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# SYNTHESIS OF METAL NANOPARTICLES USING PLANTS TO ENHANCE THE GROWTH AND GERMINATION OF AGRICULTURAL CROPS

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**Abstract:** *Introduction***.** Nanoparticles of several metals obtained through biosynthesis have interesting pharmacological properties due to extract components' sorption on their surface, which broadens their application possibilities. For these reasons, the issue of metal nanoparticles synthesis biological ways optimization is highly important nowadays. *Purpose* of the work is to present experimental data on the synthesis of metal nanodispersions using plant extracts of *Artemisia (wormwood), Psylliostachys (plantain), Achillea nobilis (yarrow)* and use in growing crops. *Methodology.* A feature of this method is the use of a more environmentally friendly approach to the synthesis of nanoparticles by preparing extracts from plant materials. Plant raw materials for synthesis, synthesis conditions (temperature, pH value, ratio of metal salts: extract) were experimentally selected. It was found that in presence of extract from bitter wormwood nanoparticles formation occurs faster than in case of using *Psylliostachys* or *Achillea nóbilis.* Sythesized silver nanoparticles were studied using microscopy and spectroscopy. *Results.* Obtained particle sizes are between 70 and 100 μm, which proves nanostructured nickel and cobalt particles formed in synthesis. Average particle size is 80 μm. New growth stimulator M-Nitro-Ni,Co was used to increase annual grasses and vegetables growth and germination; as a result, they had stronger roots, thicker stems, dark-green leaves and larger size comparing to the test ones.

**Keywords:** nanoparticles, extract, metal nanodispersion, green synthesis, *Artemisia (wormwood), Psylliostachys (plantain), Achillea nobilis (yarrow)*


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## 1. Introduction

Nowadays there are several approaches to metal nanoparticles synthesis: chemical, physical and biological ones. The latter is of high interest, especially biological ways of metal nanoparticles synthesis using plant extracts, which are often viewed as quick, eco-friendly and easily scalable technology. Metal nanoparticles formation occurs due to deoxidation by plant-based metabolites (components of phenol origin, terpenes etc.) which go over to the extract. This is why nowadays there is an important issue of improving biological synthesis methods, expanding range of used raw plants as well as of studying obtained nanodispersions' properties[1-3].

Existing synthesis methods rarely solve the issue of optimizing conditions of extraction from raw plants, particularly, acceleration of extraction process without efficiency loss or use of eco-compatible solvents. Another issue that needs a solution is conditions of metal nanodispersion synthesis: selection of raw plants, synthesis temperature and pH value. One more important point is further use of obtained nanodispersions, as plant-based metabolites presence in product makes it more compatible with living organisms. This leaves room for considering adding some metal nanodispersions to medical substances or their use as active components. The abovementioned issues require complex approach to their solution and comprehensive study of both each stage of metal nanodispersion synthesis and evaluation through a number of parameters (morphological properties, microbiological activity) of final synthesis product [4-7].

## 2. Experimental part

*Soil pH definition.* Soil water extract samples in enumerated cylindrical vessels are sent to ion meter. pH index study is done in a parallel way. The procedure on I-160 brain ion meter looks the following way: the device is powered and heated for 15 minutes. Pair of electrodes is dried with filter paper and washed with tested part of a studied solution. After the electrodes were submerged into solution and the readings were stabilized, measurement is taken.

The research involved plants collected in summers of 2020-2022 in Bayan-aul uplands and areas around Astana, Kazakhstan – *Artemisia (wormwood), Psylliostachys (plantain), Achillea nobilis (yarrow).* Dried pants were ground into fine powder and stored in special exiccators till extraction.

*Obtaining extract.* Extraction was done with 70% ethanol for 12 hours. The solution was steamed in rotary evaporator at +50°C and concentrated on water bath with ratio up to 15%. Obtaining ethanol-based extract from bitter wormwood leaves (as well as yarrow flowers, plantain stems and leaves). 0.2 g of bitter wormwood sample, ground, was put into conical flask with thin section, 100 ml of ethanol solvent were added and left to infuse in a windless place at 50±10ºC for 24 hours. Then obtained extract was filtered into 100 ml measuring flask.

