева 600 тыс./га. Наименьшей продуктивностью характеризовался крупносеменной сорт сои Айзере с диапазоном урожайности 32,9-48,8 ц/га при норме высева 200 тыс./га. При использовании разных сортов с учетом посевных норм, увеличение стоимости работ на гектар колеблется в пределах 216800 – 276200 тенге. Наибольшую прибыль в размере 1145900 тенге с га получили на сорте соя Ай Сауле.

Ключевые слова: соя; сорт, норма высева; урожайность; признаки продуктивности.