*Definition of active groups in plant-based extracts.* Various plant metabolites consisting of terpenoids, polyphenols, alcaloids, phenolic acids and proteins can deoxidize metal ions to nanoparticles. This is why mentioned groups should be defined.

*Qualitative reaction to flavonoids.* A few drops of (NaOH) were added to the extract, followed by 2-3 drops of HCl. Presence of flavonoids was defined by alkaline solution discoloration after adding HCl.

*Qualitative reaction to saponins.* Presence of saponins was defined by massive formation od strong foam after shaking the extract water solution.

*Qualitative reaction to phenols.* Presence of phenols was defined by extract obtaining purple (black) color after adding 1% $FeCl_3$.

Research was performed using commonly-accepted scientific and experimental methods. The study used modern facilities of S.Seifullin KATRU, "Physical-chemical research methods" engineering laboratory: atomic microscope JEOL JEM-2100, detection electronic microscope MIRA 3 TESCAN» Oxford Instruments, detector PIXcel PanAlytical X'Pert.

In order to study the use of deoxidized nanoparticles as new growth stimulators for agricultural crops, plants giving quick and quality harvest – radish, parsley, tomato, and dill – were selected.

## 3. Results and discussion

Area of South Black-earth type steppes is 13.4 million hectares. It is seldom found in West Kazakhstan, Aktobe, Karaganda regions but is common in Akmola, Pavlodar and Kostanay regions.

Physical-chemical properties of southern black-earth soil are given in Table 1.

**Table 1 –** Physico-chemical characteristics of soil

| **№** | **Soil name, location** | **The layer from which the sample was taken, cm** | **Humus** | **Total nitrogen** | **Exchangeably adsorbed cations,м – eq./ 100 g** | | | | **pH** | **Granulometric composition** | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Ca | Mg | Na | Total | | <0,01 мм | <0,001 мм |
| 1 | Black soil in the south (Pavlodar region) | 0 – 10 | 5.8 | 0.310 | 20.9 | 3.5 | 0.2 | 24.6 | 6 | 42.2 | 21.6 |
| 2 | | 20 – 30 | 2.3 | 0.170 | 17.4 | 3.5 | 0.2 | 21.1 | 6.5 | 42.3 | 28.8 |
| 3 | | 35 – 45 | 1.6 | 0.130 | 15.6 | 3.5 | 0.3 | 19.4 | 7 | 37.4 | 26.2 |
| 4 | | 70 – 80 | - | - | - | - | - | - | 8.4 | 29.5 | 23.6 |

Southern black-earth soil composition and properties are similar to standard black-earth soil. Its genetic layers are: A – $B_1$ – $B_2$ – C. One of the main features of southern black-earth soils and regular ones is that they are well developed and well seen in humic tongues layer $B_2$. Humic layer A average thickness is 24 cm, in plowing areas – 21-30 cm. It has dark-grey color with burgundy-red tint. Thickness of the layer $B_1$ – 16-29 cm. Color varies from dark burgundy-red to grey-burgundy-red. Thickness of the layer $B_2$ – 19-33 cm, coloring is uneven, humic tongues are easily seen. Intermediate layers BC are compressed, carbonates are abundant.

Maximum hygroscopical content of water in humic layer of this soil is 11.5-11.7, plant drooping humidity is 14.1-16.8, natural humidity ratio is 30,2-38,9. Southern black-earth soils can be sandy, sandy loam and clay according to their granulometric content.

Phytochemical screening of extracts from plants growing in Bayan-Aul area, Northern Kazakhstan. Results of plant extracts phytochemical analysis are given Table 2. The analysis suggests that phenols and flavonoids are found in bitter wormwood (*Artemisia*), plantain (*Psylliostachys*) and yarrow (*Achilléa nóbilis*), and the most saponins were found in yarrow.

**Table 2 –** Phytochemical screening

| № | Active groups | *Artemisia* | *Psylliostachys* | *Achilléa nóbilis* |
|---|---|---|---|---|
| 1 | Flavonoid | + | + | + |
| 2 | Phenol | + | + | + |
| 3 | Saponin | + | - | - |

*Nanoparticles synthesis.* During synthesis of nickel nanoparticles using plant extracts, color change in solution and nanoparticles themselves can be seen. However, color change may be caused by various factors, such as metal concentration, reaction conditions and used plant-based material.

*Extract: nickel nitrate solution ratio optimization process.* Metal solution to extract ratio optimization in metal nanoparticles synthesis is an important step that can affect quality and characteristics of obtained nanoparticles. Depending on specific experiment conditions, optimal metal-to-extract ratio can be different. Volume ratio of extract to $Ni(NO_3)_2$ solution is important for reaction between system components. One of ways to optimize metal solution to extract ratio is change of components' concentration. In this case it should be considered that too high concentration of either metal or extract may lead to side effects such as nanoparticles' aggregation or non-homogeneity.

The following ratio was studied: extract:solution $Ni(NO_3)_2$ 1:9 and extract solution:$CoCl_2$ 3:7. In the second case color saturation was quicker. Further work was done on this ratio.

*Reaction mixture pH optimization.* Another way of optimization is to change reaction environment pH. Studies show that optimal pH may vary depending on plant extract and metal type. For example, study [9] found that optimal pH for gold nanoparticles synthesis with mint leaves extract was 9, while optimal pH for nickel and cobalt nanoparticles synthesis with bitter wormwood extract is 7(Table 3).

**Table 3 –** HCl concentration and volume

| pH value | C(HCl), mol/l | V(HCl), ml |
|---|---|---|
| 3 | $5*10^{-2}$ | 1 |
| 4 | $5*10^{-2}$ | 0.1 |
| 5 | $4*10^{-4}$ | 5 |
| 6 | $4*10^{-4}$ | 0.5 |

Since reaction mixture pH is highly influential for synthesis outcome, its optimal value should be found. According to reference data, nickel and cobalt nanoparticles are formed in low-acidic environment, so synthesis was performed at pH values of each kind. According to the method, reaction was done by adding HCl to a certain pH in mixture.

Microscopy methods were used to describe nanoparticles structure and distribution, including size, shape and other parameters. EMF evaluation showed relatively equal distribution of $Co^0$, $Ni^0$. Data on $Co^0$, $Ni^0$ distribution on 10 μm are given in Table 4,which suggests that in polymeric matrix M-Nitro-2023 metal yield is high; however, relative concentration non-homogeneity can be seen (Fig.1,2).

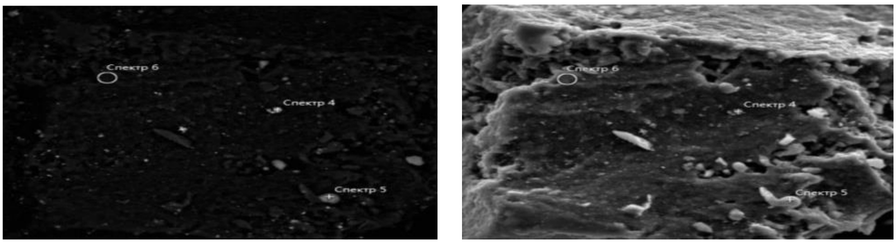
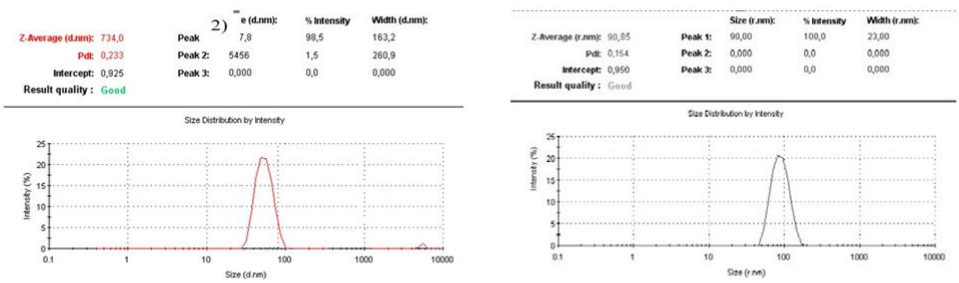



**Figure 1 –** Microphotographs of nickel and cobalt nanodispersion obtained with bitter wormwood extract (X-Act electronic microscope SEM)

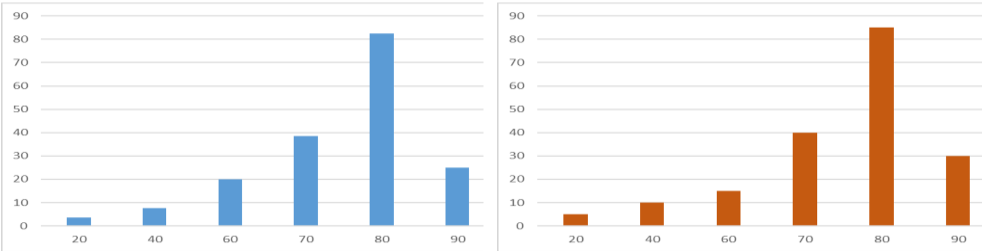
Based on data by Malvern Zetasizer Nano 28 ZS90, obtained particle sizes are between 70 and 100 μm, which proves nanostructured nickel and cobalt particles formed in synthesis. Average particle size is 80 μm (Fig. 2).




A – Co B – Ni

**Figure 2 –** Size distribution of nickel and cobalt nanoparticles

Microphotographs show that metals are close to lozenge and prism shape ($Co^0$), and $Ni^0$ are close to spherical shape. Nanoparticles distribution based n their average size was calculated to approximately 100 parts. Analysis was done by zooming microphotographs obtained using Sigma Scan software, nanoparticles distribution is shown further on the example of M-Nitro-Ni, Ni $Co^0$-2023 mono – M-Nitro (Fig.3,4).




a – Size distribution of $Ni^0$ nanoparticles b – Size distribution of $Co^0$ nanoparticles

**Figure 4 –** Size distribution of Ni, Co

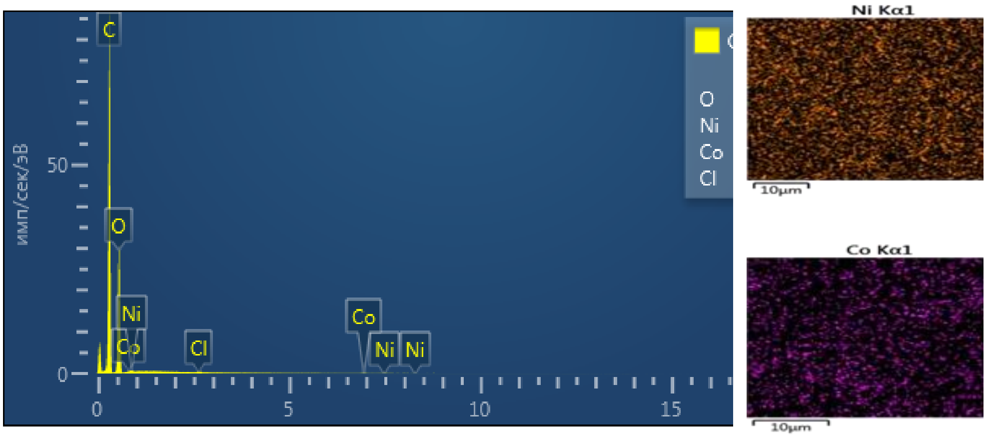



**Figure 3 –** Size distribution of $Ni^0$, $Co^0$ nanoparticles

Detailed information about the size, shape and distribution of $Co^0$, $Ni^0$ nanoparticles is summarized in Table 4, where: M-Ni-АЖ-2023 – surface characteristics of nickel nanoparticles in bitter wormwood; M-Co-АЖ-2023- characterization of cobalt nanoparticles in bitter wormwood. M-Ni-Мж–2023 – surface characteristics of nickel nanoparticles obtained from yarrow extract; M-Co-Мж-2023, M-Ni-Мж-2023 - surface characteristics of metal nanoparticles obtained from yarrow extract.

Ethanol extracts of bitter wormwood, yarrow and plantain created a favorable environment for deoxidation of nanoparticles of Ni,Co. Average amount of NPs in 10 μm:1200±10, lozenge, (M-Ni-Ac-2023); 1100±50, prism, (M-Co-АЖ-2023); 1800±150, prism, 1230±100 (M-Ni-Мж-2023); 1000±50 (M-Co-Мж-2023); 520±50 pcs. Ni/480±50 pcs. M-Ni-Жж-2023); 500±50 pcs. Co (M-Co-Жж-2023); volume sizes of nanoparticles M-Ni-АЖ-2023 were small, concentration was insignificant. Volume sizes and distribution of bitter wormwood and plantain nanoparticles were deoxidized by large amount of metal from bitter wormwood comparing to sprouts.

**Table 4 –** Main surface characteristics and complexes morphology

| Complex | NPs size, μm | NPs morphology | NPs in 10 μm |
|---|---|---|---|
| M-Ni-АЖ*-2023 | 70±5 | Lozenge | 1200±10 |
| M-Co-АЖ*-2023 | 73±5 | Lozenge, prism | 1100±50 |
| M-Ni-Мж*-2023 | 68±6 | Lozenge | 1230±100 |
| M-Co-Мж*-2023 | 74±5 | Lozenge, prism | 1000±50 |
| M-Ni-Жж*-2023 | 72±4 | Lozenge | 540±50 for Ni |
| M-Co-Жж*-2023 | 76±6 | Lozenge, prism | 500±50 for Co |

** АЖ – bitter wormwood; Мж – yarrow; Жж – plantain*

Studies of nanoparticles use as new growth intensifiers for agricultural crops show intensity and growth and germination peculiarities for the followning crops at the first stage: vegetables (tomato, radish); annuals (dill, parsley).

Table 5 below with seeds study results shows presence of rapid regionalizations of germination speed and growth energy in experimental and test versions.

**Table 5 –** Vegetables and annual plant seeds germination intensity

| Types of plant crops | Germination rate, % | | Relative superiority over the test indicator, % |
|---|---|---|---|
| | Growth stimulator use | Test (control) | |
| Dill | 75±3 | 67±3 | 12 |
| Radish | 85±3 | 75±3 | 13.3 |
| Parsley | 90±3 | 79±3 | 14 |
| Tomato | 71±3 | 63±3 | 12.7 |

Images of vegetables and annuals are given below (Fig. 5).To compare plant biomass and morphological features of the plants which used test and growth stimulator, they were separated from soil and compared to other plants of the same kind of 1–2 months old. Figure 5 shows comparative images of tomato sprouts.

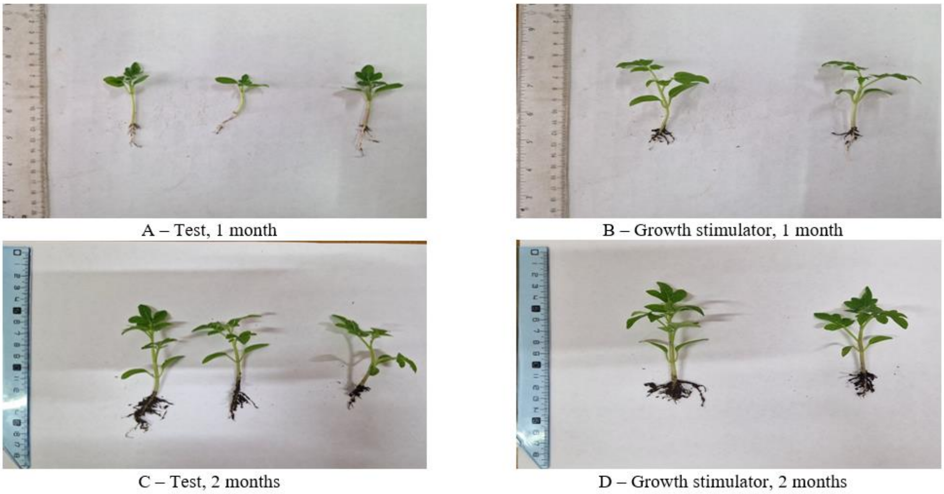



**Figure 5 –** Tomato morphological features definition

Growth intensity of vegetables grown using stimulator on southern black-earth soil increased by 12%, and one of annual plants – by 13%. Morphological features of young plants at 1–2 months are given in Table 6 (Fig. 5).

**Table 6 –** Morphological features of vegetables and annual plants

| Types of crops | Parameters | Height, cm | Diameter, cm | Leaf length, cm | Leaf width, cm | Number of leaves | Root length |
|---|---|---|---|---|---|---|---|
| Dill | Growth stimulator | 25 | 1.5 | 5 | 3 | – | 5.5 |
| | Test | 18 | 1 | 3.5 | 1.5 | - | 4 |
| Radish | Growth stimulator | 20 | 15 | 7 | 4 | 14 | 3.3 |
| | Test | 13 | 8 | 5.5 | 2.3 | 12 | 3 |
| Parsley | Growth stimulator | 10 | 8 | 3 | 4 | many | 3 |
| | Test | 10 | 7 | 1.5 | 2.5 | many | 2.3 |
| Tomato | Growth stimulator | 16 | 12 | 5 | 2.5 | 25 | 5.0 |
| | Test | 13 | 9 | 3 | 2 | 20 | 4.8 |

Comparing to new growth intensifier and test results, plants that used growth intensifier had strong roots, thick stems, dark-green leaves and overall larger size. Tomato with growth intensifier had thicker stem, stem length was 16 cm, and plant diameter was 12 cm. Test plant height was13 cm, plant diameter – approx. 9 cm. Thus, new growth intensifier M-Nitro-Ni,Co-2023 is effectively used for agricultural crops growth and germination.

## 4. Conclusion

Thus, we found primary conditions of biogenic nanoparticles synthesis using various herbal extracts. Plant screening revealed bioactive substances. Studies identified flanonoids providing strong deoxidizing features of bitter wormwood (*Artemisia*), yarrow (*Achilléa nóbilis*) and plantain (*Psylliostachys*). Nickel and cobalt nanodispersion synthesis with bitter wormwood extract was done at 45°C, pH=4, for 1 hour. Nanoparticles $Ni^0$, $Co^0$ synthesized from bitter wormwood have the following average size according to microscope data: 70 $Ni^0$, 73 $Co^0$ µm, with NPs having lozenge/prism shape.

$Ni^0$, $Co^0$ nanodispersion synthesis with yarrow extract was done at 45°C, pH=4, for 1,5 hours. $Ni^0$, $Co^0$, synthesized from ethanol yarrow extract have the following size: 68 $Ni^0$; 74 $Co^0$ µm, with NPs having lozenge and lozenge/prism shape. Nanoparticles $Ni^0$, $Co^0$, synthesized from ethanol plantain extract have the following size: 72 $Ni^0$; 76 $Co^0$µm, with NPs having prism shape.

New growth intensifier (M-Nitro-Ni,Co) was used to intensify annual plants and vegetables germination and growth; as a result, they had stronger roots, thicker stems, dark-green leaves and larger size compared to test ones. New growth intensifier M-Nitro-Ni,Co-2023 increased agricultural crops growth and germination rate by approximately 15%.

**Conflict of Interest:** There is no conflict of interest between the authors.

## ӨСІМДІКТЕР НЕГІЗІНДЕ МЕТАЛЛ НАНОБӨЛШЕКТЕРІН СИНТЕЗДЕУ ЖӘНЕ ОЛАРДЫ АУЫЛ ШАРУАШЫЛЫҒЫ ДАҚЫЛДАРЫНЫҢ ӨСУІ МЕН ӨНУІН ЖАҚСАРТУ ҮШІН ҚОЛДАНУ

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**Түйіндеме:** *Кіріспе.* Биосинтез арқылы алынған кейбір металдардың нанобөлшектері олардың бетіндегі сығындыдан компоненттердің сорбциялануына байланысты оңтайлы фармакологиялық қасиеттерге ие, бұл өз кезегінде олардың қолдану аясын кеңейтеді. Осы себептерге байланысты металл нанобөлшектерін синтездеуде биологиялық әдістерді оңтайландыру мәселесі қазіргі уақытта өзекті болып табылады. *Жұмыстың мақсаты: Artemisia (ащы жусан), Psylliostachys (жолжелкен), Achillea nóbilis (мыңжапырақ)* өсімдік сығындыларын пайдаланып, металл нанодисперсияларын синтездеу және ауылшаруашылық дақылдарын өсіруде қолдану бойынша тәжірибелік мәліметтерді ұсыну. *Әдістеме.* Бұл әдістің ерекшелігі өсімдік материалдарынан сығындыларды дайындау арқылы нанобөлшектерді синтездеудің экологиялық таза тәсілін қолдану болып табылады. Синтезге арналған өсімдік шикізаты, синтез жағдайлары (температура, pH мәні, металл тұздарының қатынасы: экстракт) эксперименталды түрде таңдалды. Ащы жусанның сығындысы болған кезде *Psylliostachys немесе Achillea nóbilis* қолданғанға қарағанда, нанобөлшектердің түзілуі тезірек жүретіні анықталды. Синтезделген күміс нанобөлшектері микроскопия және спектроскопия көмегімен зерттелді. *Зерттеу нәтижелері.* Алынған бөлшектердің өлшемдері 70 пен 100 мкм аралығында, бұл наноқұрылымды никель мен кобальт бөлшектерін дәлелдейді. Бөлшектердің орташа мөлшері 80 мкм. Бір жылдық шөптер мен көкөністердің өсуі мен өнуін арттыру үшін жаңа өсу стимуляторы M-Nitro-Ni,Co қолданылды; нәтижесінде олардың тамыры күшті, сабақтары қалың, жапырақтары қара-жасыл және сыналғандармен салыстырғанда үлкенірек болды.

**Түйін сөздер:** нанобөлшектер, сығынды, металл нанодисперсиясы, жасыл синтез, *Artemisia (жусан), Psylliostachys (жолжелкен), Achillea nobilis (мыңжапырақ)*


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## СИНТЕЗ НАНОЧАСТИЦ МЕТАЛЛОВ С ИСПОЛЬЗОВАНИЕМ РАСТЕНИЙ ДЛЯ УВЕЛИЧЕНИЯ РОСТА И ВСХОЖЕСТИ СЕЛЬСКОХОЗЯЙСТВЕННЫХ КУЛЬТУР

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**Резюме:** *Введение.* Наночастицы некоторых металлов, полученные путем биосинтеза обладают интересными фармакологическими свойствами из-за сорбции на их поверхности компонентов из экстракта, что расширяет круг их применения. По этим причинам вопрос оптимизации биологических способов синтеза наночастиц металлов актуален в настоящий момент. *Цель настоящей работы* – представить экспериментальные данные по синтезу нанодисперсий металлов с использованием растительных экстрактов *Artemisia (горькая полынь), Psylliostachys (подорожника), Achillea nóbilis (тысячелистника)* и использования при выращивании сельскохозяйственных культур. *Методология.* Особенностью этого метода является использование более экологичного подхода к синтезу наночастиц путем приготовления экстрактов из растительного сырья. Экспериментально подобрано растительное сырье для синтеза, условия синтеза (температура, значение pH, соотношение солей металлов:экстракт). Установлено, что в присутствии экстракта полыни горькой образование наночастиц происходит быстрее, чем при использовании *Psylliostachys (подорожник) или Achillea nobilis (тысячелистник).* Синтезированные наночастицы серебра были изучены методами микроскопии и спектроскопии. *Результаты.* Полученные размеры частиц составляют от 70 до 100 мкм, что свидетельствует о наноструктурированных частицах никеля и кобальта. Средний размер частиц составляет 80 мкм. Новый стимулятор роста M-Nitro-Ni,Co использовался для увеличения роста и всхожести однолетних трав и овощей; в результате они имели более сильные корни, более толстые стебли, темно-зеленые листья и больший размер по сравнению с опытными.

**Ключевые слова:** наночастицы, экстракт, нанодисперсия металлов, зеленый синтез, *Artemisia (полынь), Psylliostachys (подорожник), Achillea nobilis (тысячелистник)*

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